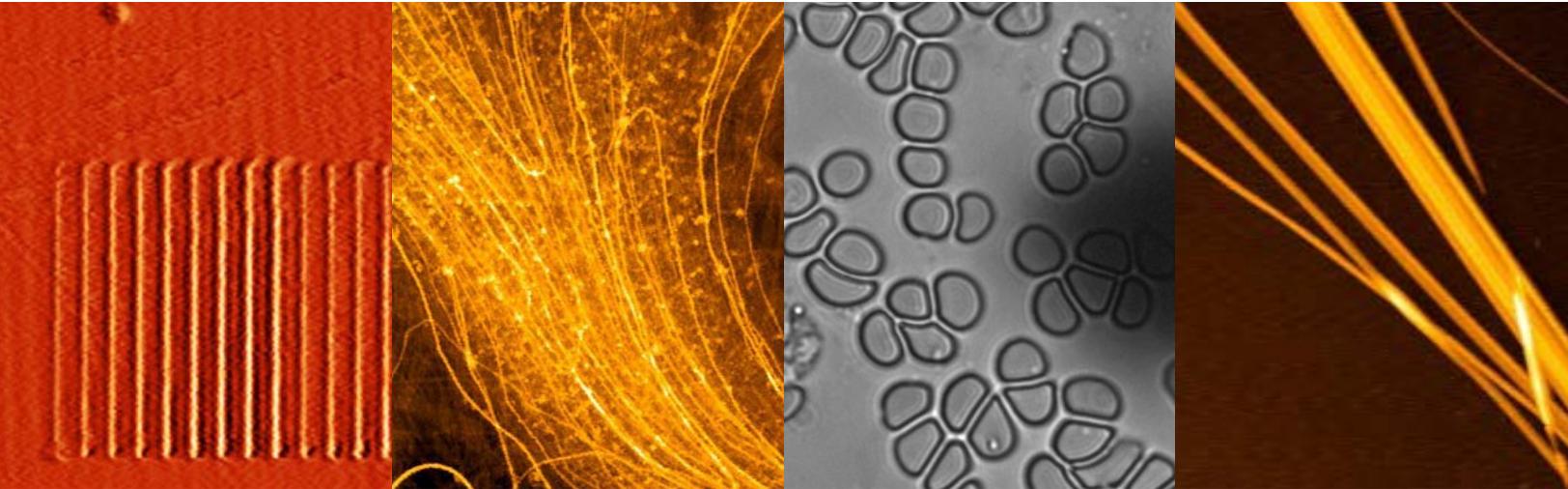


AFM WORKSHOP™



AUTOMOTIVE INDUSTRY APPLICATIONS OF ATOMIC FORCE MICROSCOPY

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1.0 INTRODUCTION

The automotive industry continually develops advanced materials to overcome the challenges it faces in making safer, more fuel efficient automobiles that are less dependent on fossil fuels. Employment of nanotechnology has the potential to bring the necessary advancements into materials utilized in many automotive parts, from metals to polymers. Nanoscale development and processing of the materials requires imaging, analysis, and measurement on a nanoscale level. Imaging techniques, such as electron microscopy, provide necessary resolution, but generally require painstaking sample preparation, imaging in a vacuum, and only provides a 2D image of the surface as an output. However, atomic force microscopy (AFM) can readily image any surface under ambient conditions and provide 3D topography of the surface. Moreover, AFM allows the measurement of many other surface properties, such as conductivity and stiffness, which could be relevant to the automotive industry. Scientific research already shows many automotive-related applications of AFM. It is clear that AFM is an indispensable tool in many aspects of automotive material development and in the future, AFM may be required for advanced manufacturing.

