Designing Piezoelectric Films for MEMS: Using Local Measurements

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funding provided by an NSSEFF fellowship, NSF, and the Center for Dielectric Studies

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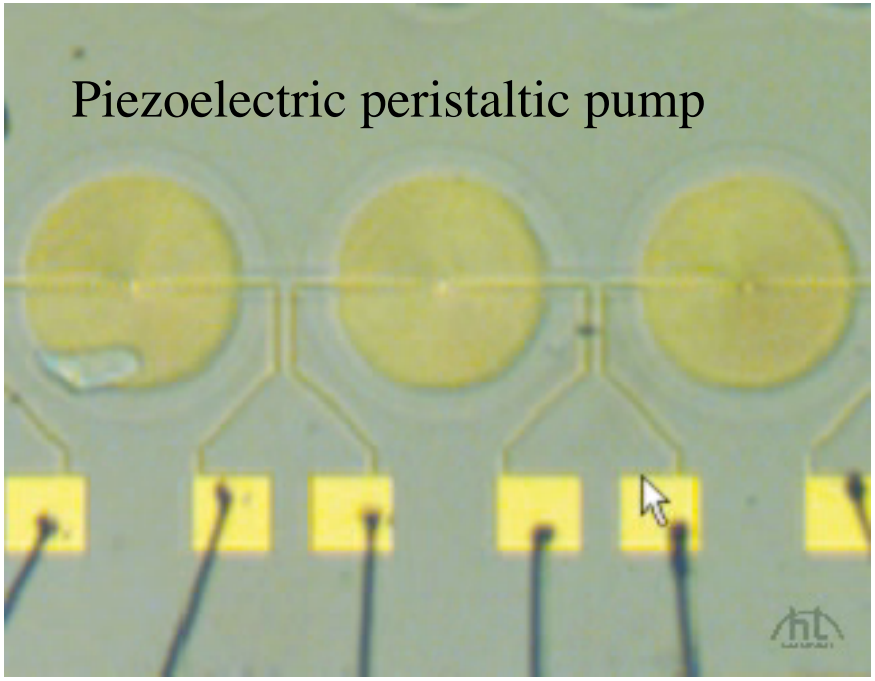
Outline

- Motivation for Piezoelectric MEMS
- Optimizing Piezoelectric Responses for Actuators
- Local Measurements of Piezoelectric Response
- Conclusions



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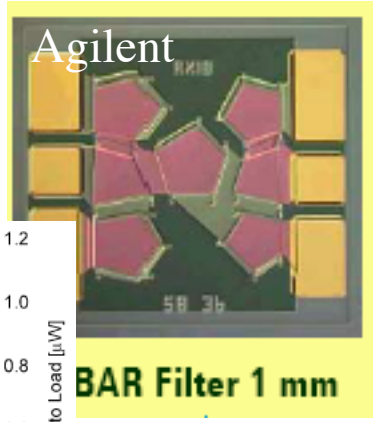
Piezoelectric MEMS

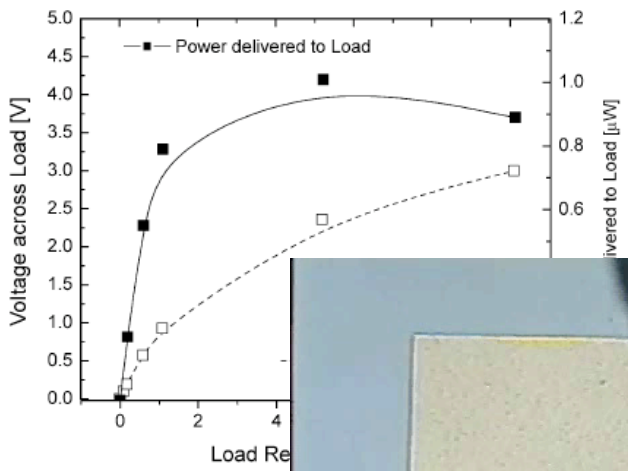
FBAR Resonators: High frequency filters, sensors



Sensors: High sensitivity sensors

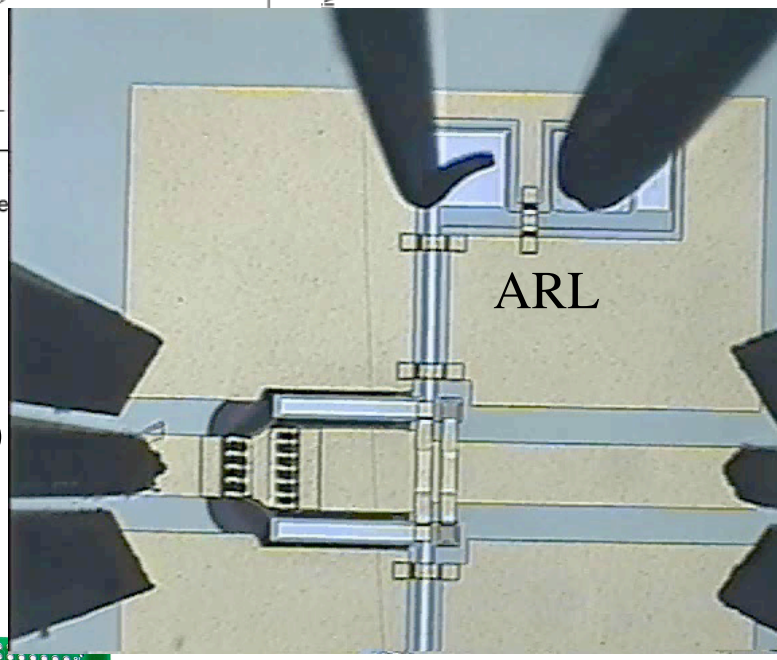
Sensors with Energy Harvesting

Jeon et al., Sens. Act. A, 2005:

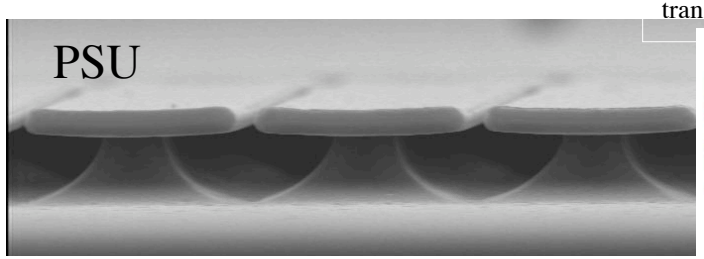


Actuators: Low power

Low voltage (CMOS compatible)

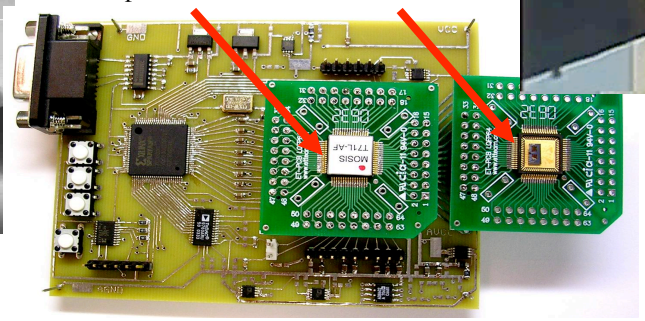


Transducers (e.g biomedical, acoustic microscopy)



CMOS transceiver chip

The xylophone transducer



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Selecting Piezoelectrics for MEMS

$$D_3 = \varepsilon_o \varepsilon_{33,f} E_3 + e_{31,f}(x_1 + x_2) + d_{33,f}\sigma_3$$

$e_{31,f}$ relates

- induced out-of-plane polarization to in-plane strain
- induced in-plane stress to out-of-plane electric field

Sensors

- High e_{ijk}/ε_r for voltage-based sensors
- High e_{ijk} for charge-based measurements
- High signal to noise ratio

$$\frac{e_{ijk}}{\sqrt{\varepsilon_o \varepsilon_{ij} \tan\delta}}$$

Actuators

- High e_{ijk}
- High drive field capability
- High T_{tr}

Transducers

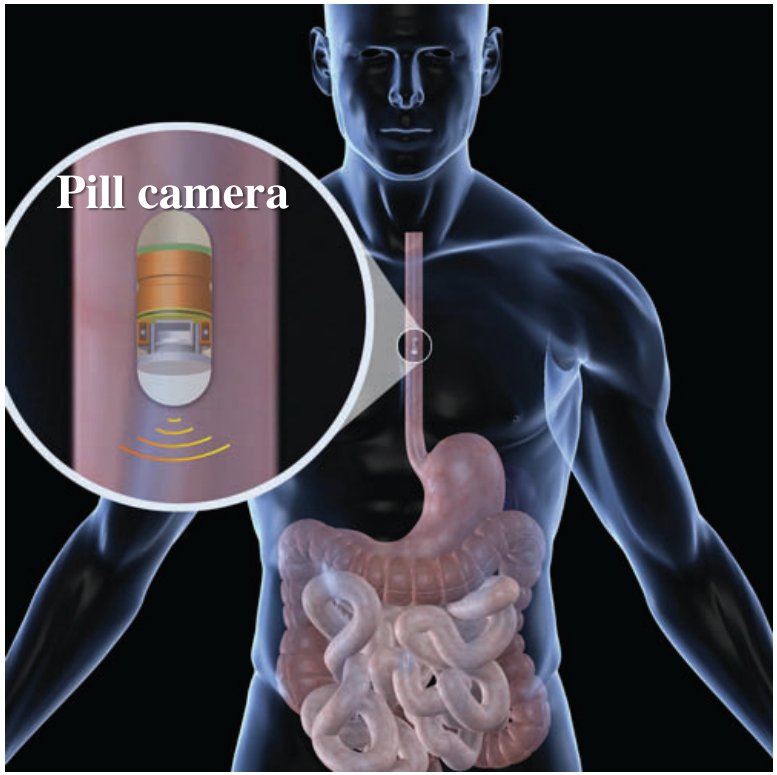
- High e_{ijk}
- High coupling coefficients (bandwidth)

Energy Harvesting

- High $k^2 \propto e_{ijk} h_{ijk}$
- Low mechanical losses

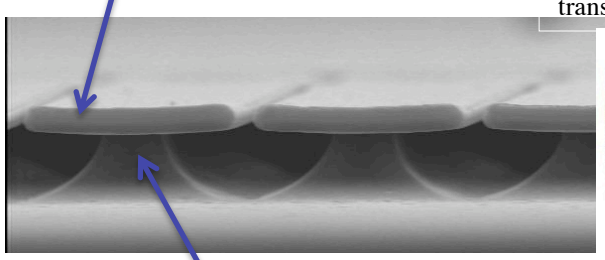
	ZnO	AlN	PZT 52/48
$\varepsilon_{33,f}$	8 - 12	10.4	900 - 1300
$d_{33,f}$ (pC/N)	10 - 12	3.4 - 3.9	90 - 110
$e_{31,f}$ (C/m^2)	-0.43 to -0.8	-0.9 to -1.0	-6 to -18
$k^2_{p,f}$	0.06 -0.085	0.065 - 0.11	0.1-0.20

Miniature Ultrasound Systems with Close-Coupled Electronics



- Ultrasound pill camera with close-coupled electronics for sub-surface, high resolution imaging of features < 100 μm in size
- Diagnosis for ailments in GI tract, including Barrett's esophagus, as well as liver diseases
- Large piezoelectric coefficients are required to generate adequate sound pressure

