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Scanning Probe Microscopy for Nanoscale Characterization of Electrical and Magnetic Properties

Olivia Maryon Boise State University

Kari Higginbotham

Medha Veligatla Boise State University

Armen Kvryan Boise State University

Peter Müllner Boise State University

See next page for additional authors

Scanning Probe Microscopy for Nanoscale Characterization of Electrical and Magnetic Properties

Abstract

Atomic force microscopy (AFM) is a nanoscale scanning probe microscopy (SPM) characterization technique useful for obtaining topographical maps of surfaces and their associated nanomechanical properties. Complementary SPM modes such as Kelvin probe force microscopy (KPFM) and magnetic force microscopy (MFM) can simultaneously elucidate the electrical and magnetic properties of materials with nanoscale resolution, thereby expanding AFM's utility. KPFM measures the Volta potential difference between a conductive AFM probe and the sample surface, which can be related back to the work function of the material and correlated with co-localized elemental mapping via energy dispersive spectroscopy (EDS). This can be useful for understanding and predicting initiation and propagation of galvanic corrosion in metal alloys. MFM employs a magnetized AFM probe tip to detect magnetic interactions between the sample and the tip, thereby mapping out the magnetic structure of the sample surface. Here we present KPFM studies of case-hardened stainless steels engineered for bearing applications in high performance jet engines destined for operation in corrosive marine environments. MFM studies of Ni-Mn-Ga, a magnetic shape memory alloy, connect experimental data with computational modeling to understand the growth of twins in response to bending. Together, these studies highlight the widespread applicability of AFM, KPFM, MFM, and other SPM techniques for illuminating nanoscale structure-property relationships in material systems.

Authors

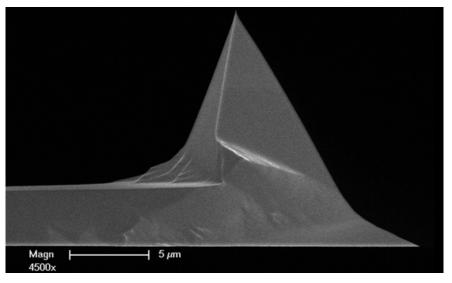
Olivia Maryon, Kari Higginbotham, Medha Veligatla, Armen Kvryan, Peter Müllner, Mike Hurley, and Paul H. Davis



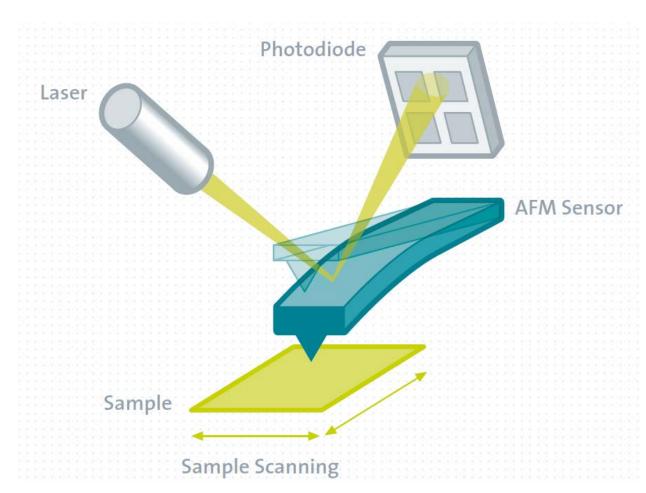
Atomic Force Microscopy (AFM)

AFM is a nanoscale scanning probe microscopy (**SPM**) technique useful for obtaining topographical maps of surfaces while simultaneously characterizing and mapping out their associated nanomechanical properties.

To do this, an extremely sharp (few nm radius of curvature) probe is brought in contact (or near-contact) with the sample and rastered across its surface.