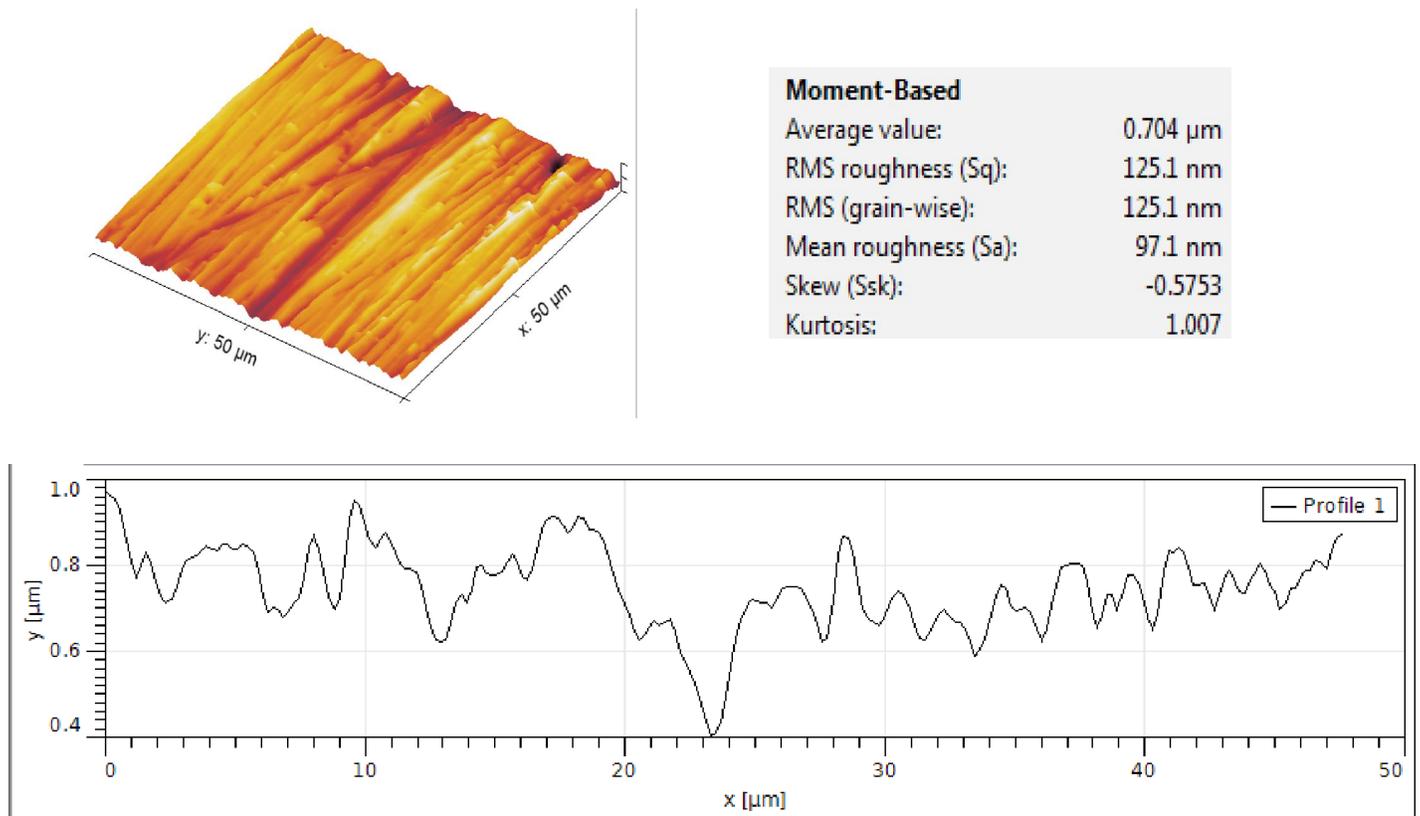


Figure 1: The upper left depicts a 3D AFM image of a highly polished copper substrate with surface texture parameters for the image. Below is a line profile showing the heights of the sample's surface features.

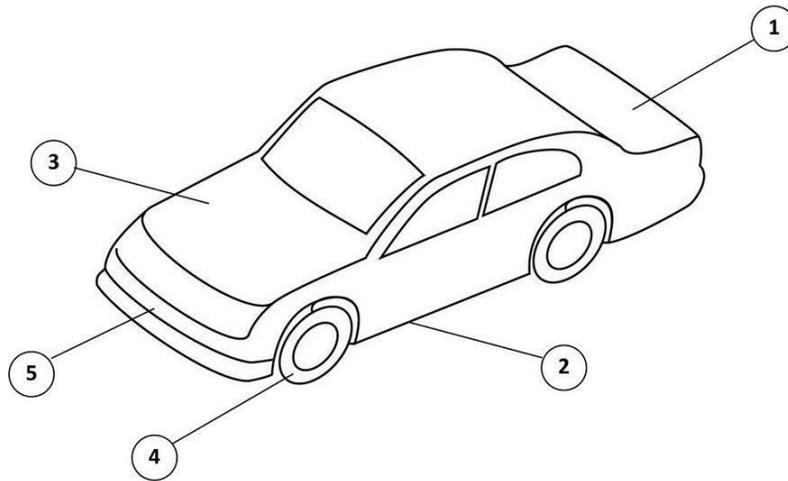
2. AUTO MANUFACTURING INDUSTRY NEEDS AND PROBLEMS

Some of the significant challenges the automotive industry faces include increased competition, emission goals imposed by governments, and digital transformation. Increased competition requires car companies to adopt advanced technologies to increase comfort and driving pleasure. Similarly, emission goals force the car industry to produce lighter vehicles with better tire rolling resistance, engine efficiency, and catalysts. Last but not least, digital transformation is everywhere and cars are expected to comply with that. In order to catch up, the car industry needs disruptive innovations, primarily in the materials it utilizes. Nanotechnology stands as the greatest candidate to deliver these innovations.

As nanotechnology continues to revolutionize material technologies, it will have big impacts on the auto industry, which exploits a diversity of materials - from metals and ceramics to polymers and advanced materials. Nanotechnology/nanomaterials are expected to bring reduced engine emissions, enhanced fuel efficiency, safe driving, quiet vehicles, and self-healing body and windscreens.

The basic ways nanomaterials can revolutionize the car industry:

1. Providing lighter yet stronger materials (limiting fuel consumption/increasing safety)
2. Improving catalytic efficiency (emission reduction)
3. Developing more efficient batteries for electric vehicles
4. Additives for tires (reducing tire rolling resistance and wear)
5. Coatings/Lubricants/fuel additives (improving engine efficiency, reducing friction, wear & tear, etc.)



Considering these developments, we can envision that devices and materials used in the car industry will increasingly become processed at the nanoscale. Their structure and capabilities at the same scale will play a decisive role. Therefore, in parallel, the car industry will need technologies to measure and analyze these new nanodevices and nanomaterials. Regarding the broad range capabilities of AFM, we believe it is a proper investment for the car industry now. Below are five ways how nanomaterials will impact the car industry with an accompanying description of roles that AFM will play.

Weight reduction - increased safety: Nano-enabled lightweight materials have already started to find a place in automobiles. For example, polymeric nanocomposites are used by Toyota Motor Company to make a timing-belt cover. Nanocomposites are obtained by embedding nanoparticles into a matrix of conventional materials, such as polymers, so that the latter has superior mechanical, thermal or processing features. Nanocomposite plastic parts are expected to provide 25% and 80% weight reduction when compared to highly filled plastics and steel, respectively.² These efforts will pave the way to decrease material and fuel consumption, while increasing safety (i.e. crash resistance). On the other hand, a thorough analysis will be required to understand the structure-function-process relationship at the nanoscale to develop better materials, as well as to control quality and eliminate any contamination. AFM can provide a 3D surface map of morphological, mechanical, and thermal properties at the nanoscale.

Increased catalytic efficiency: Nanoscale engineering improves the efficiency of catalysts via increasing the surface area that comes into contact with substrates. Current catalytic converters rely on precious metals, such as platinum or palladium, to catalyze the conversion of three key pollutants - carbon monoxide, nitric oxides, and hydrocarbons - into less toxic byproducts.¹ The catalytic efficiency of these metals and non-precious metals can be enhanced by making these metals nanostructured. Moreover, porous nanocomposites could also be used as a pollution filter to suppress the emission of soot particles or toxic gases.² Identification of the surface topography at the nanoscale could serve to delineate the

structure-function relationship for catalysts and engineer more effective and cheaper versions.

Batteries for electric vehicles: Nanomaterials will be a significant player in the future of electric cars as they have the potential to improve the lifetime, safety, cycling stability, and tolerance to harsh conditions, while decreasing size, weight, and recharge time.^{1,2} Nanoparticles of silicon for lithium-silicon batteries, nanostructured platinum, and ultracapacitors are some examples of these materials. Nanostructured electrode materials, such as Si anodes and LiFePO₄ cathodes, have already entered the marketplace.² Nanotechnology could also pave the way for hydrogen-powered cars via nanostructured materials having higher hydrogen adsorption capacity. All these development processes will require measurement and analysis of many related issues, such as conductivity at the nanoscale, conductivity distribution on the surface, and the relationship between topography/mechanical properties/conductivity. AFM could provide answers to all of these questions with its various techniques, such as topography imaging, conductivity imaging, and stiffness imaging.