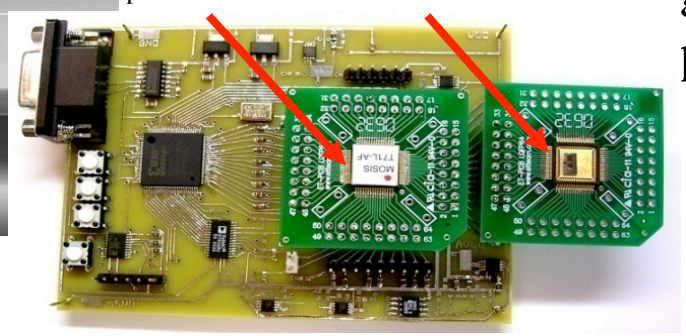
PZT coated xylophone



Si

CMOS transceiver chip

The xylophone transducer



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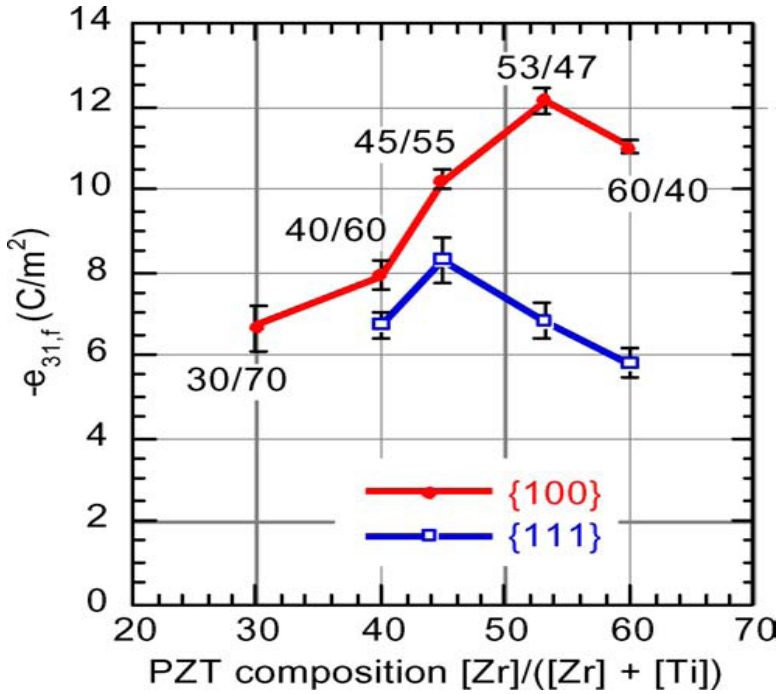
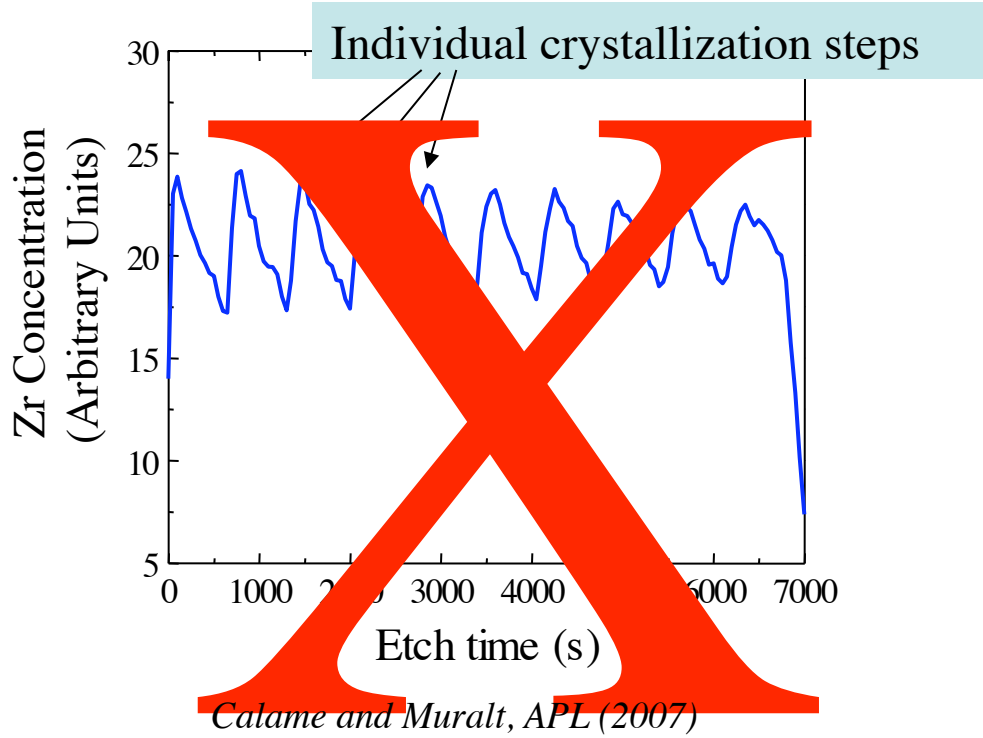
PZT Film Piezoelectric Response

Optimized response achieved in gradient-free, oriented PZT



N. Lederman et al., Sens. Act., 105(2002)



Calame and Muralt, APL (2007)

$e_{31,f} = -6$ C/m^2 → -12 C/m^2 → -18 C/m^2

{001} orientation Reduced Zr/Ti gradients

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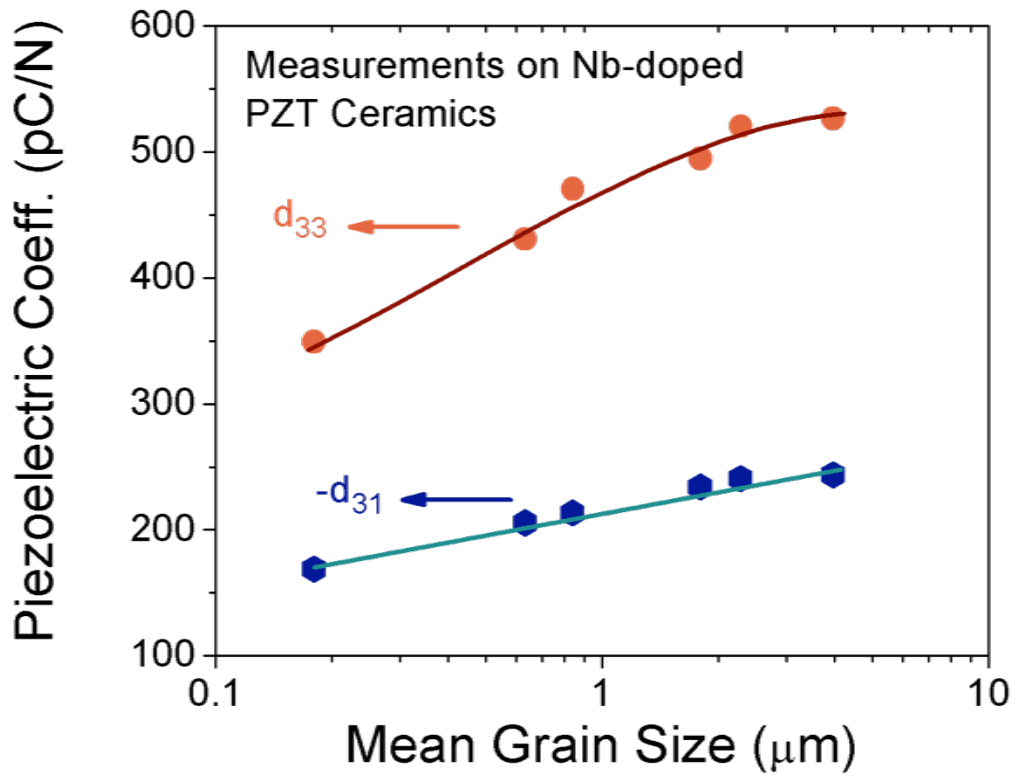
Grain Size in PZT Ceramics

- Small grained PZT ceramics show a diminution in piezoelectric properties with grain size associated with changes in domain structure and domain wall mobility
- What about grain size in films?



Randall *et al.* J. Am. Ceram. Soc. (1998)

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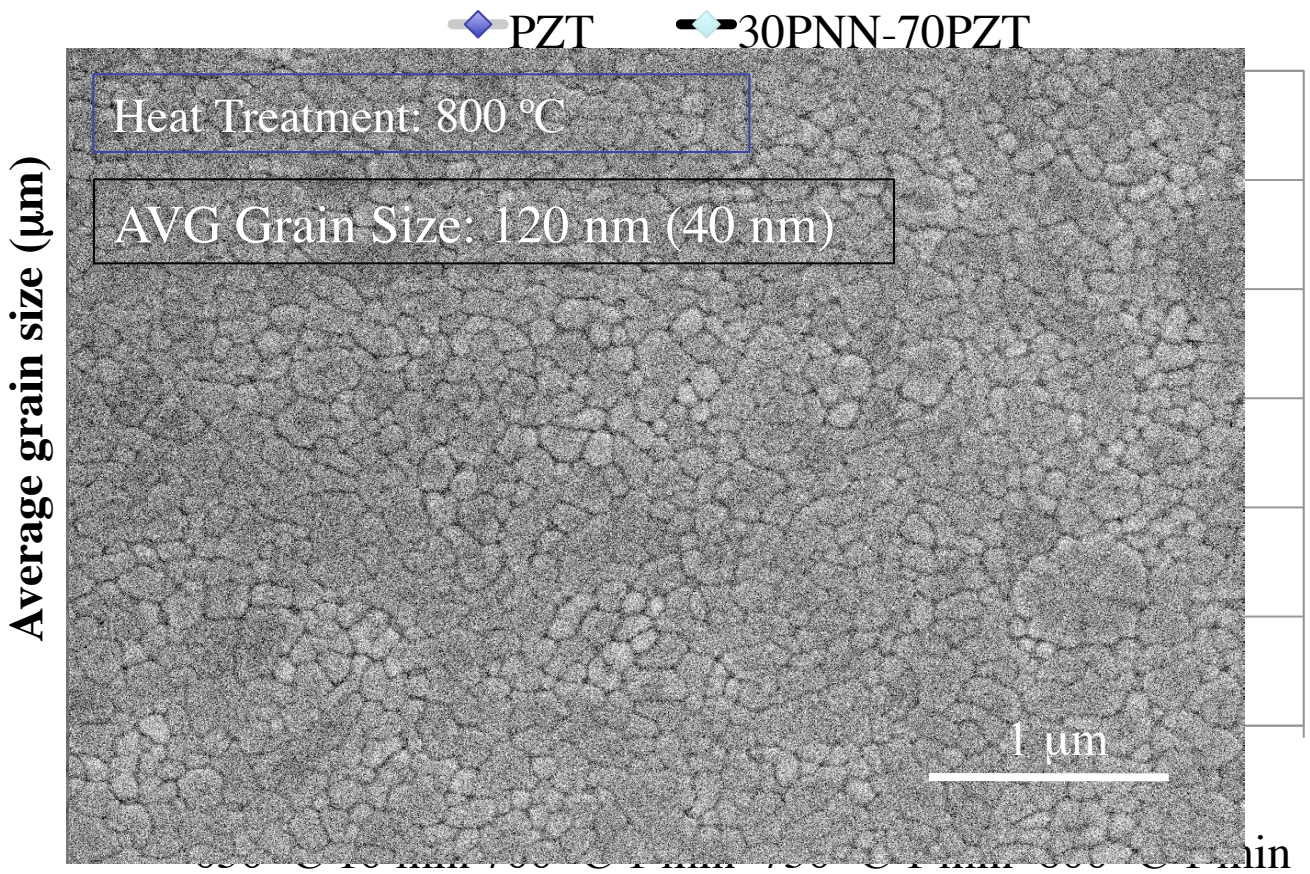
Importance of PNN Fluxing

Sample compositions:

- $Pb(Zr_{0.45}Ti_{0.55})O_3$
- $30PbNi_{1/3}Nb_{2/3}O_3$-$70Pb(Zr_{0.45}Ti_{0.55})O_3$
- 10 mol % Pb excess

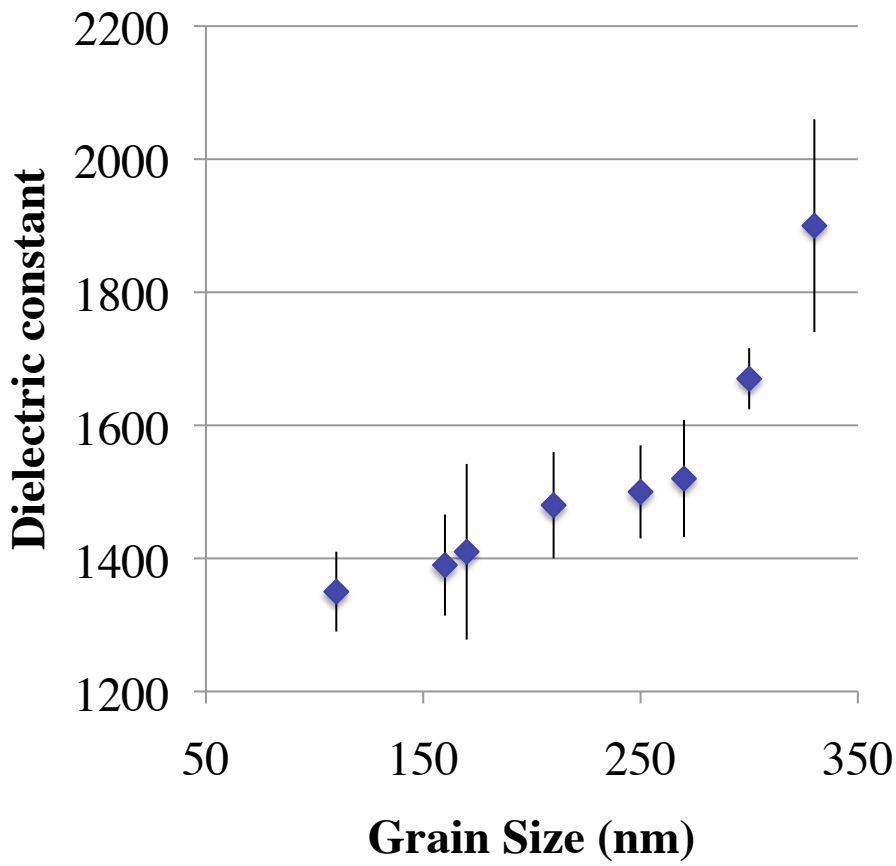
Sample preparation:

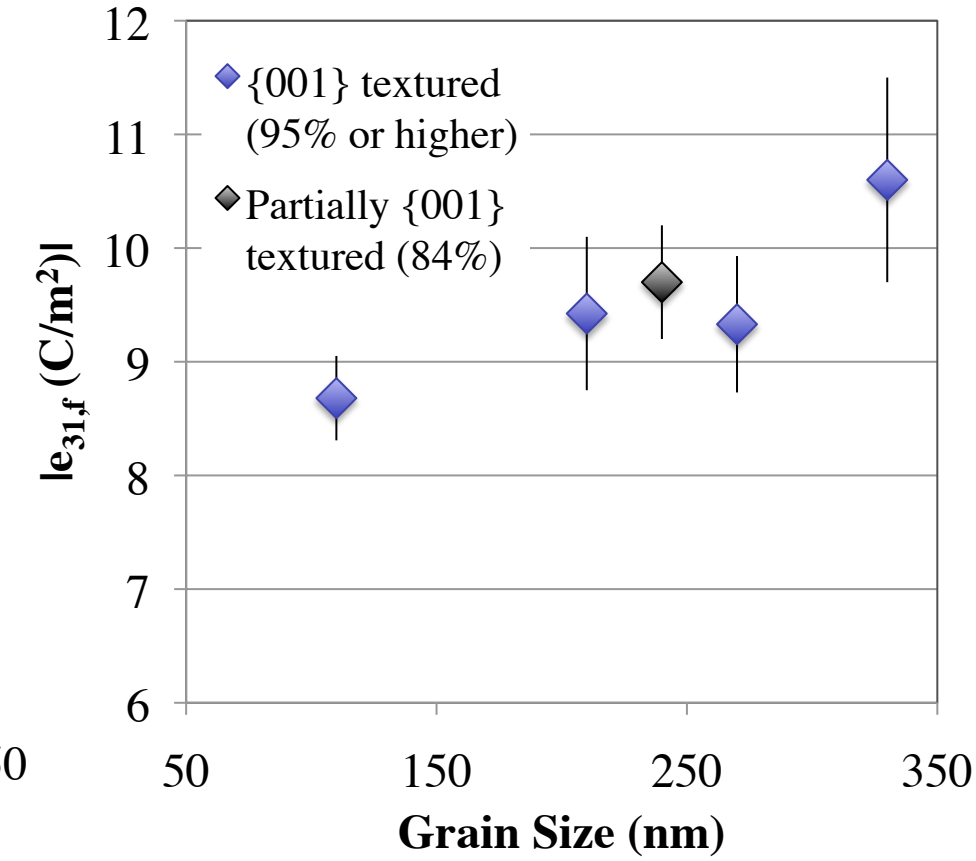
- 4 pyrolyzed layers
- 1 annealing step
  - 650 ºC 10 min
  - 700 ºC 1 min
  - 750 ºC 1 min
  - 800 ºC 1 min



The presence of PNN enhances mass transport and promotes grain growth

Grain-size Effects in PNN-PZT Thin Films



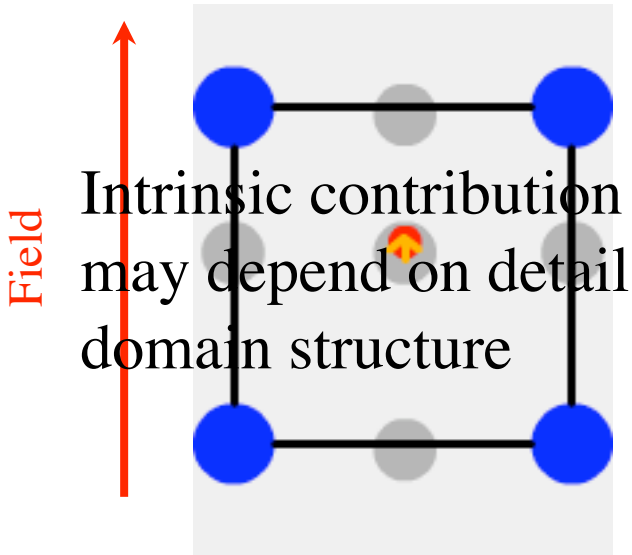


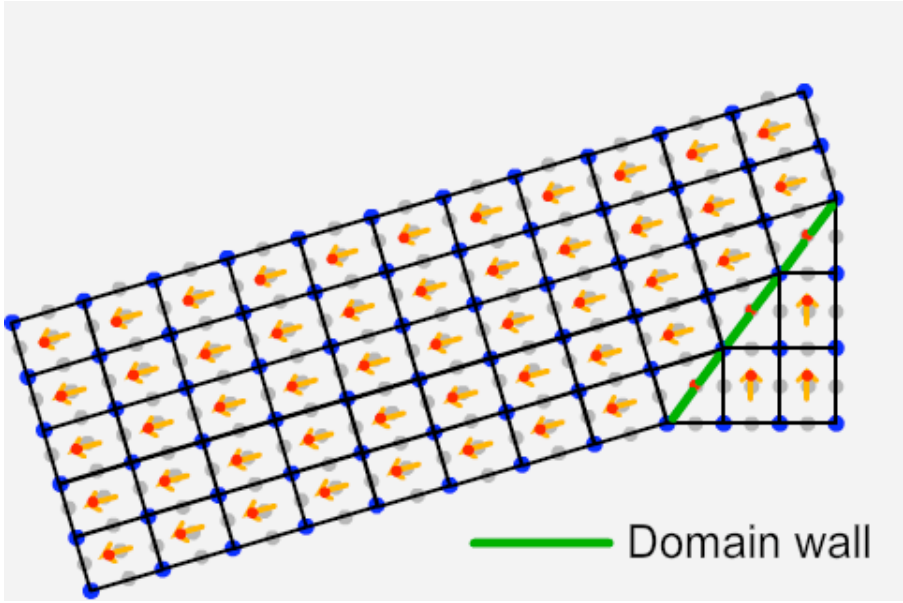
- Larger grain-size films showed improved dielectric and piezoelectric properties for films of comparable thicknesses
- Partially oriented sample seemed to obey the $e_{31,f}$ vs. grain-size trend

Why Does Grain Size Matter?

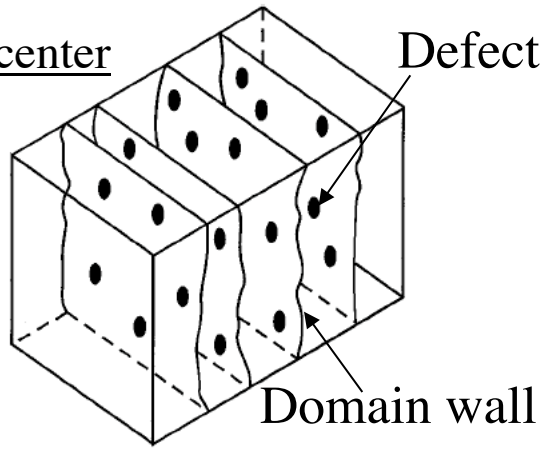
Intrinsic – deformation of lattice

Extrinsic – domain wall or phase boundary motion

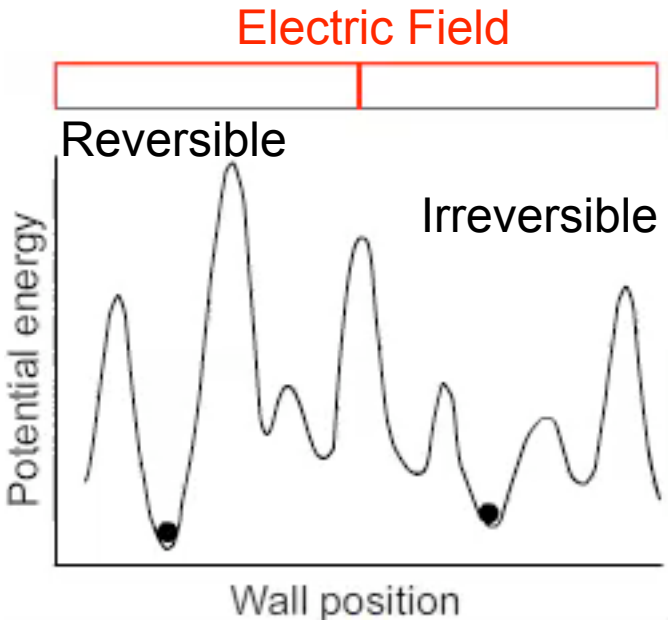




Defect: pinning center



D. Damjanovic, *NATO Science Series 3. High Technology* **76** (2000) 123



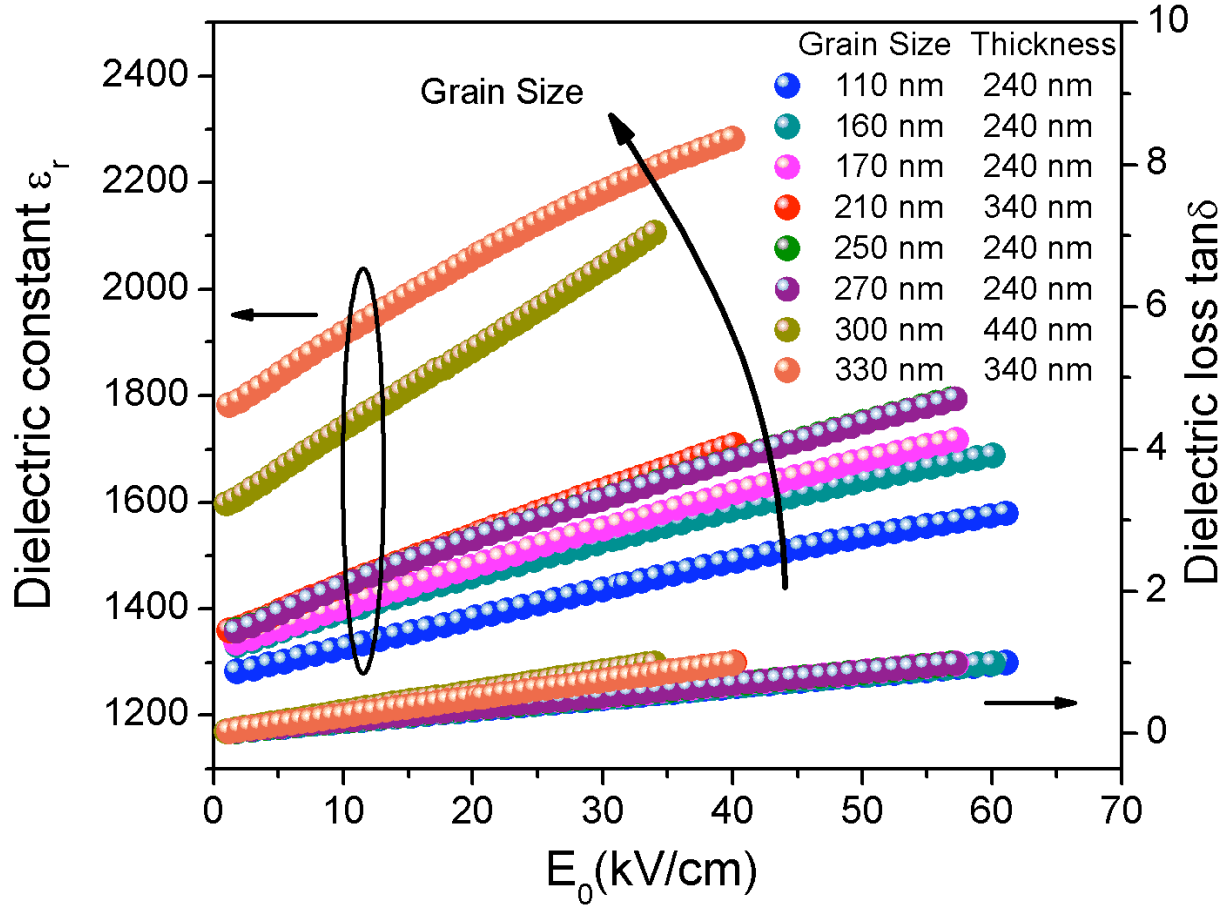
Nonlinearities in PNN-PZT Thin Films

- Distinct region where ε_r fits the Rayleigh relation: $\varepsilon'(E_0)=\varepsilon'_{init}+\alpha' E_0$

where:

α' irreversible Rayleigh coefficient

ε'_{init} intrinsic dielectric response + contribution from reversible domain wall vibration



- The ratio $\alpha'/\varepsilon'_{init}$ increa
and 0.0084 cm/kV wit
- This suggests a considerable increase in the domain wall (extrinsic) contributions to the dielectric response with grain size and thickness