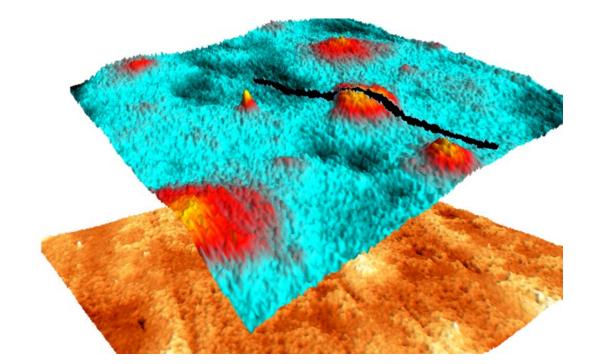


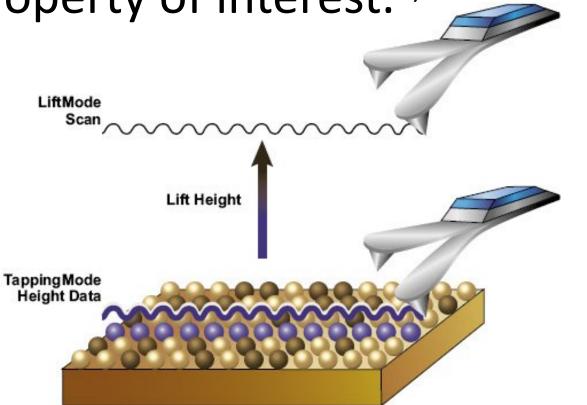




SEM image of an AFM probe. As shown at left,¹ a laser reflects off the back of the AFM probe and into a 4 quadrant photodetector that tracks changes in probe height or deflection, converting this into a topographical map of the surface.

Magnetic force microscopy, or MFM, employs a magnetized AFM probe tip to detect magnetic force interactions between the tip and the sample, thereby mapping out the magnetic structure of the sample surface. Kelvin probe force microscopy, or KPFM, measures the Volta potential difference between a conductive AFM probe and the sample surface, which can be related back to the work function of the material and correlated with co-localized elemental mapping via energy dispersive spectroscopy (EDS).² Both MFM and KPFM use an interleaved lift-off mode where the probe will first map out the topography and then lift off the surface to a designated height and retrace the surface topography while measuring the electromagnetic property of interest.^{1,3}





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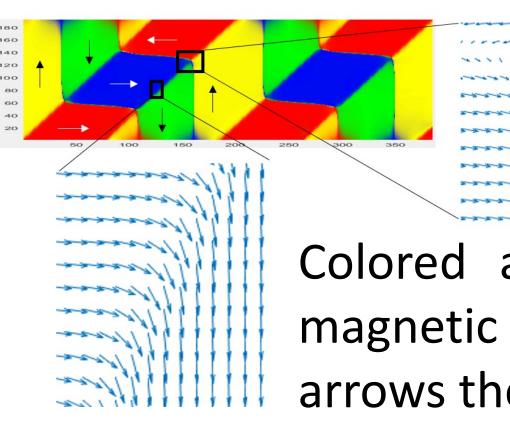
Olivia Maryon Undergraduate Research Assistant Surface Science Laboratory Materials Science & Engineering oliviamaryon@u.boisestate.edu



Scanning Probe Microscopy for Nanoscale Characterization of Electrical and Magnetic Properties Olivia Maryon,¹ Kari Higginbotham,² Medha Veligatla,¹ Armen Kvryan,¹ Peter Müllner,¹ Mike Hurley,¹ and Paul H. Davis¹ ¹Micron School of Materials Science & Engineering, ²Department of Mechanical & Biomedical Engineering

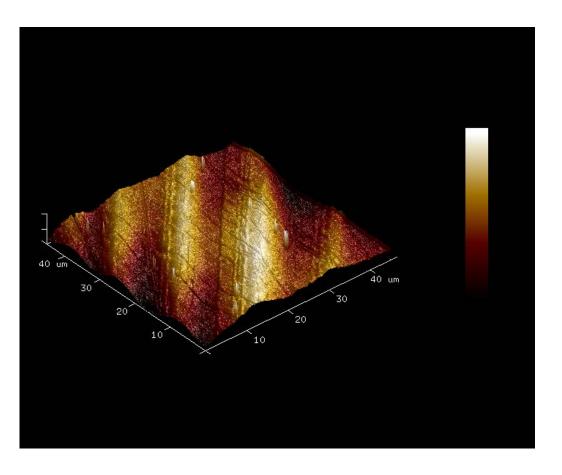
Magnetic Force Microscopy (MFM)

Our MFM studies on Ni-Mn-Ga, a magnetic shape memory alloy (MSMA), connect experimental data and computational modeling to understand the growth of twins in response to bending. MSMAs can be deformed by an applied magnetic field, or conversely can reorient their easy magnetization axis in response to an applied force via crystal twinning.