Additives for tires: On top of being an economical issue, the limited lifetime of tires is also an environmental issue. Almost a billion tires are wasted each year according to the World Business Council for Sustainable Development. In order to increase wear resistance, reinforcing filler materials, such as carbon black micro/nanoparticles, are used in tires (30%).^{1,2} Carbon nanotube sensors could also be used to monitor tire wear. Studying the effect of various filler materials on tire wear requires measuring tread depths upon stress. AFM is the most suitable technique to delineate such 3D topographies at the nanoscale. Using phase imaging mode, it can also differentiate various filler materials from each other and rubber. This would help to visualize filler material distribution on the tire and distinguish the damage occurred to rubber from the damage to filler materials.

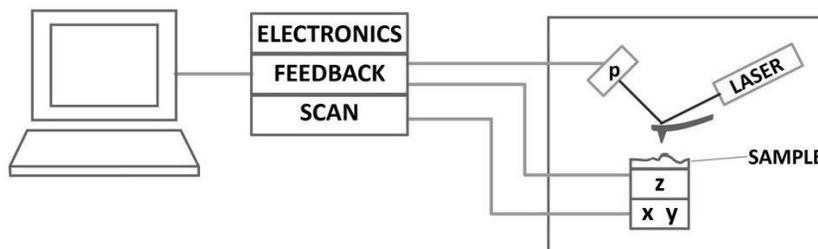
Coatings & Lubricants: Automobile paints are expected to be resistant to scratch and everyday wear and tear, also repel dirt, and ultimately, have self-healing ability. Nanotechnology has the potential to play a fundamental role in many ways here as well. Surfaces will be patterned at the nanoscale since nanostructured surfaces improve paint adhesion and color durability.¹ Nano-enabled paints and varnishes will also add another protection against scratches and wear and tear.³ Active nanosurfaces that can clean themselves will be a standard application on windscreens and car body shells. Similarly, dirt-repellent and antimicrobial textiles and surfaces, nanoparticulate air filters, and anti-glare films of mirrors and instruments will increase comfort, driving pleasure, and aesthetic experience. The antireflection coating based on multiple nanolayers on glass is used by Audi and by Daimler Chrysler.²

Nanotechnology-based coatings are also promising for reducing friction and wear in engine parts, hence limiting fuel consumption. Nanoparticles are a matter of interest here as they mediate between bulk material and molecular structure. Protective coatings made up of ceramic nanoparticles for various engine parts, CrN nanoparticles for piston ring and piston head or thin diamond-like carbon coatings for fuel injector will reduce friction, wear, and abrasion while improving fuel efficiency.^{2,4} Nanoparticles are also investigated as a means to improve thermal and rheological properties of lubricants.

AFM will bring numerous capabilities in the analysis of coatings, such as determining the thickness of nanolayers, monitoring wearing in coats, scratch tests to evaluate scratch resistance, and detection of surface contamination.

3. INTRODUCTION TO AFM

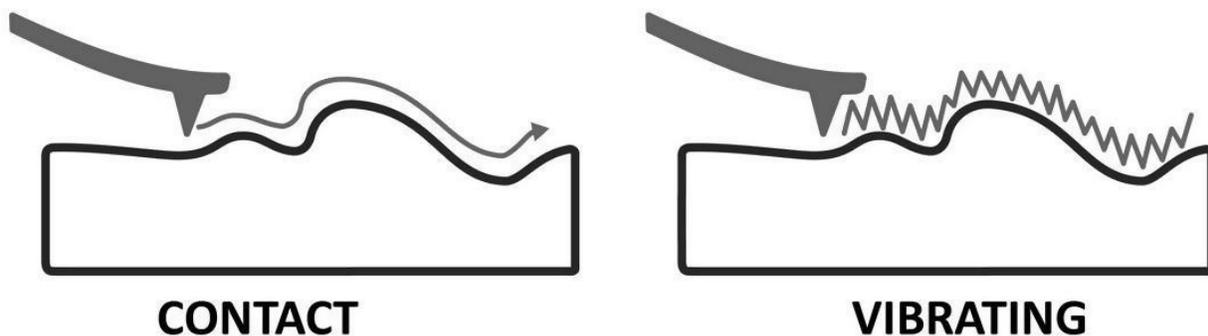
The working principle of the atomic force microscope (AFM) is based on the forces that arise when a sample surface is scanned with nanometer-sized tip (a few to 10s of nm) attached to a cantilever.



The advancement of the AFM over traditional stylus surface profilers is that a feedback loop is used to control the forces between the surface and probe. As the forces are controlled, very small probes may be used and will not break while capturing an image. There are two primary modes used for measuring the topography of a sample: contact mode and vibrating mode.

Contact mode: The probing tip is in contact with the surface throughout the imaging in contact mode. The short-range forces between the surface and tip cause the deflection of the cantilever, which is recorded to generate the topographical image of the surface. The tip-surface contact in this mode can potentially damage the surface or wear the tip. Hence, this mode may not be suitable for imaging soft surfaces. On the other hand, continuous contact with the surface allows the identification of other features, such as friction (lateral force imaging) or stiffness/elasticity map of the surface (force modulation imaging). In lateral force or frictional force microscopy, lateral deflections of the cantilever arising due to forces which are parallel to the plane of the sample surface, such as friction force, are measured. ⁵This allows finding inhomogeneities on the material which give rise to variations in surface friction.

Vibrating Mode: In this mode, a probe at the end of the cantilever is vibrated up and down. As the vibrating probe begins to interact with a surface, the vibration amplitude is dampened. The amount of damping is proportional to the amount of force placed on the surface by the probe on each oscillation of the vibrating probe. A feedback loop is used to maintain a fixed vibration amplitude as the probe is scanned across a surface. Forces between the probe and surface in vibrating mode can be as low as a few 10s of piconewtons.



Although AFM is widely known for mapping surface topography, that alone does not always provide the answers that researchers need to understand the material. ⁶ Fortunately, as a result of its capability to measure varying forces arising between the tip and sample, AFM can characterize a wide array of mechanical properties (e.g. adhesion, stiffness, friction, dissipation, and viscoelasticity), electrical properties (e.g. capacitance, electrostatic forces, work function, electrical current, conductivity, surface potential, and resistance), magnetic properties, optical spectroscopic properties and thermal properties, and solvent effects (via imaging at liquid environment) in almost real time. ⁷

Phase imaging: A phase difference between the oscillation of the cantilever and the signal that drives cantilever oscillation (e.g., piezoelectric crystal) is measured in tapping mode and visualized in phase imaging. ⁸ There is no phase contrast when the surface is homogenous or when there is no interaction between the tip and surface (i.e., the cantilever is well above the surface). However, if specific regions of the surface have distinct mechanical properties, they can be captured with phase imaging. This is because the cantilever loses a different amount of energy as the probe taps the surface areas with differing mechanical properties. Hence, phase imaging can be helpful to detect variations in mechanical properties, such as friction, adhesion, and viscoelasticity on surfaces. It could also be used to detect patterns of various materials such as polymers on the surface or used to identify contaminants which can not be distinguished with topography imaging.