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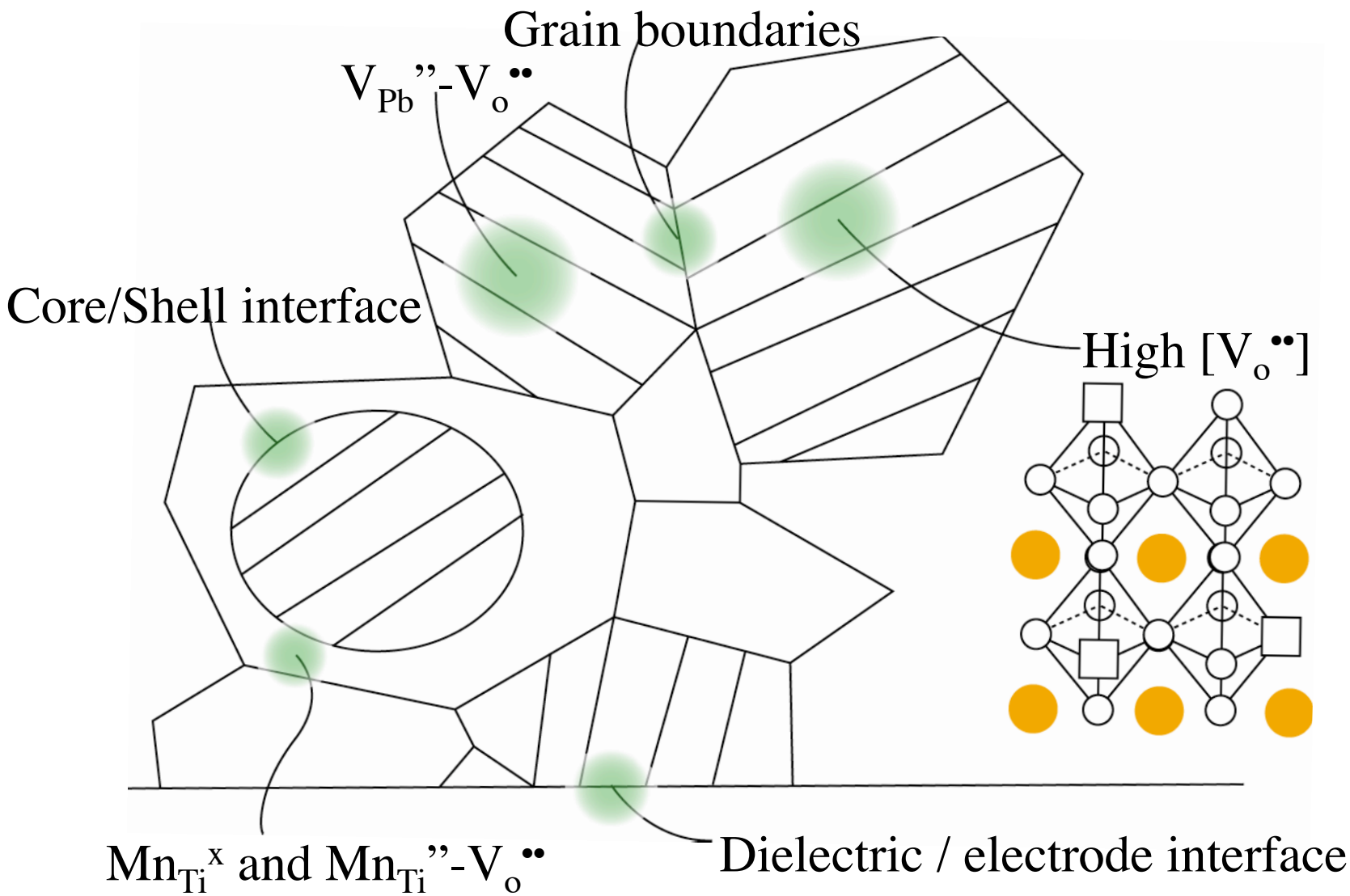
Typical Pinning Sources in Ferroelectrics



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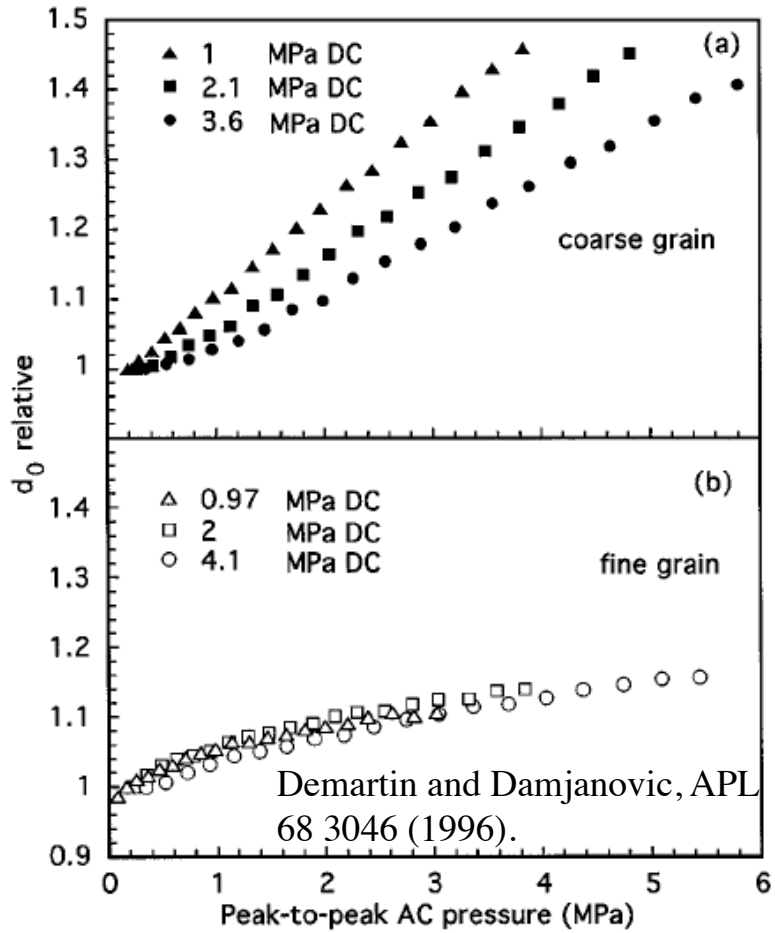
Factors Affecting Wall Mobility

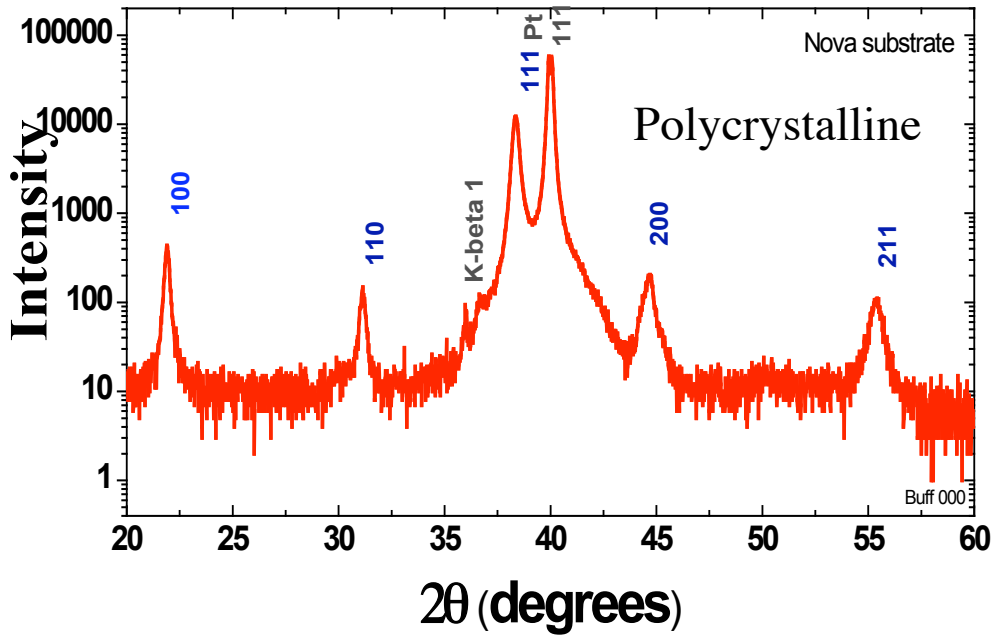
- Defect chemistry
- Tilt domain structures
- Temperature
- Applied bias
- Dislocations
- Phase boundaries
- Grain boundaries
- Domain wall density
- Residual stresses

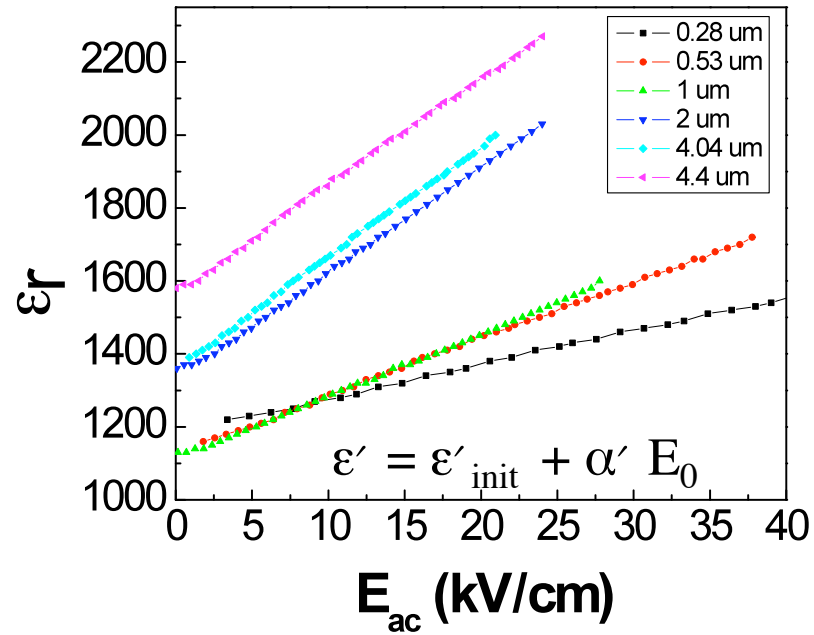


Demartin and Damjanovic, APL 68 3046 (1996).

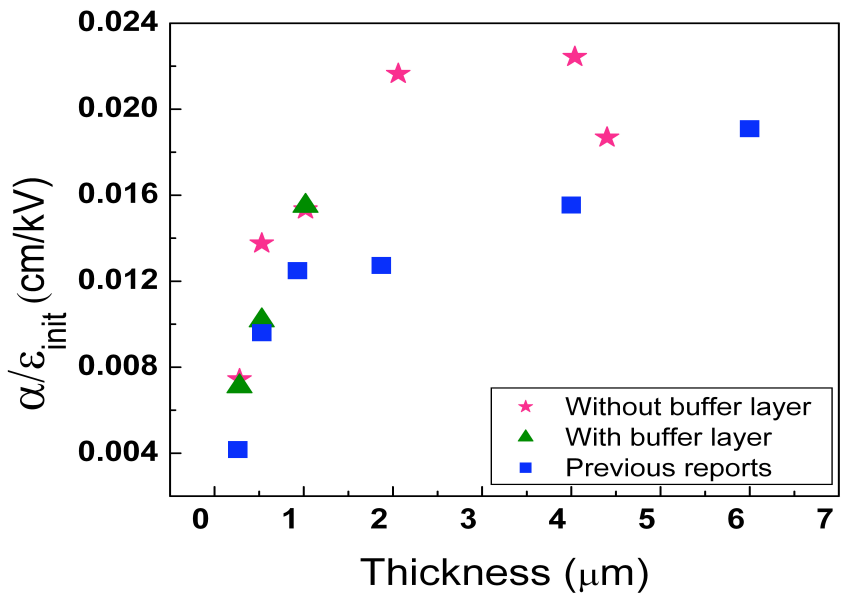
Can we begin to understand the relative importance of each of these factors? Can local measurements help?

Model System: CSD PZT 52/48 Films





- At higher ac field, extrinsic contributions become more prominent, increasing the measured ε_r.
- There is some thickness dependence to dielectric nonlinearity measured using large area electrodes



