Keysight Technologies

New Investigations into Energy

Keysight Nanomeasurement Systems for Energy-Related Research

Application Note

Applications

- Mechanical reliability and defect structures of GaN thin films
- Mechanical properties of a lithium rechargeable battery cathode
- Epitaxial growth of gallium nitride on sapphire substrate
- Characterization of photovoltaic materials at the nanoscale
- Conductivity measurement of Nafion membranes for fuel cells
- Surface potential changes of PEDOT: PSS films for solar cells

Overview

Keysight Technologies, Inc., the world's premier measurement company, offers powerful and intuitive instrumentation for researchers, scientists, and engineers whose primary focus is on providing better energy answers. The data presented here demonstrates just a few of the ways in which the latest atomic force microscopy (AFM), nanoindentation, and field-emission scanning electron microscopy (FE-SEM) systems from Keysight can be utilized to enhance energy-related materials research.

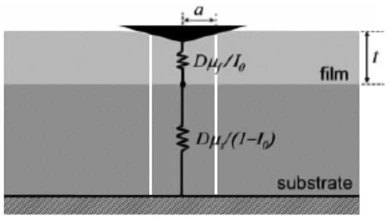


Figure 1. Modeling for Thin Films.

Mechanical Reliability and Defect Structures of GaN Thin Films

Thin films can be modeled by using two springs in series for calculating mechanical response of material (Figure 1). The Keysight Nano Indenter G200 characterizes dislocation nucleation and its effect on mechanical deformation behavior in semiconducting devices. The synthesis of epitaxial GaN (a hexagonal crystal) induces defects in the material. Thus, by studying the anisotropic nanomechanical behavior (e.g., modulus, hardness, and elastic-plastic transition) in different orientations of the material, the underlying physical nature of the defects can be identified. (Figure 2.)

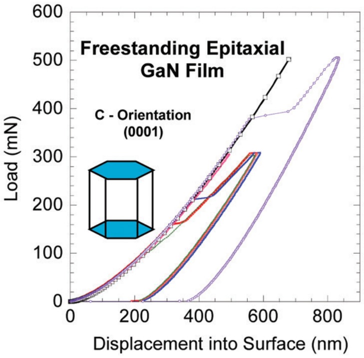


Figure 2. 3 µm GaN film characterization: arrows show defect nucleation in GaN.

Mechanical Properties of a Lithium Rechargeable Battery Cathode

In this example (Figure 3), a lithium-ion battery cathode composed of a mixture of active particles (LiMn2O4) and carbon black in a polymeric matrix of polyvinylidene fluoride (PVDF) was embedded in epoxy and mirror polished before mechanical testing with a Keysight Nano Indenter G200. The thickness of the cathode was approximately 150 µm. Lithium particles exhibit significant variance in their size and internal porosity, which leads to a different response in terms of mechanical properties (e.g., hardness and elastic modulus). Standard CSM and ultrafast Express Test DCM methods were utilized.[1]

Epitaxial Growth of Gallium Nitride on Sapphire Substrate

Here, GaN grown on sapphire substrate was investigated using Keysight's unique scanning microwave microscopy (SMM) mode, an exclusive technology that combines the nanoscale spatial resolution of AFM and calibrated electrical characterization capabilities. The heteroepitaxial growth of GaN layers on sapphire substrate can be compromised by unintentional doping during the growth process. Identification of the origin and the mechanism of incorporation of dopants is needed to optimize the GaN-based heterostructures for electronic devices.[2]

SMM Imaging of a cross-section sample of sapphire with alternating doped and undoped GaN layers in shown in Figure 4. The sapphire substrate is located on the left edge of the data set. The topography shows a step from the substrate to the GaN layers, several steps within the GaN, and some undefined contaminations at the right edge. The capacitance map shows some contrast at the substrate/film interface and a regular pattern of bright and dark lines towards the wafer surface. In the SMM dopant density image, dark regions correspond to the sapphire substrate, the highly doped marker layers, and the undoped GaN layers. The bright features are regions with a density lower than that of the highly doped GaN layer, but higher than that of the undoped GaN and the substrate.

