



### Characterization Needs for Manufacturable Integrated Nanosystems

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NSF



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## "One must be able to measure and quantify phenomena in order to understand and use them, which is true also for nanoscale phenomena."

Small Wonders, Endless Frontiers: Review of the National Nanotechnology Initiative, report from the National Research Council, June 2002



Founded in 2004 by Stanford and IBM research

Supported by the National Science Foundation as a Nanoscale Science and Engineering Center



## Goals:

- To develop novel probes that dramatically improve our capability to observe, manipulate, and control nanoscale objects and phenomena.
- To educate the next generation of scientists and engineers regarding the theory and practice of these probes.
- To apply these novel probes to answer fundamental questions and to shed light on technologically relevant issues.
- To disseminate our knowledge and to transfer our technology so that other research scientists and engineers can make use of our advances, and so that corporations can manufacture and market our novel probes.
- To inspire thousands of middle school students by training their teachers at a Summer Institute.

### http://cpn.stanford.edu



nature

## Nanoscale Science and Engineering Center

- Measure electronic properties at the 10 nm scale
  - Scanning Microwave Microscopy, Scanning Gate Microscopy, Novel Cantilever Design and Fabrication

## Individual Nanomagnet Characterization

- Magnetic sensitivity and spatial resolution for nm magnet characterization
  - Scanning SQUID Microscopy, Polar Kerr-effect Measurements combined with bowtie nanoantennas

## **Bio-Probes**

- Understand biological processes that take place in cell membranes
  - Bio-functionalized probe tips combined with ultrafast probe sensors
- Nanoscale Magnetic Resonance Imaging
  - Molecular structure microscope with chemical sensitivity
    - Magnetic force detection from nuclear spin flips







## spatial resolution sensitivity to quantity of interest magnetic field spin capacitance dielectric constant bandwidth invasiveness interpretability ease of use temperature range