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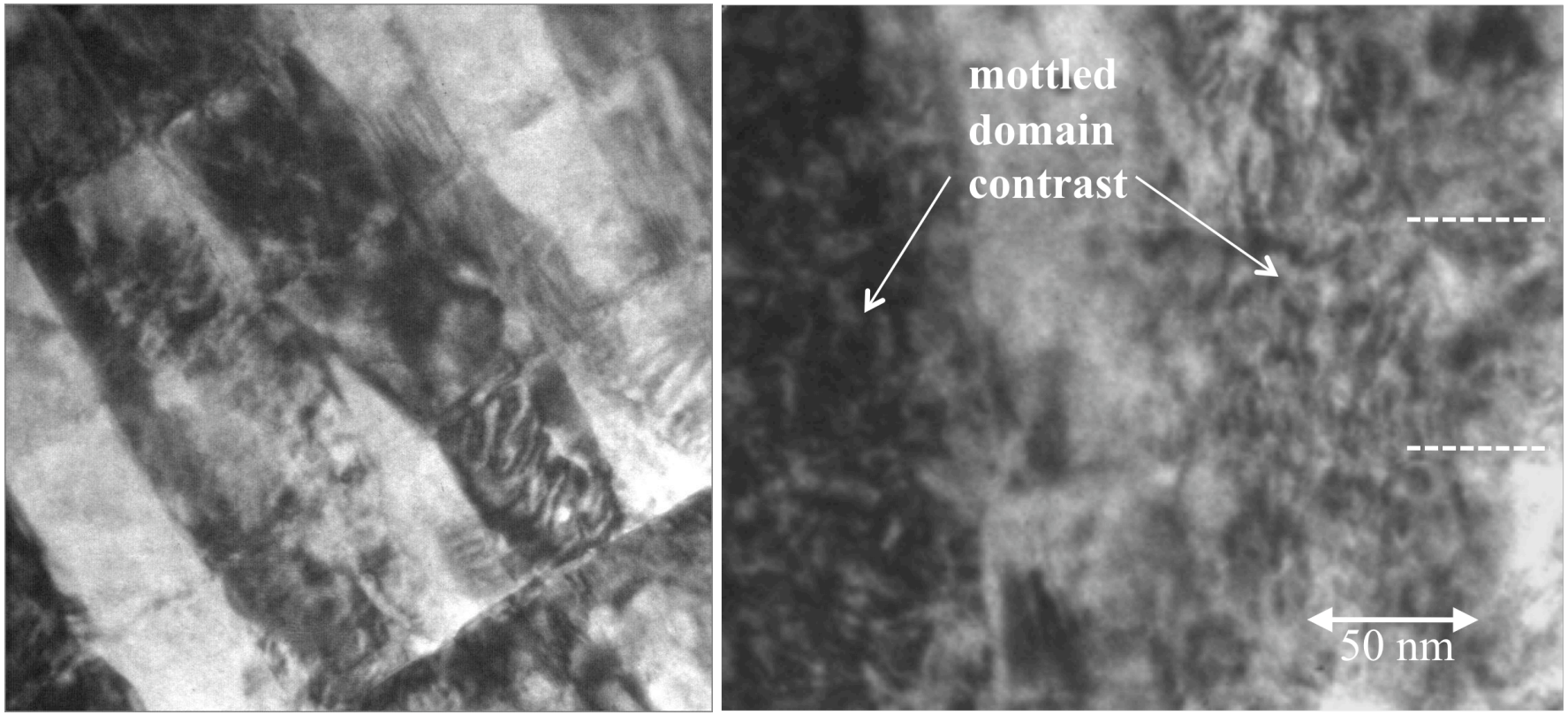
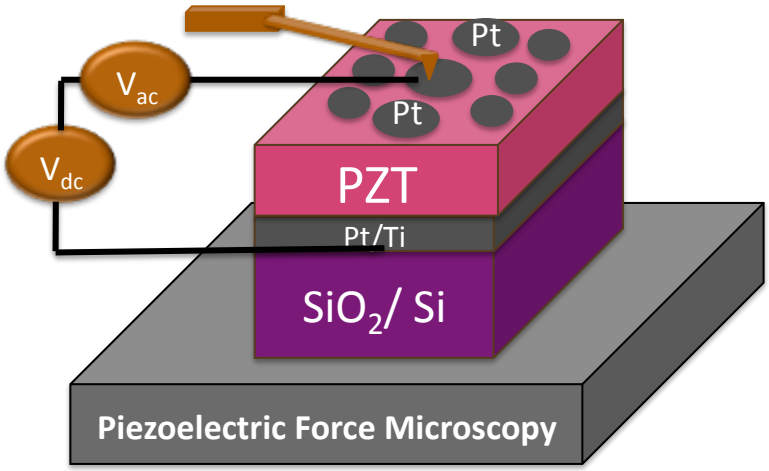
Microstructure of PZT 52/48 Films



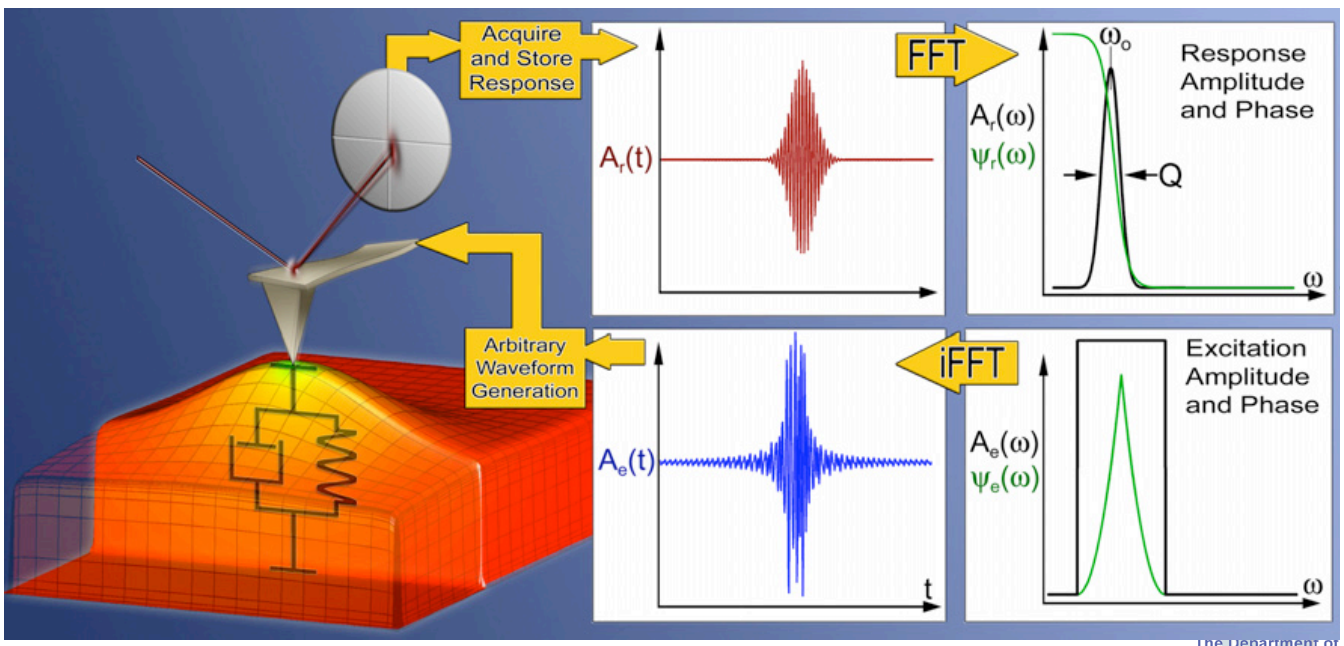
- Microstructures are predominantly columnar
- Planar defects associated with each crystallization step are visible
- Mottled contrast at very fine length scale is associated with complex ferroelectric domain structure

Band Excitation Piezoelectric Force Microscopy



$$d_{33} = \frac{Amplitude\ Signal * Invol}{V_{ac}}$$

$$x_{ij} = d_{kij} E_k$$



Jesse, et al., Nanotechnology, 18 (2007)

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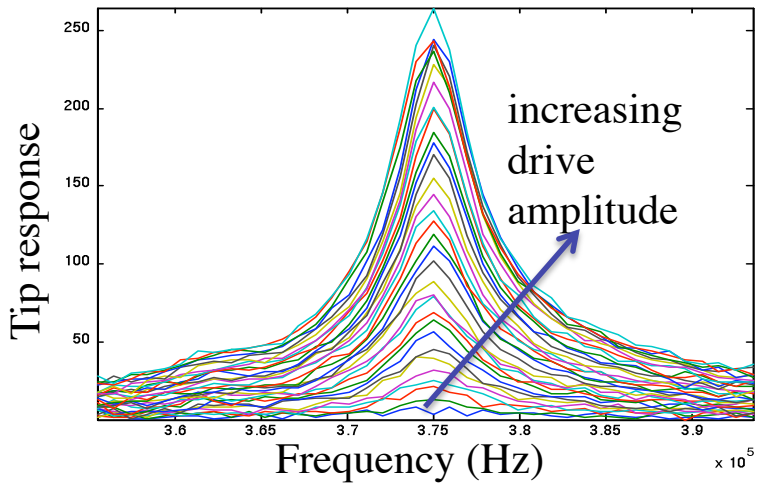
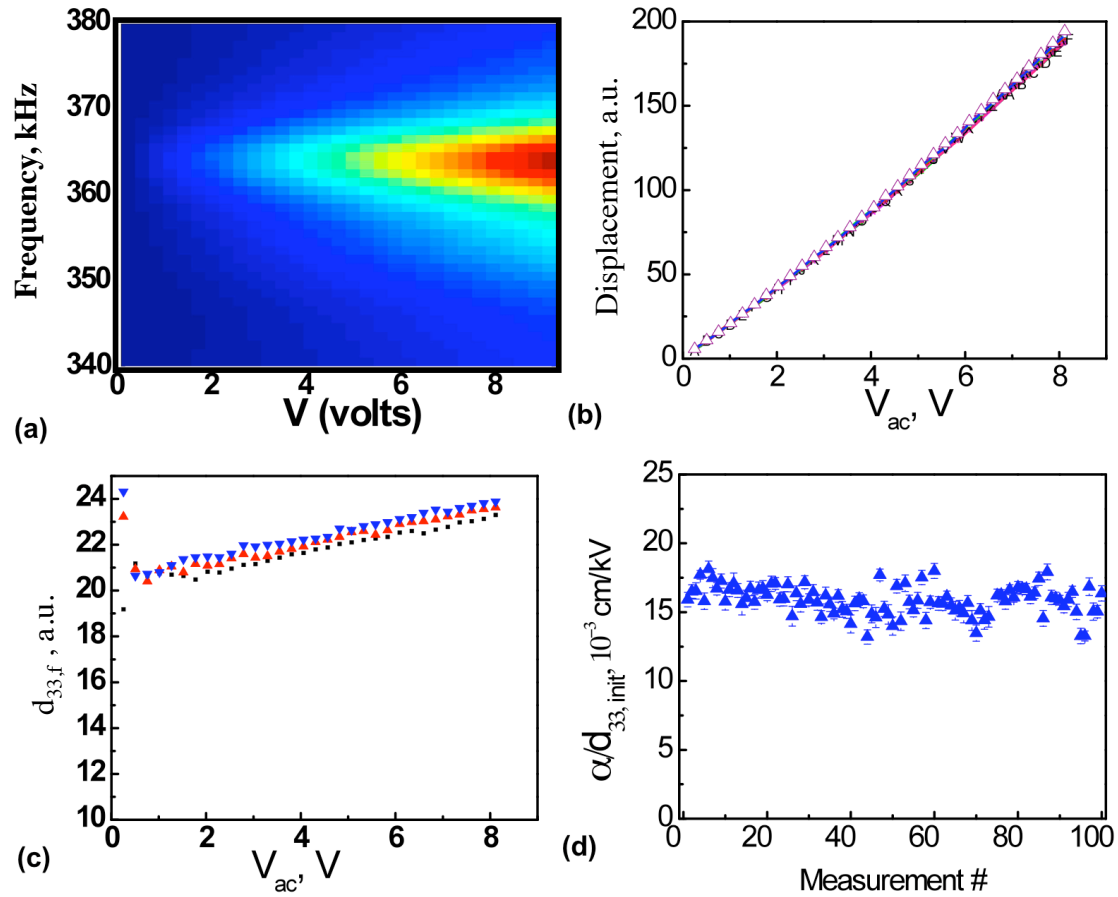
Local Mapping of Rayleigh Parameters





- Band excitation PFM used to improve signal to noise ratio
- Cantilever sent into resonance
- Conditions where tip contact nonlinearity is modest chosen to measure displacements

Bintachitt et al., PNAS (2010)