AFM compared to SEM/TEM

Traditionally, the scanning electron microscope (SEM) and transmission electron microscope (TEM)

are used for measuring nanoscale images of samples for the automobile industry. The AFM offers an alternative to these costly techniques. Advantages of the AFM over electron microscopes include:

- Three dimensional images so surface textures can be directly measured.
- AFM measures images in ambient air and in liquids, and does not require large vacuum chambers.
- Images of very smooth and flat materials are readily imaged with AFM.
- The cost of acquisition and ownership of an AFM is a fraction of an SEM.

4. AUTOMOTIVE RELEVANT APPLICATIONS OF AFM⁹

4.1. Failure analysis

Failure analysis of adhesive bondings : The use of adhesives for joining body parts of an automobile shows an increasing tendency in the automotive industry since they leave minimal traces behind, enable joining of dissimilar materials with minimal risk of galvanic corrosion, and show excellent fatigue resistance. Regarding this purpose, AFM could be used to generate nanometer-scale topography of auto parts which adhesives join together. It can also be used to determine the failure mode of adhesive bonding, whether that is cohesive, adhesive, or a combination of both.

Failure analysis of systems based on MEMS sensors (e.g., airbags): Surface analysis techniques are crucial for MEMS because the latter have a high surface-area-to-volume ratio. Also, the common cause of MEMS-related failure is surface aberrations.¹¹ Hence, AFM stands out as a reliable tool for failure analysis of MEMS. For example, AFM could be used to identify surface roughness at the nanoscale, which is essential to identify a failure in MEMS devices.¹²

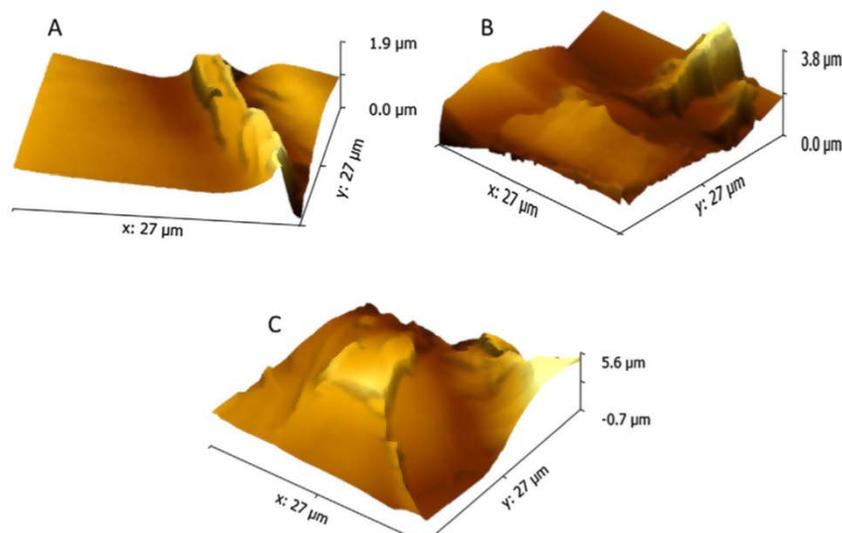


Figure 2: Nanocomposites of thermosetting polymer EPON 862 becomes more fracture-resistant when reinforced with nano-graphene. Here, scientists aimed to understand the effect of the crack deflection in newly gained fracture properties inhibiting crack propagation. AFM (AFM Workshop) was used to analyze the surface roughness, a parameter used to quantify crack deflection. Surfaces of the specimens reinforced with 0 wt% (a), 0.1 wt% (b), 0.5 wt% nano-graphene platelets (NGP) were studied with AFM after fracture. As can be seen from the images, fracture surface roughness increased with increasing NGP concentrations in the nanocomposite material. Roughness parameter (Ra) calculated from AFM results indicated 150% and 260% increase in the 0.1 wt% and 0.5 wt% NGP reinforced material, respectively, when compared to baseline material.

4.2. Corrosion analysis

Automobiles are often exposed to extreme environmental conditions, such as temperature, road salt, and UV, which can induce corrosion.¹⁴ Therefore, materials that can endure corrosion are highly needed. This requires understanding corrosion mechanisms, its initiation, and progress at the nanoscale. AFM allows us to understand and monitor corrosion in real-time. Since AFM enables measurement in both gaseous and liquid environments, it can be used to investigate solid-liquid interfaces. Having nanoscale initiation sites and happening in solid-liquid interfaces, the corrosion process can be successfully analyzed with AFM. High-speed AFM (HS-AFM) has been used at contact mode to investigate localized corrosion events occurring on thermally sensitized AISI 304 stainless steel in an aqueous solution of 1% sodium chloride (NaCl). High-speed scanning allows faster surface scanning compared to happen conventional AFM, thus corrosion events happening in real-time can be mapped. This reveals insights about the initiation mechanism of steel corrosion. For example, intergranular pits form as chains along grain boundaries. Such efforts could pave the way to engineer materials at the nanoscale to increase their resistance against corrosion.

4.3. Composition analysis

AFM is adept in several ways in the characterization of polymer blends. For example, it allows understanding polymer microstructure via exquisite techniques, such as phase imaging. This characterization is critical to identify the relationship between structure-performance relationship and develop optimal materials.^{15, 16}

In the automotive industry, AFM could be used to analyze tire treads. Treadwear, which is caused by various kinds of damage upon tires, is a critical process that defines the lifespan of the tires. Filler materials are used to reinforce tire tread step against damages. AFM phase imaging, which allows distinguishing different materials, has been used to determine the depths of treads supported with various filler materials.⁸ In this way, different tread-filler formulations can be studied by exposing them to the same kind of stress/wear and measuring tread depths.