Colored areas represent different predominant magnetic domain orientations within the twins; arrows the in-plane magnetic moment vector.

Vortices occur at intersections of the twins and are believed to have both in-plane and out-of-plane components that possibly help minimize the energy of the sample.

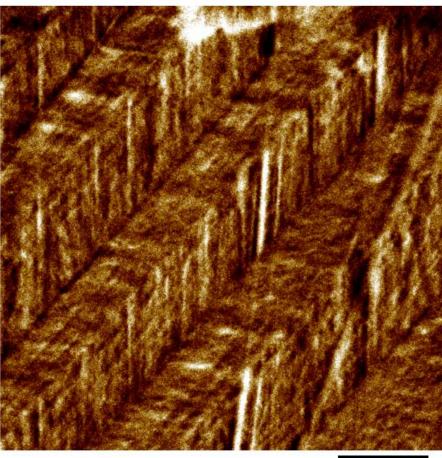


The 3D image above at left shows the topographical relief arising from the twin boundaries on a Ni-Mn-Ga sample. The 2D image at right above is the corresponding MFM phase, which provides information on the magnitude and sense of the out-of-plane magnetic moment component for the same area of the sample. The stair steps in the magnetic moment orientation predicted by the simulation (colored map above) can clearly be seen in the MFM phase data, validating the computational modeling prediction.

References:

- I. AFM schematics adapted from Bruker.
- 2. A. Kvryan, K. Livingston, C. M. Efaw, K. Knori, B. J. Jaques, P. H. Davis, D. P. Butt, & M. F. Hurley, *Metals* **6**: 91-107 (2016). doi: <u>2075-4701/6/4/91</u>
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- 4. A. Kvryan, C. M. Efaw, K. A. Higginbotham, O. O. Maryon, P. H. Davis, E. Graugnard, H. K. Trivedi, & M. F. Hurley, *Materials* **12**: 940 (2019). doi: <u>10.3390/ma12060940</u>.

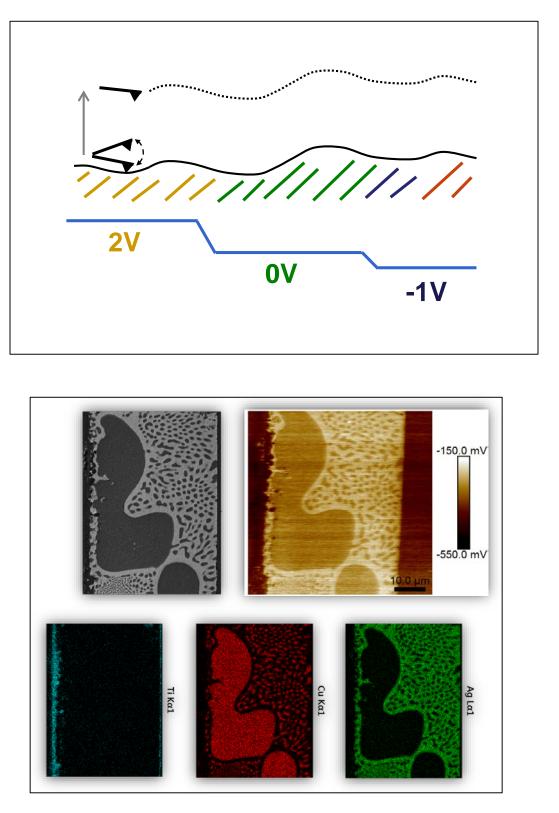
A simulation of the twin boundaries that form upon bending is shown at left.