It is clear that low charge carrier density regions are formed at the boundaries between the doped/undoped GaN layers due to the carrier diffusion. A region of "unintentionally doped" GaN film, between the substrate and the smooth GaN layer, is also visible from the carrier density image. These results helped to investigate the origin of "unintentionally doping" and the quality of doped layers.

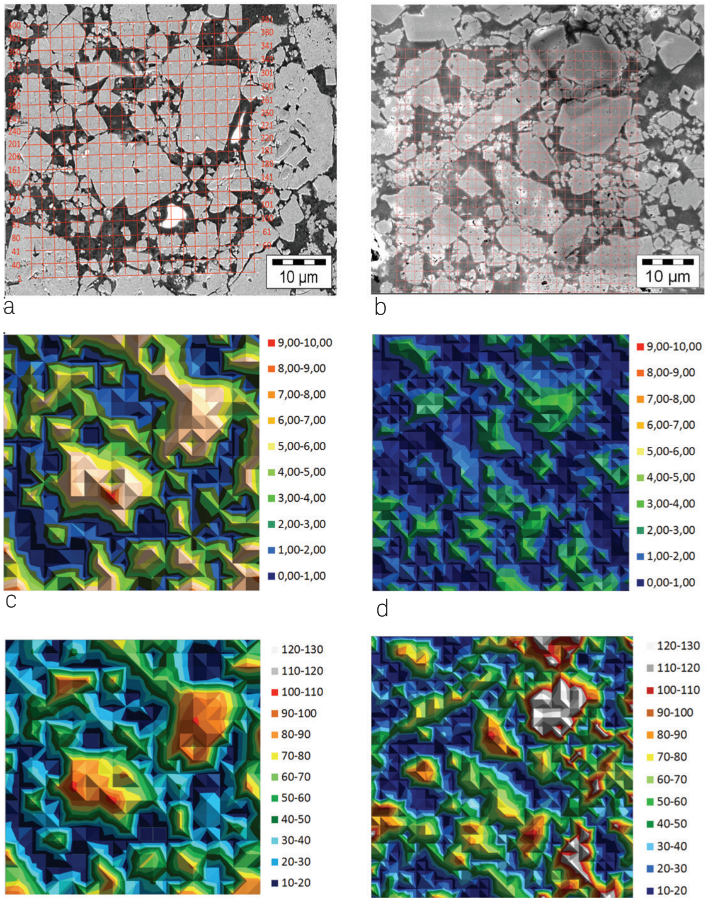


Figure 3. (a) XP-CSM Nanoindentation Grid. (b) DCM Express Nanoindentation Grid. (c) XP CSM Standard – Hardness [GPa], calculated at depth 100 nm. (d) DCM Express – Hardness [GPa]. (e) XP CSM Standard – Modulus [GPa], calculated at depth 100 nm. (f) DCM Express – Modulus [GPa].[1]

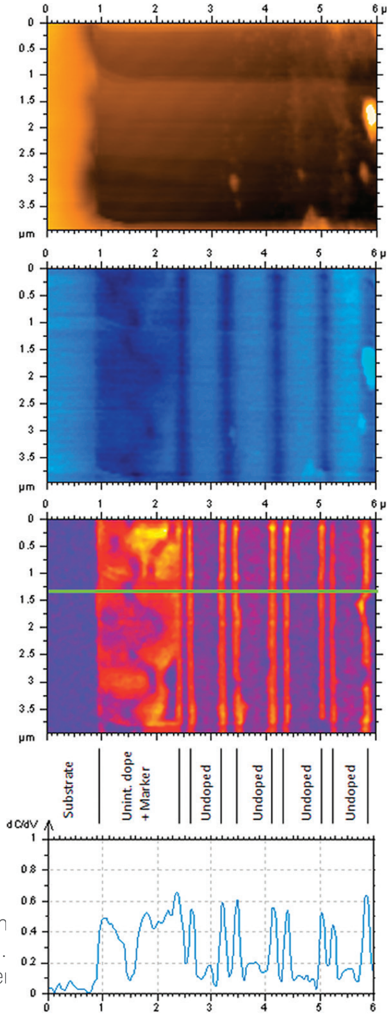


Figure 4. Topography, capacitan and dopant density map of GaN. Collaboration with Dr. R.A. Oliver University of Cambridge, UK.