Nanocomposites are expected to have enhanced usage in the automotive industry. Their performance is a structural function: the existence of optimal interfaces between the polymer matrix and nanofiller. Nanomechanical characterization with AFM allows identifying interface structure with unprecedented precision.¹⁷

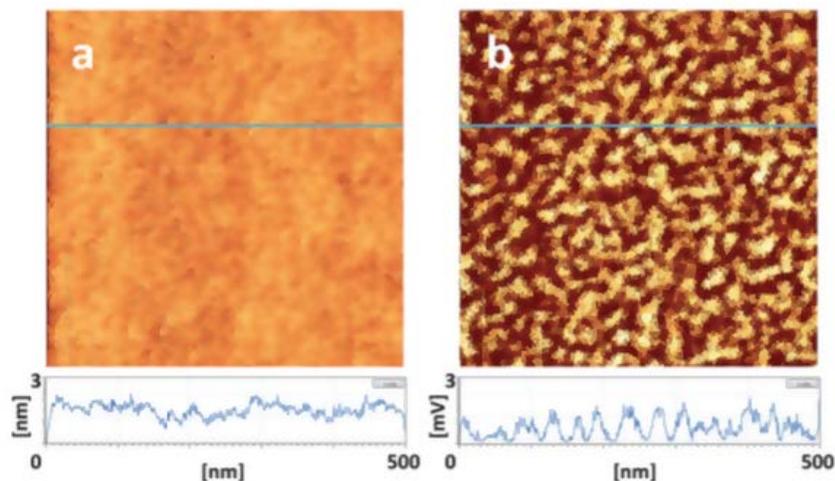


Figure 3. Nanoscale structure of membranes used as separators in vanadium redox flow batteries is critical for their optimal functioning. Here, PLA-PSU-PLA triblock copolymer membranes of polysulfone (PSU) and polylactide (PLA) are synthesized and evaluated as separator membrane. Tapping mode AFM (AFM Workshop, TT-AFM) was used to characterize the nanoscale structure. While topography imaging indicates an almost flat surface at the nanoscale, phase imaging reveals a phase-separated morphology with distinct domains of 20-30 nm size on the same surface. Such phase contrasts are generated by

differences in the mechanical properties of domains which are highly likely caused by variations in material composition. This study demonstrates the richness of information that can be obtained by employing various AFM techniques to investigate any surface.

4.4. Morphology and composition characterization of surfaces and coatings

Surface analysis of automotive materials: Polypropylene has many optimal features for the auto industry, such as low density, inertness, corrosion resistance, and ease of recyclability. However, traditional methods of joining polypropylene parts to other parts of the vehicle, such as mechanical fastening, are suboptimal. Therefore, the use of adhesive bonding stands as a promising alternative. However, polypropylene surfaces should have more suitable surface energy and wettability to be applied for adhesive bonding. These properties are functions of surface chemistry and nano-topography. Guild et al. investigated the nano-topography produced as a result of a forced air-plasma pre-treatment of polypropylene surface by using AFM. This method showed more promise when used with a polyurethane adhesive. AFM was able to differentiate nanoscale “pits” and “nodules” formed when the surface was exposed to different speeds of the pre-treatment. Again, it is noteworthy that AFM could be used to identify the “right” surface topography required for the desired physical characteristics, such as wettability in this case.

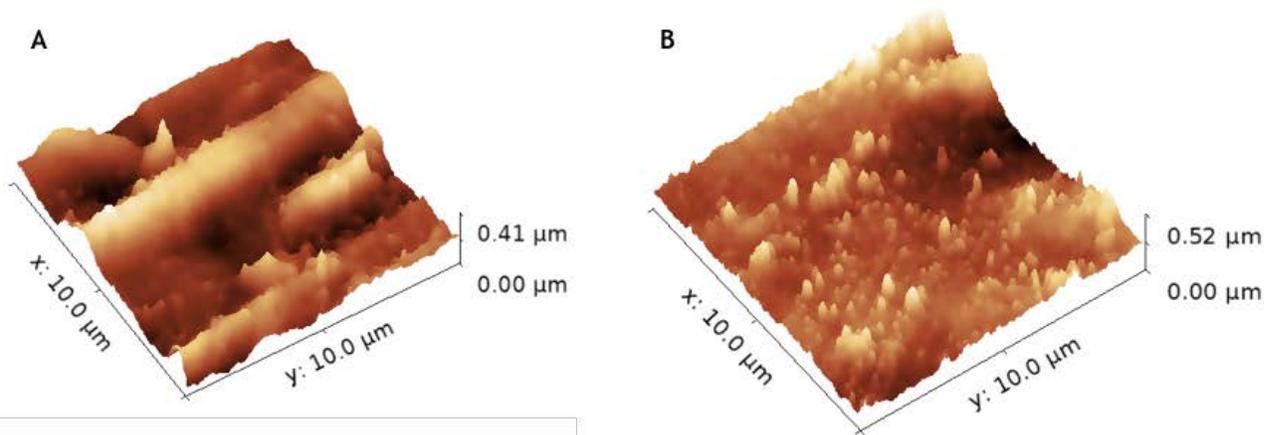


Figure 4. Long glass fibers reinforced polypropylene (PPLGF) are used for various purposes by the automotive industry, such as in instrument panels, front end systems, and as engine covers (http://www.temp.speautomotive.com/SPEA_CD/SPEA2006/PDF/b/b1.pdf; <https://www.lfirt-plastic.com/news/long-glass-fiber-polypropylene-comp-10781637.html>). Therefore, their morphological characterization upon different processing conditions is crucial. These images are obtained using AFMWorkshop equipment employed by one of the industry’s players. Fibrous structures and their dimensions can be seen in (a), which depicts PPLGF obtained by a normal processing method. However, fibers are destroyed in (b) when the PPLGF sample is treated with fire on the surface.

AFM to characterize automotive coatings: Many contemporary coating products consist of multiple layers to provide optimal performance. AFM can be used to delineate thickness, morphology, mechanical properties, and the composition of coatings utilized in automotive manufacturing. For example, it can resolve diverse polymer phases and discover their nanomechanical features. Additives used in coating material can change nanoscale specialties of the structure, which is highly critical for the performance of the coating. AFM can be used to differentiate those structures and find appropriate additives to generate optimal nanoscale structures.