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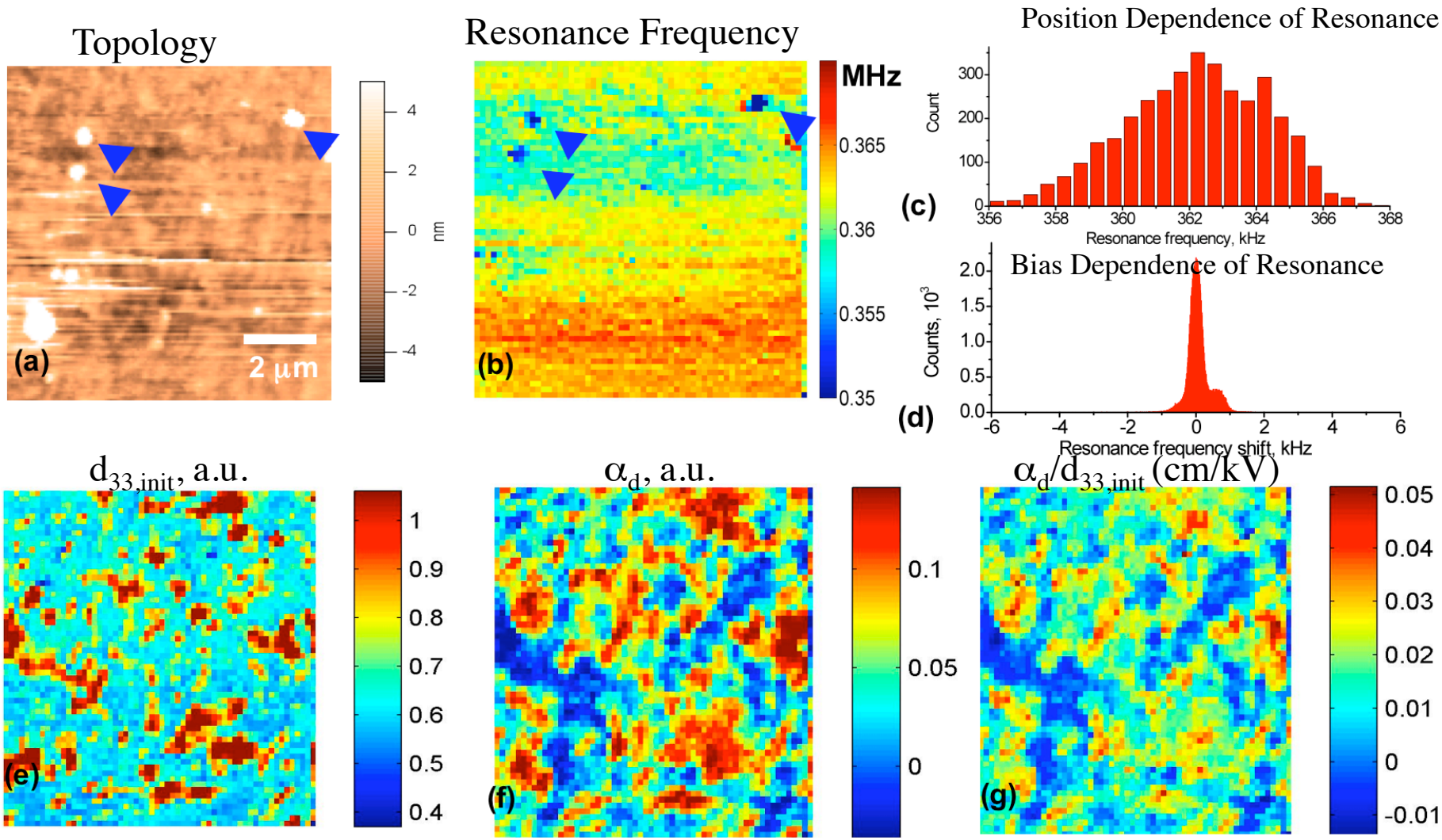
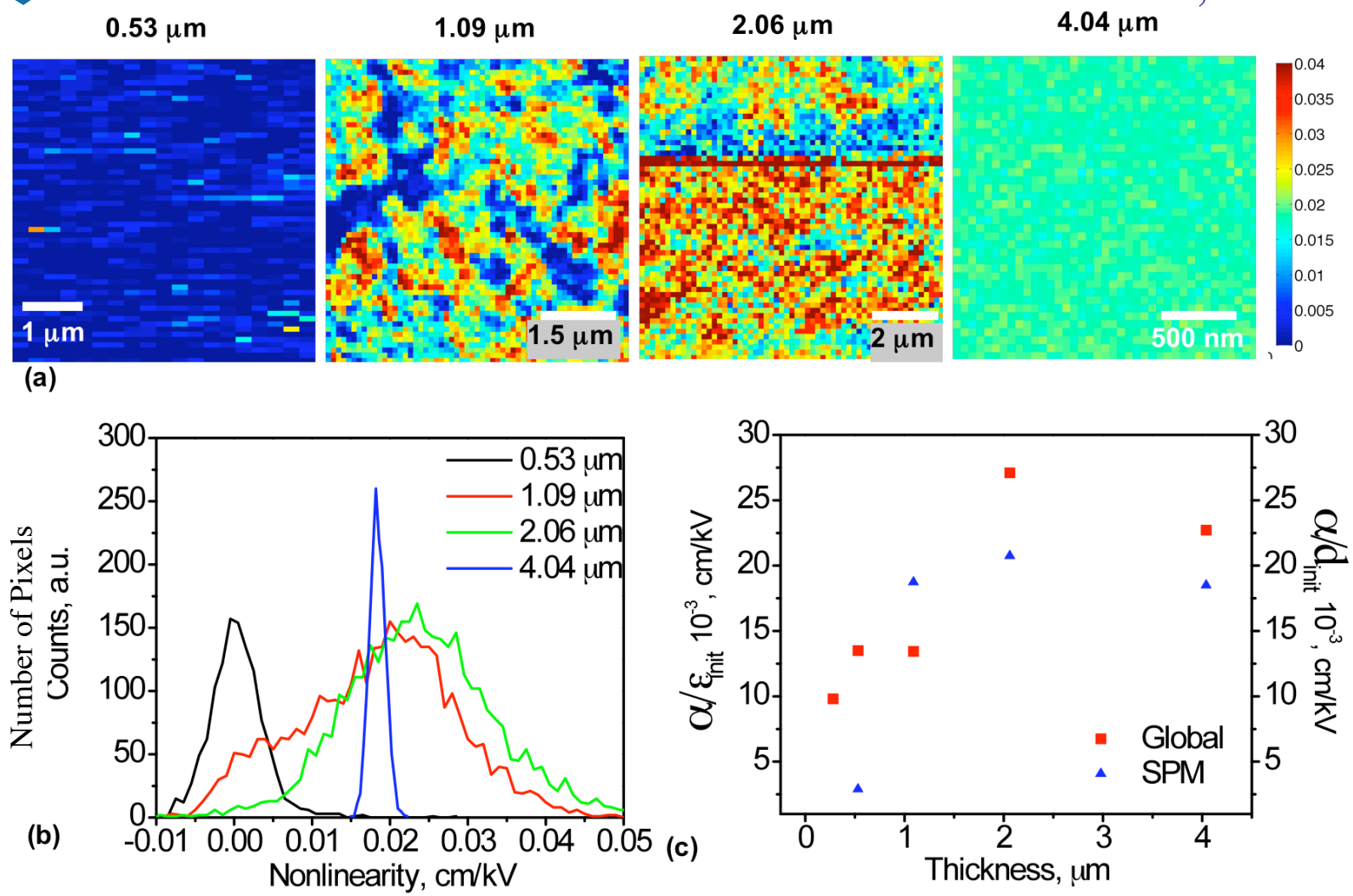
Mapping Rayleigh Response, 1.09 μm PZT Film



- In dielectric data, $\alpha/\varepsilon_{init} \sim 0.0155$ cm/kV, similar to average behavior in film
- Not all of the features in $\alpha/d_{33,init}$ are associated with topology

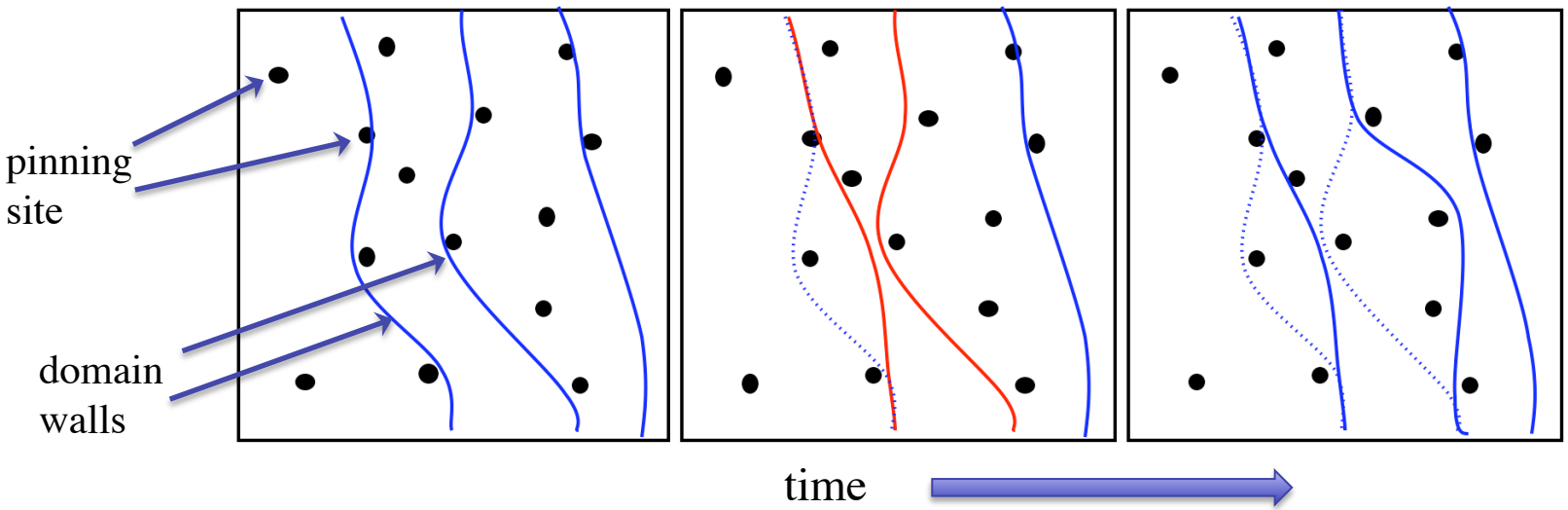
PENNSTATE 1855
MATSE The Department of Materials Science and Engineering

Thickness Evolution of $\alpha_d/d_{33,init}$



- Nonlinearity evolves through clusters, which ultimately merge to produce homogeneous response

Why Clusters? Why This Length Scale?

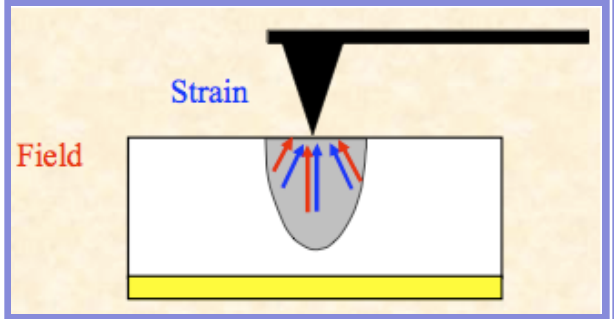
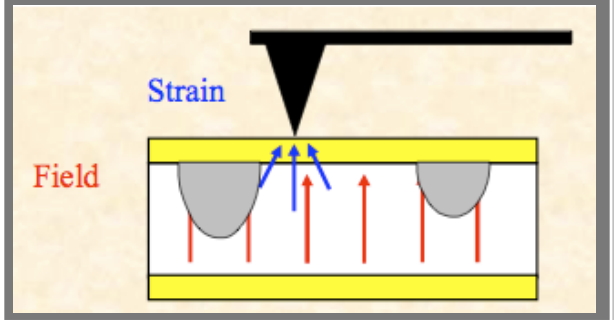


- Equilibrium domain densities are governed by energy balance
- Motion of one domain wall leads to collective motion of other walls, yielding clusters over some finite interaction volume via long-range elastic and electric fields
- Presumably the limiting speed of interaction will be the speed of sound
- Speed of sound x dielectric relaxation time for domain walls ~ 4 -5 μm for PZT
- Is this the link that controls length scale?

Bintachitt et al., PNAS (2010)

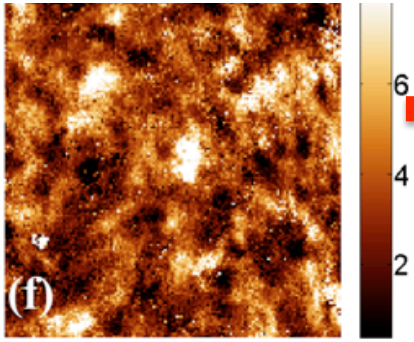
Other Evidence of Correlated Motion of Domain Walls in Similar Films?

Full-Scale Switching Studies: PFM With and Without a Top Electrode

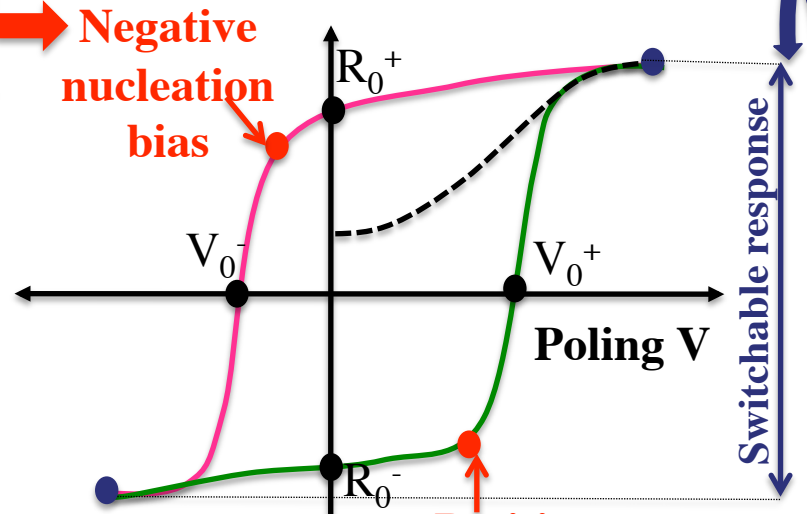
Method	Advantages	Drawbacks
PFM without a top electrode 	• High lateral resolution • Correlation between domain patterns and microstructure • Analysis of domain wall structure and its interaction with microstructural features	• Highly inhomogeneous electric field • Possible effect of surface contamination • Difficult to quantify absolute magnitudes of piezoelectric response • Asymmetric boundary conditions
PFM with a top electrode 	•Homogeneous electric field •No electrostatic contribution •Quantitative characterization	• Detrimental effect of top electrode on imaging resolution • Wafer flexure influences magnitude of apparent piezoelectric responses

Kalinin S., PFM Workshop, Oak Ridge Nat. Lab. (2007)
Gruverman, PFM Workshop, Oak Ridge Nat. Lab. (2007)