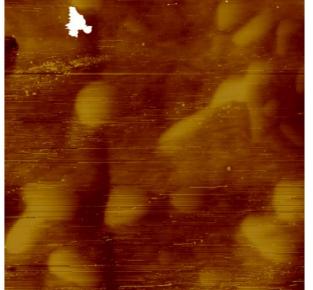
Kelvin Probe Force Microscopy (KPFM)

KPFM studies can be informative for predicting and understanding the nanoscale mechanisms underpinning galvanic corrosion initiation and propagation. Studies were conducted on a Cu-Ag braze² and a case-hardened stainless steel engineered for bearing applications in high performance jet engines destined for operation in corrosive marine environments (Pyrowear 675, or P675).⁴ KPFM revealed voltage potential differences (VPDs) between the phases present in each sample, giving insight into the thermodynamic driving force for galvanic corrosion. In the case of P675, differing carburizing treatments resulted in different corrosion behavior and resistance in 1M NaCl as seen below, where attack along grain boundaries in the carbo-nitrided (CN) sample led to etching of the matrix surrounding the carbides.

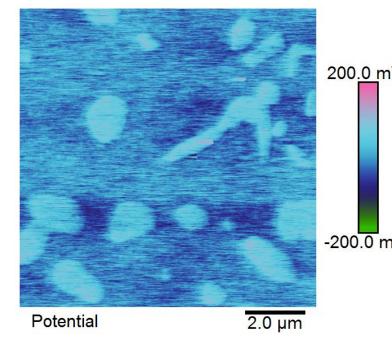


Co-localized BSE SEM (top left), KPFM (top right), and EDS maps (bottom row) of a copper-silver eutectic braze.² Phase separation within the braze leads to galvanic corrosion due to significant VPDs between the phases.

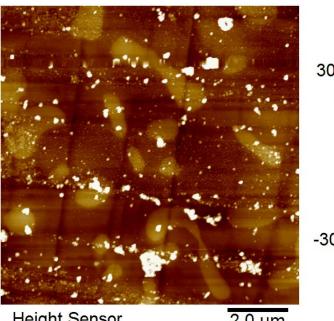
Co-Authors: Kari Higginbotham, <u>karilivingston@u.boisestate.edu</u> Medha Veligatla, <u>medhaveligatla@u.boisestate.edu</u> Armen Kvryan, <u>armenkvryan@u.boisestate.edu</u> Peter Müllner, <u>petermullner@boisestate.edu</u> Mike Hurley, <u>mikehurley@boisestate.edu</u> Paul H. Davis (mentor), pauldavis2@boisestate.edu



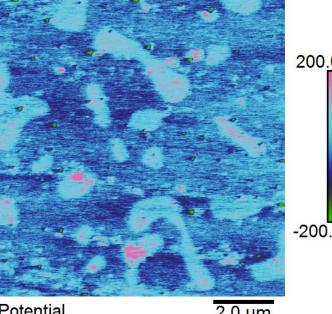


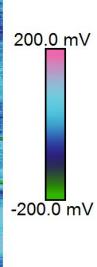


15 minutes corrosion on high temperature tempered (HTT) P675 (Left: Topography, Right: Potential).

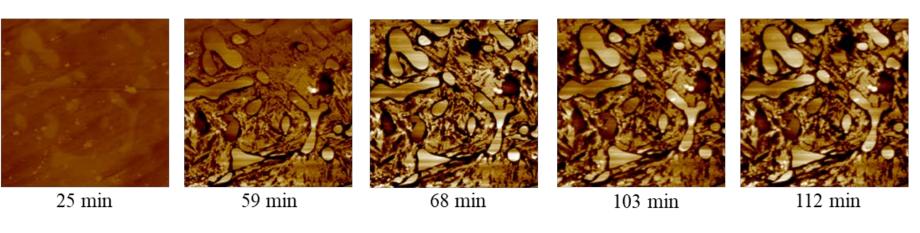








15 minutes corrosion on CN P675. (Left: Topography, Right: Potential)



Time lapse *in situ* AFM showing corrosion progression and intragranular attack on CN P675.

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