Figure 5. An image of PVD indium thin film coating, which is used inside a car as a standard ACC cruise plating. This layer of film makes the AAC auto-cruise logo look metallic. AFM imaging allowed for a comparison between the quality of thin film in various regions of coating directly in the PVD

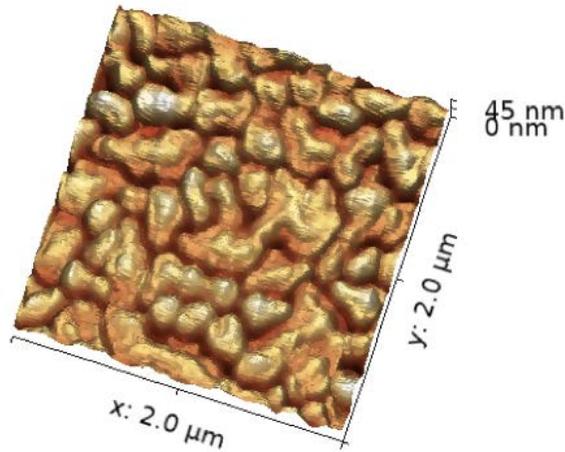


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4.5. Mechanical analysis

Scratch testing of paints and coatings: ²¹ One of the powerful applications of the AFM is to analyze mechanical features of the surface. In this context, researchers use AFM to perform scratch tests to measure scratch resistance of various automotive paints. Scratch resistance is defined as the resistance to permanent deformation caused by mechanical force applied by the AFM tip. Here, instead of silicon or silicon nitride tips, which break easily during nano scratching, diamond tip mounted to a metal foil cantilever is used. The depth of the scratch can range from 50 nm to several 100 nm, while the width can range from several 100 nm to 1-2 μm. Therefore, the damage is confined within clearcoat, the thickness of which is about 15-20 μm. Nanoscratching is performed via predetermined scratch force. Finally, the depth and width of scratches are measured through surface topographical analysis of the surface. Pourdeyhimi & Wang compared scratch resistance of three clearcoat materials by using a scratch test: acrylic polyol - melamine-formaldehyde (MF) - crosslinked clearcoat, acrylic acid - epoxy-crosslinked clearcoat and acrylic polyol - isocyanate-crosslinked clearcoat.

4.6. Tribological analysis

A systematic study of friction, lubrication, and wear help to improve the lifespan of mechanical components. In the automobile industry, successful lubricants are expected to form nanometer-sized layers on the surface of the substrate material. The thickness of the lubricant layer significantly affects macroscale lubrication behavior. Therefore, characterization of lubricant films is critical for the development of novel lubricants with optimal functioning.²³

Gosvami et al. were interested in the functioning mechanism of anti-wear additives, zinc dialkyldithiophosphates (ZDDPs), and investigated the tribofilms formed by ZDDPs.²³ Anti-wear additives in lubricants can prolong the lifetime of automotive parts by minimizing metal-to-metal contact. ZDDPs reduce wear by decomposing at rubbing surfaces and forming protective tribofilm. However, the mechanisms of tribofilm growth and what determines its morphology and thickness (50-150 nm) is not known. Moreover, ZDDP can produce Zn-, P-, and S-containing compounds in automobile engine exhaust, which can lower catalytic converter efficiency and lifetime. Thus studying the growth mechanism of tribofilm is relevant for the development of better anti-wear additives. Here, researchers investigated the effects of pressure exerted through the single point of contact on tribofilm growth by using contact mode AFM. They managed to obtain the real-time images of tribofilm growth on the surface as the AFM tip slid over the surface. The AFM tip, in this case, was used simultaneously for two functions: 1) to induce pressure on the surface which simulated metal-metal contact in real-time and 2) to obtain topographical images of the surface. Results indicated that tribofilm formation starts from the deposition of ZDDP molecules at random nucleation sites, while film growth is subsequently driven by contact forces induced by AFM tip. They observed that the growth rate of the tribofilm increases until the contact pressure of 5.2 GPa after pressure-induced wearing limits film growth (Figure 6). Altogether, the data suggest that the anti-wear property of ZDDP is caused by mechanical protection provided by the tribofilm instead of corrosion inhibition. This study shows that the AFM could be used to simulate pressure exerted by the metal-metal contact at the nanoscale and the functionality of various lubricant additives could be tested in this way.

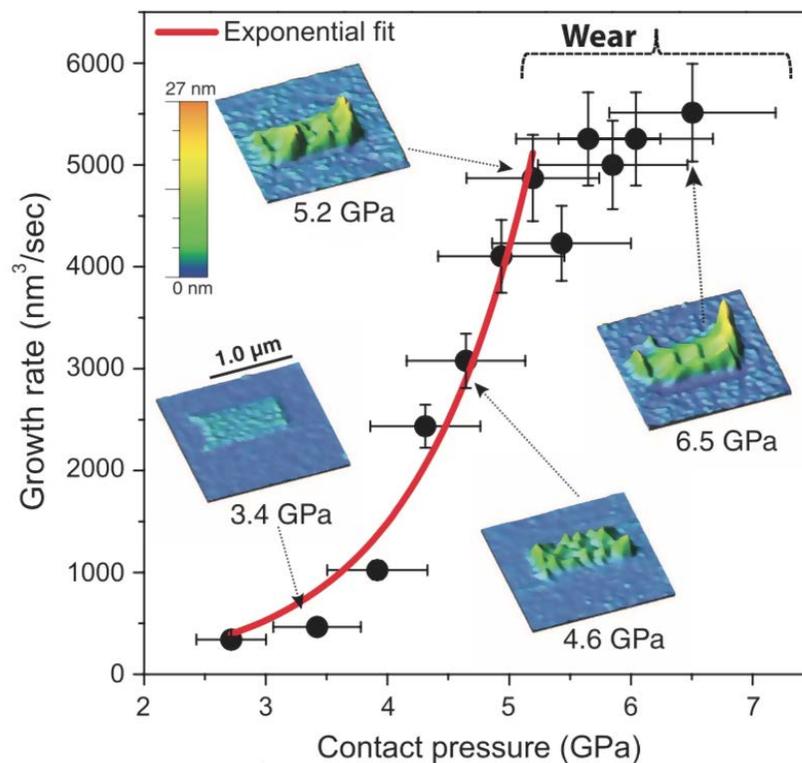


Figure 6. Dependence of ZDDP tribofilm growth rate on contact pressure induced by AFM tip.