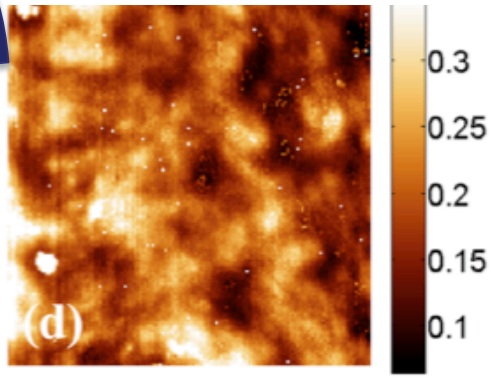
Local Measurement by SSPFM



Piezoelectric response

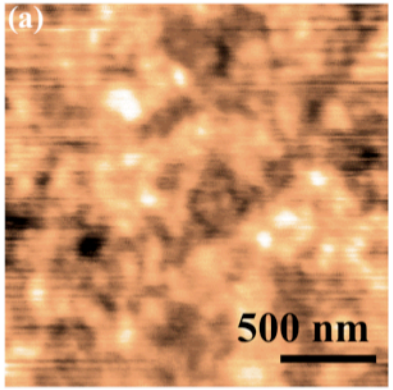


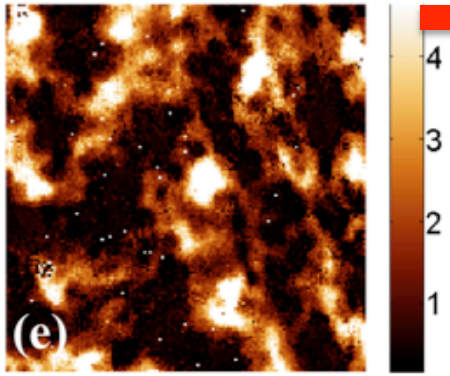
Switchable response



Switchable response

Topography





after Jesse et al., Rev. Sci. Instrum. 77 073702 (2006)

- ✧ Piezoelectric measurements made at zero bias after excitation with a dc bias to "pole"
- ✧ The features in the positive nucleation bias map are ~200 nm
- ✧ The feature size of topography is 50 nm
- ✧ The switchable responses are independent of the surface morphology

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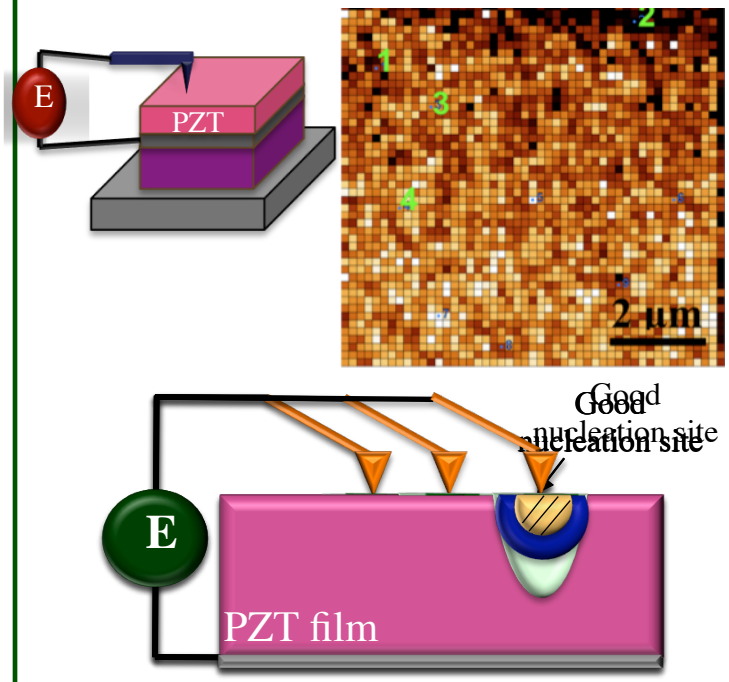
Nucleation Bias Maps by SSPFM

Seal et al., PRL 103, 057601 (2009)

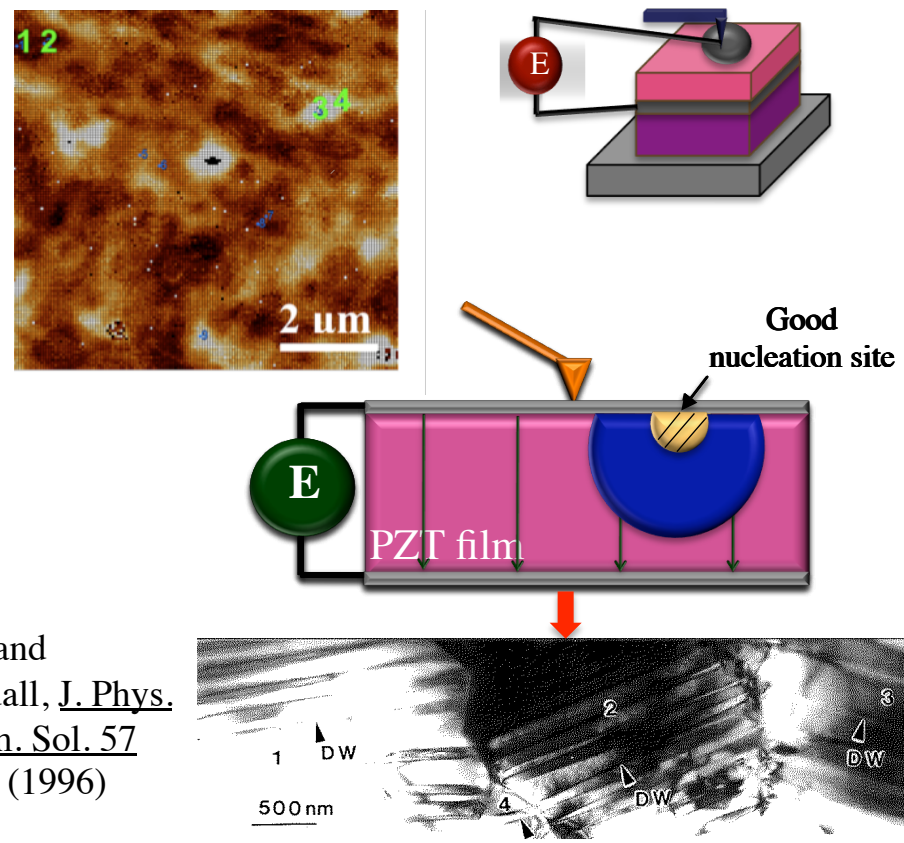
On bare PZT film



On bare PZT films:

- Local hysteresis loops are generally similar for different areas of the sample
- Observation of anomalous loops suggests that electric field is strongly confined under the tip, as expected

On electrode



Cao and Randall, J. Phys. Chem. Sol. 57 1499 (1996)

On electrode: Large regions with uniform nucleation bias suggests that the measurements probe transgranular coupling of the switching (e.g. switching is accomplished if there is a near-by nucleation site)

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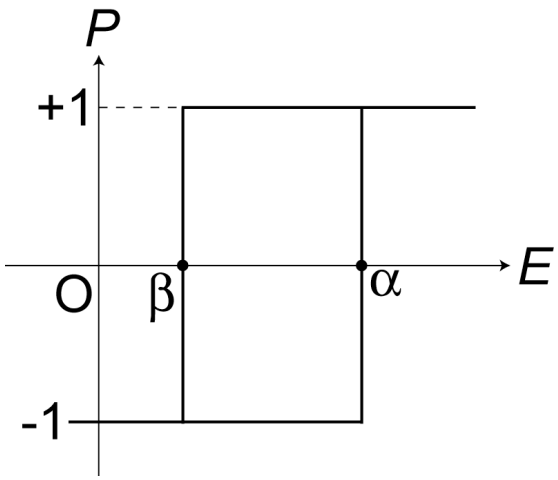
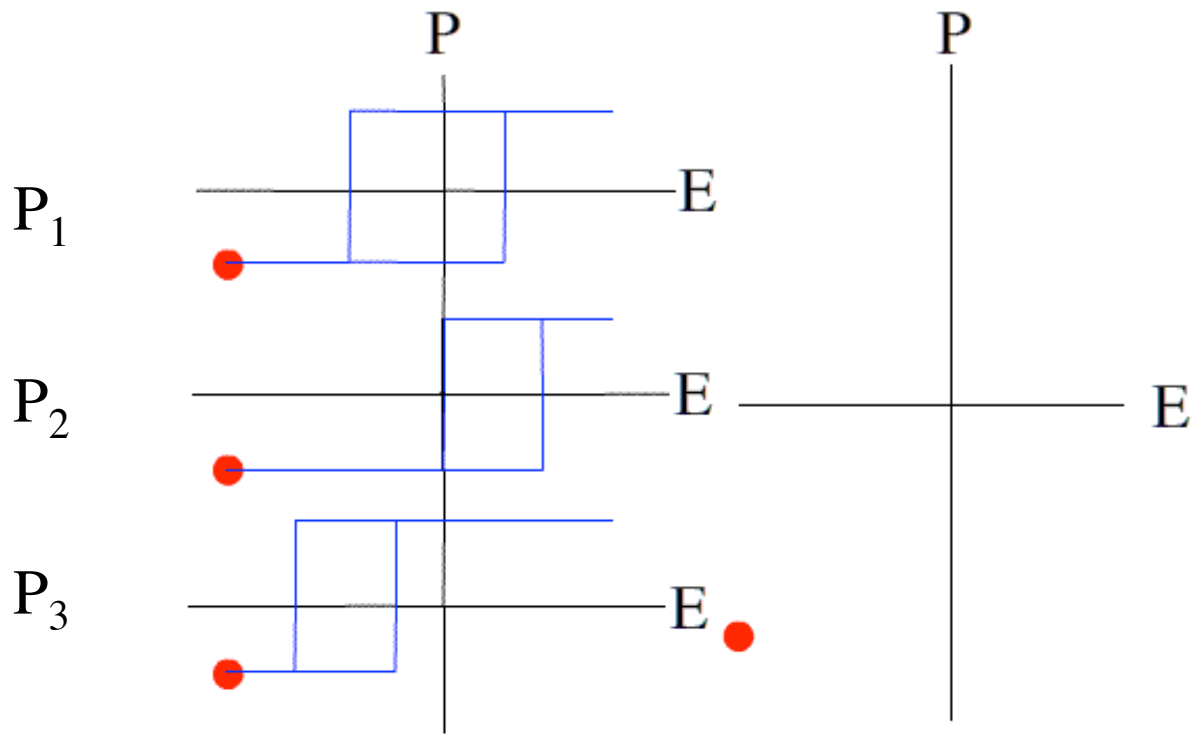
Handling Higher Fields
Preisach Model: Ensembles of Hysterons

If a hysteresis loop consists of 3 hysterons,



α: up-switching field
β: down-switching field



$$P_{total}(E) = \sum_{i}^{3} P_i(E) = P_1(E) + P_2(E) + P_3(E)$$

F. Preisach, *Zeitschrift fur Physik* 94 277 (1935).
I. D. Mayergoyz, *Mathematical models of hysteresis and their applications*, 2003

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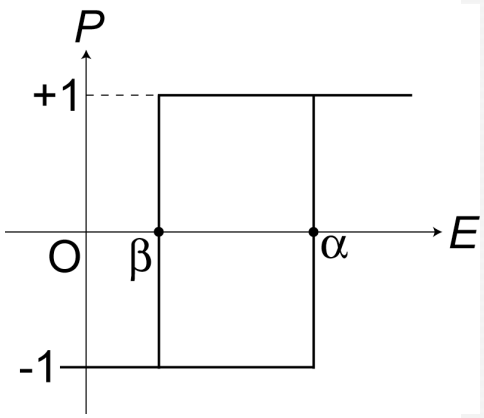
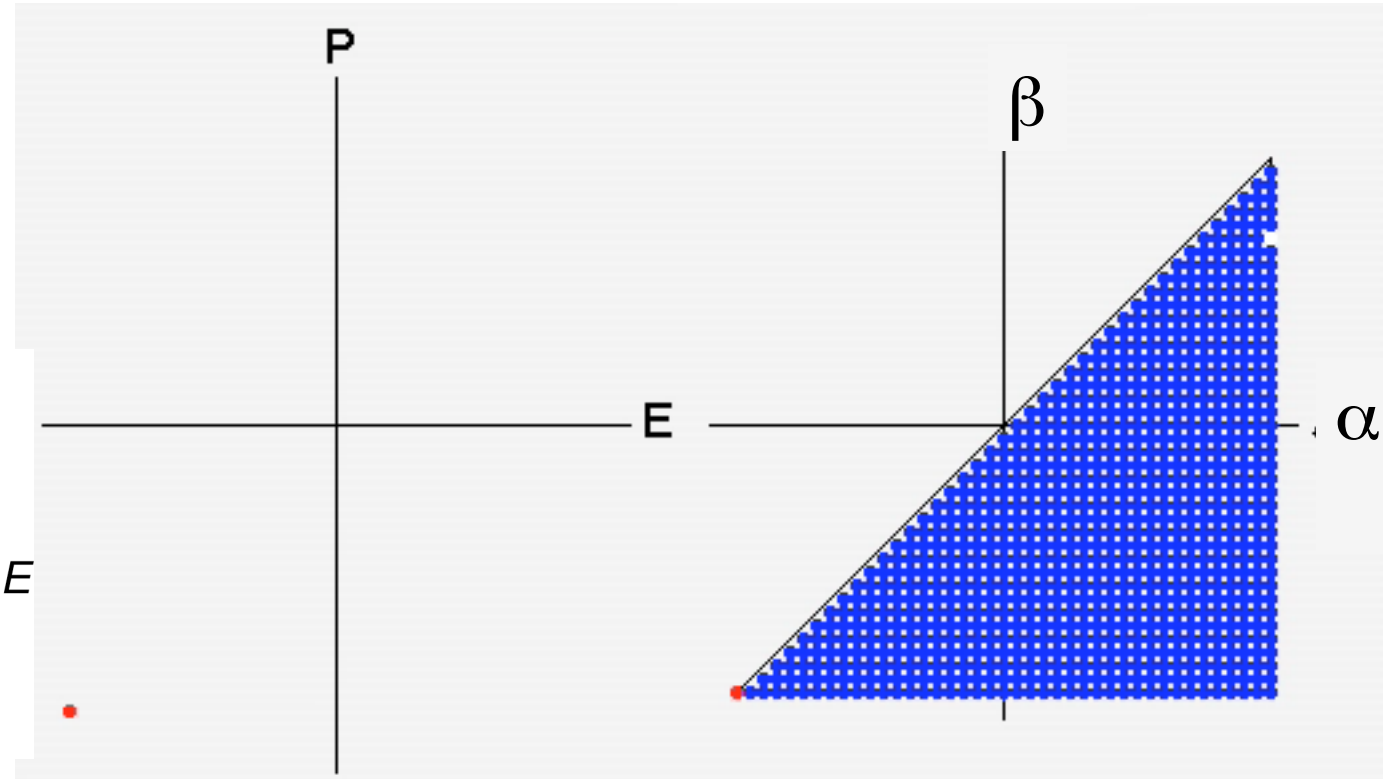
Preisach Calculations





$$P(E) = \iint_{\alpha \geq \beta} q(\alpha,\beta) \cdot p(\alpha,\beta)\, d\alpha\, d\beta$$

$$= -\iint_{\textbf{Blue}} \boldsymbol{p(\alpha,\beta)\, d\alpha\, d\beta} + \iint_{Yellow} p(\alpha,\beta)\, d\alpha\, d\beta$$