Nanoscale Science and Engineering Cente

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Signal-to-Noise at the Nanoscale
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Less stuff => less signal
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Fewer particles
=>
more fluctuations
=>
more noise
```



## What do you want in 10 years?

# I want 1 nm spatial resolution.

I want 1  $\mu_B$  spin sensitivity.

Giwalk

I want 1 ns time resolution.

I want it all at once at room temperature next to a real production line.





## **Scanning SQUID Microscopy**



Moler Group

## **Magnetic Measurements**





Superconducting Quantum Interference Device (SQUID)



#### Design

- Produced by 12-layer optical lithography process
- Pickup loops as small as 500 nm defined by FIB
- Terraced tips allow positioning of pickup loop 300 nm from sample

### Performance demonstrated

- Spin sensitivity < 100  $\mu_B$ /rt-Hz





#### Operate as Magnetometer without field coil

- DC measurement of flux captured by pickup loop.



## Scanning SQUID Microscopy - Susceptometry e Science and Engineering Center Operate as Susceptometer with field coil Response in the pickup loop to AC current in the field coil Pick up loop reads the field applied by the field high above SQUID field coil M=Susceptibility signal **Susceptibility** SQUID pickup coil approach curve in contact Sensor-sample distance Superconductor Hicks, et al., PRL 103 (2009)

Hicks, *et al., PRL* **103** (2009) Kalisky, *et al., PRB* **81** (2010) Luan, *et al., PRB* **81** (2010)



Magnetotactic bacteria

### Magnetometry image



SEM image





Distribution of Magnetic Moments In Populations of Magnetotactic Bacteria







## **Nanoscale MRI**



Rugar Group (IBM/Stanford)



#### The Quest for a Molecular Structure Microscope



- Molecular structure is key to understanding function
- Most proteins in your body have no known structure due to limitations of current techniques (radiation damage, crystallization...)
- Can a microscope be built that can directly image the 3D structure of molecules?

#### Extend Magnetic Resonance Imaging (MRI) to the Nanoscale



- Features
  - True 3D imaging, chemically selective, non-destructive

#### But ...

• Conventional MRI requires  $10^{12}$  -  $10^{18}$  nuclear spins per voxel and is limited to resolution of (3  $\mu$ m)<sup>3</sup>

#### Interferometer laser beam

Ultrasensitive cantilever

# Resonant slice (B = 2.70 T)

- <sup>1</sup>H Nuclear spin ( $\gamma$  = 42.6 MHz / Tesla)

N Magnetic tip

S

Microwire generating 115 MHz magnetic field



## Nanoscale MRI Schematic





## First MRFM Images of a Biological Sample

## **Tobacco Mosaic Virus**

100 nm thick shaft

1 μm

1 µm thick mass loading

TMV particle size:

18 nm diameter Up to 300 nm long



C. L. Degen et al., Proc. Natl. Acad. Sci USA 106, 1313 (2009)



## **3D** reconstruction of proton density



## Detail from one horizontal slice



Combined ultrasensitive MRFM detection with 3D image reconstruction delivers < 10 nm voxel resolution

100 millionfold improvement in volume resolution over conventional MRI

Elementally selective imaging

No radiation damage

Cross-section - showing depth resolution



C. L. Degen et al., Proc. Natl. Acad. Sci 106, 1313 (2009)

# **CPN** Spin Measurements - Nitrogen-Vacancy Center

Small changes in sample's magnetic field gives rise to variations in fluorescence intensity

Technique can work under ambient conditions (no cryo, no vacuum)



Theoretical estimates suggest that NV centers will enable imaging of both magnetic fields and spins at room temperature comparable to what MRFM and SQUID microscopy can do at low temperature.



## Nanoscale Microwave Impedance Microscopy



### Keji Lai and Michael Kelly, Zhi-Xun Shen Group





## Microwave impedance microscopy (MIM)

*Why*... For microwave frequency:

- Big contrast between metal & insulator
- f >> DC, therefore no electrodes required
- $f \ll 0$  optic, energy small, therefore no inter-band transitions



# Commercial atomic-force microscope



Customized cryogenic / magnet system







## Sub-surface imaging

 a layer of Al<sub>2</sub>O<sub>3</sub> is sputtered onto a SiO<sub>2</sub> sample and then the surface is polished





K. Lai et al., Rev. Sci. Instrum. 78 (2007)



### Electronic Inhomogeneity in Phase-change In<sub>2</sub>Se<sub>3</sub>





#### K. Lai et al., Nano Lett. 9 (2009)

# Graphene derived from various sources



Annealed graphene sheets, chemically derived from graphite



W. Kundhikanjana et al., Nano Lett. 9 (2009)



### Colossal Magnetoresistance Nd<sub>1/2</sub>Sr<sub>1/2</sub>MnO<sub>3</sub> Phase Transitions

# Temperature-driven phase transitions

- T<sub>curie</sub> ~ 250K: paramagnetic to ferromagnetic
- $T_{coo} \sim 160$ K: charge/orbital-order

- Magnetic field driven phase transition below  $T_{coo}$ 
  - From antiferromagnetic COO insulating to ferromagnetic metallic
  - Hysteresis below T<sub>coo</sub>



![](_page_26_Picture_0.jpeg)

![](_page_26_Picture_1.jpeg)

![](_page_26_Figure_2.jpeg)

![](_page_27_Picture_0.jpeg)

A

## Motivation

An NSF Nanoscale Science and Engineering Center

 Create an access port to study cell activity and transport mechanisms through cell membranes

## Technology

 Use a bio-functionalized probe tip to mimic membrane proteins and insert into the cell membrane's lipid bi-layer

![](_page_27_Figure_5.jpeg)

![](_page_27_Picture_6.jpeg)

B

B.D. Almquist and N.A. Melosh, PNAS. 107 (2010)

![](_page_28_Picture_0.jpeg)

Sensitivity, Resolution ...

- Continued research into new measurement techniques
- Improvements of current techniques to push spatial and temporal resolution, sensitivity, ...

### Multi-modality characterization

 Simplified construction and advances in nanofabrication are enabling characterization techniques that can probe several physical properties at the same time in one instrument, especially important for integrated nanosystems

Ease of Use

Synergy between characterization research and design & manufacture of integrated nanosystems

![](_page_29_Picture_0.jpeg)