4.7. Electrical and magnetic characterization

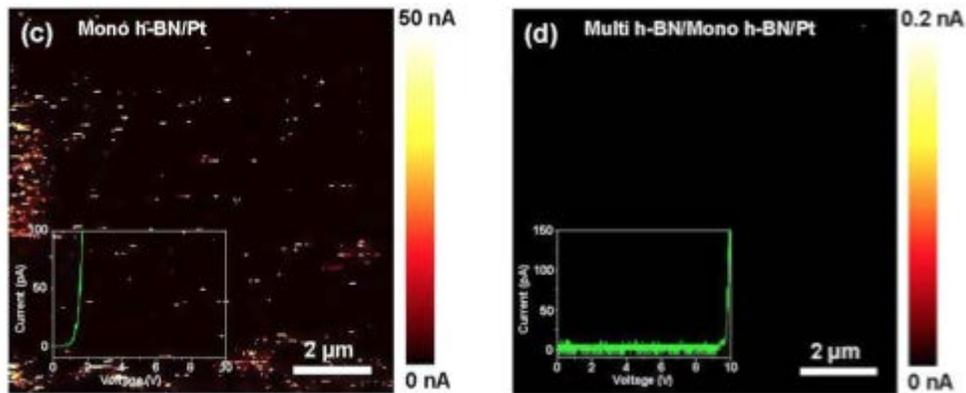


Figure 7. The insulating properties of monolayer and multilayer hexagonal boron nitride (h-BN) were investigated by using conductive AFM (TT-AFM, AFM Workshop).²⁴The insets are the I-V curve for the breakdown voltage measurement. Monolayer (a) and 11-nm-thick multilayer h-BN film (b) were grown on Pt foil. These surfaces were mapped using a Pt-coated AFM tip, which was used to apply 1 V to sample. The multilayer film showed higher insulating performance than the monolayer film. Spots on monolayer h-BN film are the leakage current caused by defects on the material. On the other hand, a multilayer h-BN film showed a spotless conductivity map indicating high insulation performance.

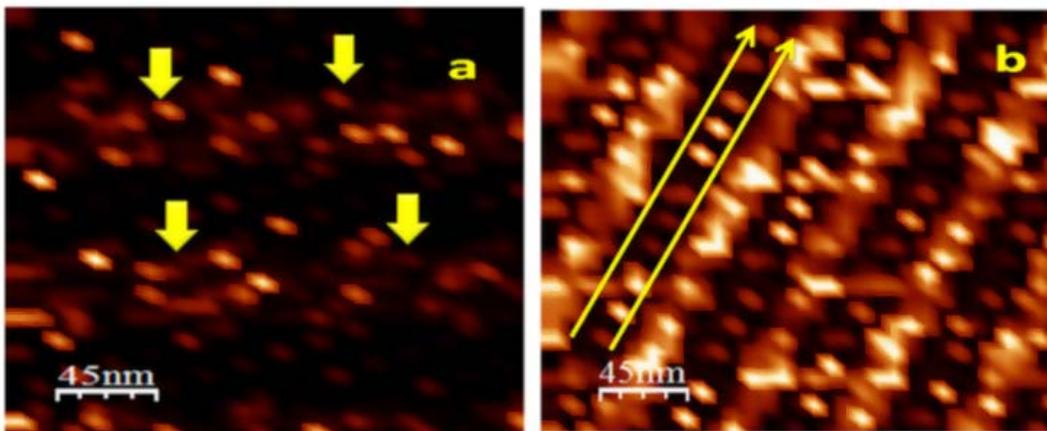


Figure 8. Nanoscale magnetic properties of nanocomposite multiferroic materials are characterized with TT-AFM (AFM Workshop). AFM images of nanocomposite $\text{Bi}_{0.85}\text{La}_{0.15}\text{Fe}_{0.75}\text{Co}_{0.25}\text{O}_3$ without magnetic field (a) and $\text{Bi}_{0.85}\text{La}_{0.15}\text{Fe}_{0.75}\text{Co}_{0.25}\text{O}_3$ integrated graphene oxide nanocomposite in the presence of external magnetic field (b) are shown. The magnetic core from cobalt is more on the surface of the particles which might cause in the increment of anti-ferromagnetic ordering in the $\text{Bi}_{0.85}\text{La}_{0.15}\text{Fe}_{0.75}\text{Co}_{0.25}\text{O}_3$. To support this assumption, AFM analysis was carried out by applying an external magnetic field from toy magnets. When an external magnetic field is applied to the $\text{Bi}_{0.85}\text{La}_{0.15}\text{Fe}_{0.75}\text{Co}_{0.25}\text{O}_3$, the particles are aligned along the field direction because of the nanosize grains and the magnetic surface due to high cobalt concentration in the nano-multiferroic sample. Besides, $\text{Bi}_{0.85}\text{La}_{0.15}\text{Fe}_{0.75}\text{Co}_{0.25}\text{O}_3$ integrated graphene oxide (GO) nanocomposite showed a change in the magnetic hysteresis that indicates the effect of GO's semiconducting behavior on the ordered magnetic moments in the multiferroic. This kind of magnetic property could bring advanced magneto-electric sensing to high-performance devices.

4.8. Analysis of energy storage devices

Many AFM modes have been used to understand morphological, mechanical, and electrical properties of electrode materials. The knowledge gained by these types of investigations will allow for optimizing electrolytes and guide the structural design of electrodes.²⁵ Moreover, it will contribute to our notion of failure mechanism, ion transport mechanism, and electrode material phase transformation. Understanding fundamental electrode degradation mechanisms is crucial for battery performance improvements. For example, the pattern of degraded regions on the surface of positive electrodes need to be identified to improve the lifespan of batteries.²⁶ AFM could serve to delineate morphological heterogeneities at the surface of both used and new batteries.