$q(\alpha,\beta)$ = +1 (Yellow) or -1 (Blue), depending on history

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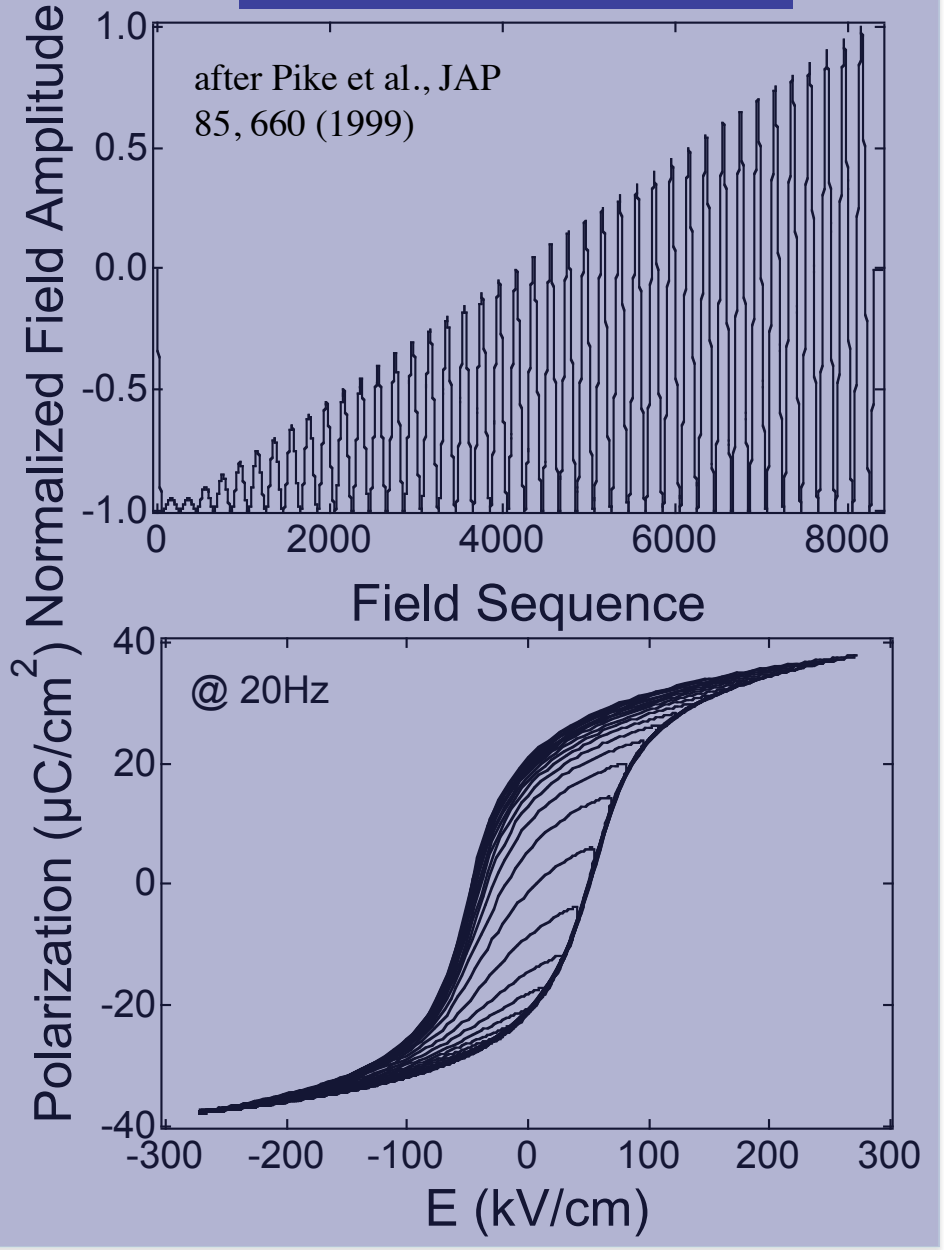
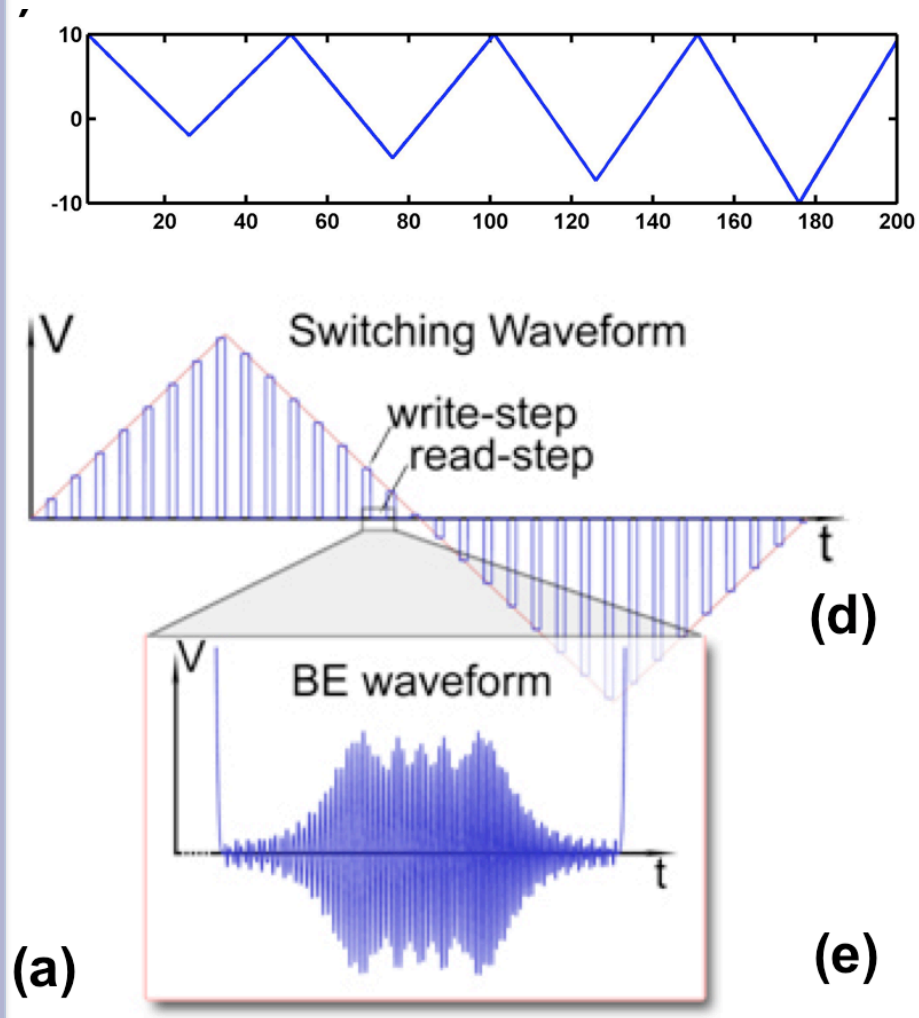
First Order Reversal Curves (FORCs)

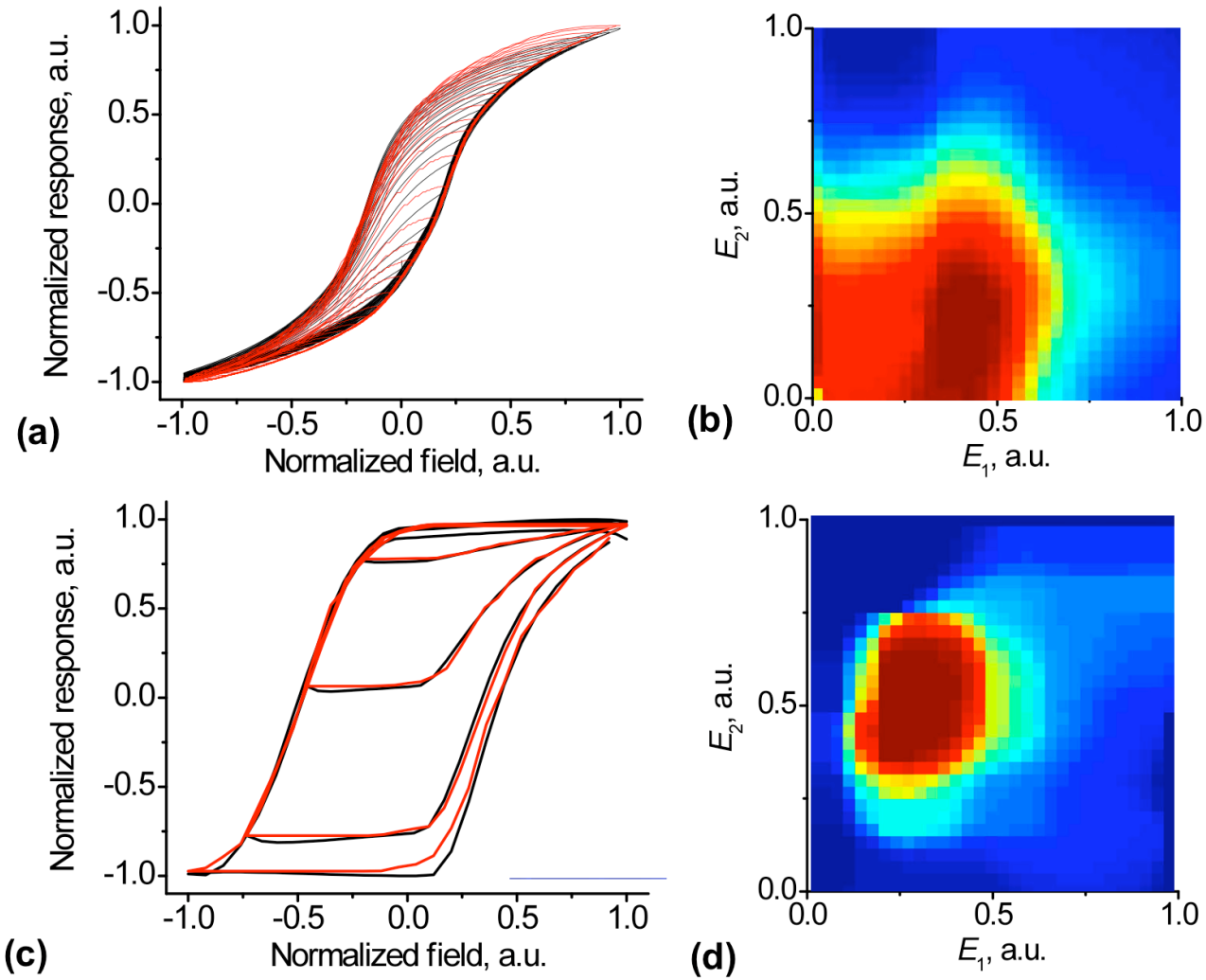
Macroscopic Electrodes



BE-PFM with Top Electrodes



Global and Local FORC



Macroscopic

Microscopic

- Local FORC measurements reproduce many of the essential features of the macroscopic measurements

Ovchinnikov et al., APL 112906 (2010)

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Prospects for Understanding Factors Affecting Wall Mobility using BE-PFM

- Defect chemistry
- Tilt domain structures
- Temperature
- Applied bias
- Dislocations???
- Phase boundaries
- Grain boundaries
- Domain wall density
- Residual stresses

Factors in red should be readily probed by PFM mapping

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Conclusions

- Design of ferroelectric films for MEMS is facilitated by use of film orientation, grain size, and composite connectivity patterns
- Piezoelectric nonlinearity evolves locally in “hot spots,” presumably due to long range coupling of domain wall motion
- Local probes provide insight into designing next generation piezoelectric MEMS

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