Characterization of Photovoltaic Materials, Fuel Cells, and Solar Cells at the Nanoscale

Keysight’s unique single-pass KFM technique, which allows simultaneous topography and surface potential measurements, is well suited to many *in situ* studies. Kelvin force microscopy (KFM), current-sensing AFM (CSAFM), and scanning tunneling microscopy (STM) techniques from Keysight are utilized to perform single-molecule conductivity measurements for organic photovoltaic materials and devices. As organic photovoltaics accrue more interest in terms of creating cheaper solar cell devices, basic research into the conductive properties of the molecular components will become more crucial to synthesis and design.[3] Finding defect states in the cell tunneling junction material (used for battery separators) is key. (Figure 5.)

The ionic conductivity of Nafion (DuPont) depends on the hydration level of the membrane, and the control of proper hydration of the polymer electrolyte membrane (PEM) in a fuel cell has become a challenge in engineering design. Thus, it is critically important to understand the dependence of the ionic transport property of a PEM on its hydration state.[4] Keysight’s industry-leading environmental control and advanced electrical characterization techniques are ideal for performing *in situ* AFM studies of PEMs. (Figure 6.)

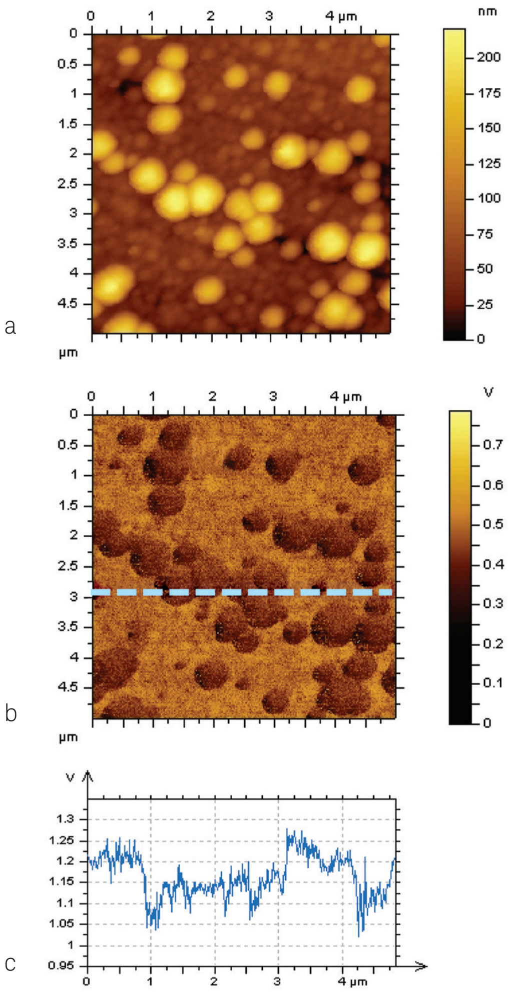


Figure 5. Topography (a) and surface potential (b) images of Si crystalline clusters deposited on amorphous Si. The potential profile along the dotted line is presented in (c).[3]