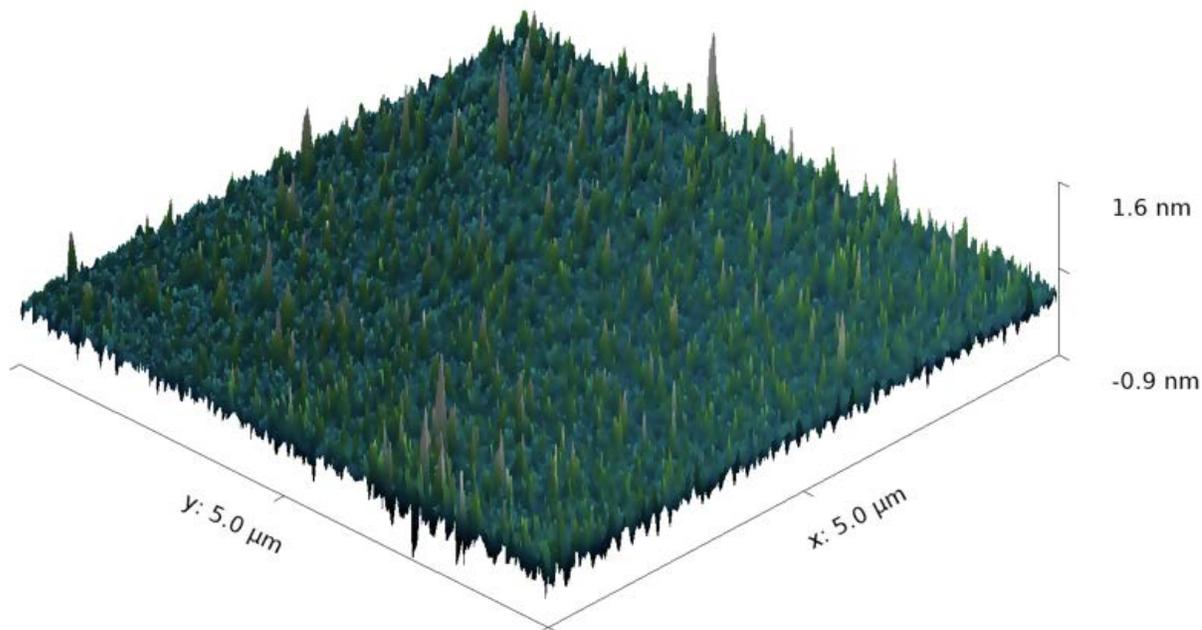


Figure 9. Image of a quantum dot doped mica surface. Graphene quantum dot doping improves the unit density of lithium batteries. Topographical analysis with AFM allows for the delineation of quantum dot density distribution on the surface, the dimensions of individual quantum dots, and the height of the quantum dot layer. Image was taken by AFMWorkshop equipment used by an automotive company.

Three-dimensional all-solid-state microbatteries (ASB), which generally use solid and dense materials as an electrolyte, are an emerging battery technology. Since this technology is based on nanoscale ionic processes in thin films, new measurement techniques are required to probe and understand the mentioned processes. Employing conductivity AFM (C-AFM), researchers were able to measure the electrical properties of materials used in Li-ion batteries with nanometer resolution. By using a conductive AFM tip, they were able to measure the current (using the tip as a nanoscale electrode) and the tip deflection at the same time, and thus, obtain 2D maps of local conductivity and topography (Figure 10a). Samples were prepared by depositing conductive material (Ni or Pt) onto a silicon wafer and the battery material under investigation onto the conductive material. The AFM tip serves as an electrode that provides electrical current into the sample which is collected from Ni. In this way, the detection of conductivity differences between the two materials (MnO₂ and LiMn₂O₄) was possible. Li-containing materials showed an increase in conductivity at lower voltages (Figure 10b). C-AFM also allowed for determining the distribution and density of highly conductive paths in cathode materials. Also, it is possible to combine this technique with secondary ion mass spectrometry (SIMS) to identify local chemistry, such as Li-ion concentration. This will help achieve structural, electrical, and chemical information from the same nanoscale surface.

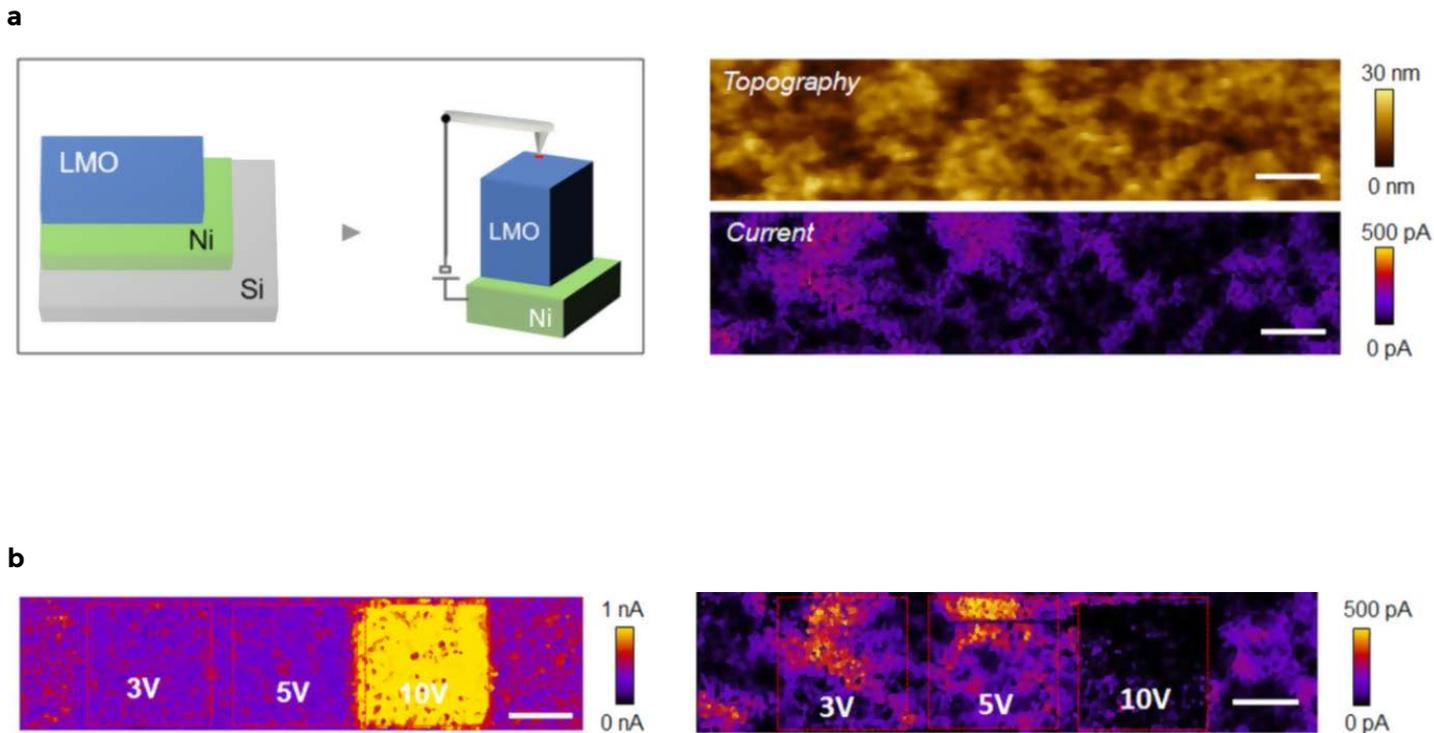


Figure 10. Measurement of electrical properties of solid electrolyte materials MnO₂ and LiMn₂O₄ (LMO).²⁸
 a) Left: Schematic of C-AFM experiment with LMO. Right: Topography and current maps on the electrodeposited LMO sample. b) Left: Current map of three areas from MnO₂ sample previously stressed at different dc bias values applying 1.5 V. Right: Current map of three areas from LMO sample previously stressed at different dc bias values applying 1.5 V.

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