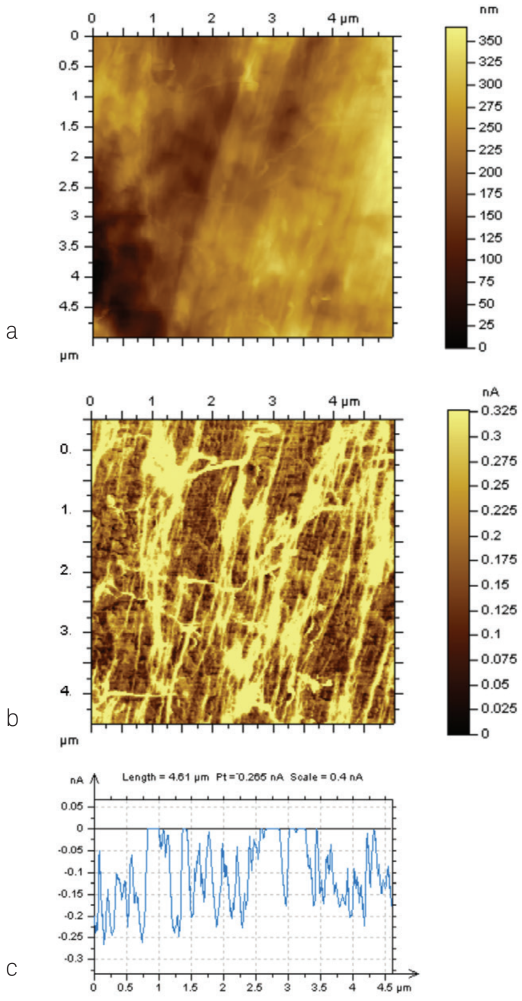
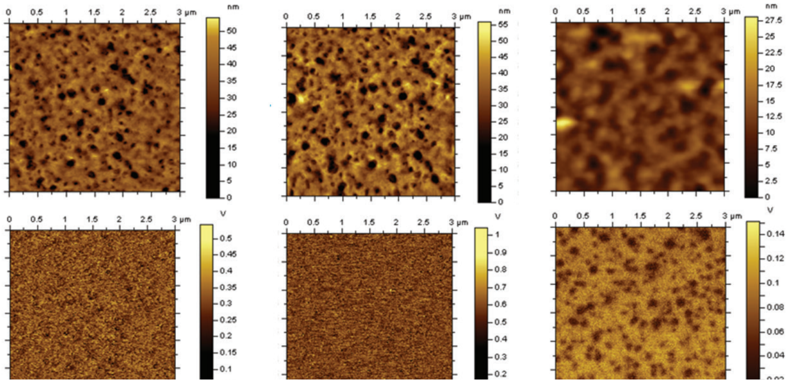


Figure 6. Topography (a), current (b), and current profile (c) for Nafion 212 at 50% RH.

Humid-environment studies can be helpful for compositional imaging of materials with a hydrophilic component, such as a conducting blend of poly(3, 4-ethylenedioxythiophene):poly(styrenesulfonate) [PEDOT:PSS]. Intrinsically conducting PEDOT:PSS blends are often used as a component in organic solar cells. Therefore, the morphology and electrical properties of PEDOT:PSS films are among the factors that influence cell performance.[5] (Figure 7.)



<5% humidity

40% humidity

>90% humidity overnight

Figure 7. PEDOT:PSS topography and surface potential images at three different humidity levels. As humidity increases, hydrophilic PSS swells and the surface potential contrast reveals the two components of PEDOT:PSS film. Cooperation: Dr. R. Berger (MPIP-Mainz, Germany).

Nanomeasurement Systems from Keysight Technologies

Keysight offers high-precision, modular nanomeasurement solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight's leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use nanomeasurement system technologies.

References

1. Sebastiani et al. "CSM and DCM-Express Nanoindentation Mapping on Lithium/Polymer Battery Composites," Application Note 5991-3519EN. 2013, Keysight Technologies, Inc.
2. M.A. Fenner, R.A. Oliver, "Investigating Highly Doped Marker Layers in GaN on Sapphire Using Scanning Microwave Microscopy," Application Note 5990-9693EN. 2012, Keysight Technologies, Inc.
3. Kibel et al. "Applications of KFM and CSAFM/STM in Characterizations of Photovoltaic Materials," Application Note 5990-5025EN. 2009, Keysight Technologies, Inc.
4. S. Wu, D-M. Zhu, "Applications of KFM, CSAFM and Env ronmental Control in Fuel Cell Research," Application Note 5990-4516EN. 2012, Keysight Technologies, Inc.
5. S. Magonov, J. Alexander, "Compositional Mapping of Materials with Single- Pass Kelvin Force Microscopy," Application Note 5990-5480EN. 2010, Keysight Technologies, Inc.

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