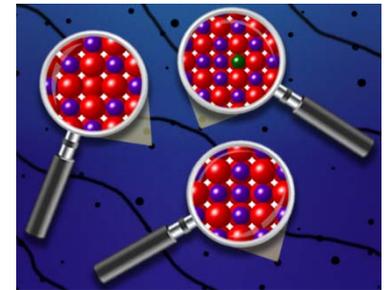
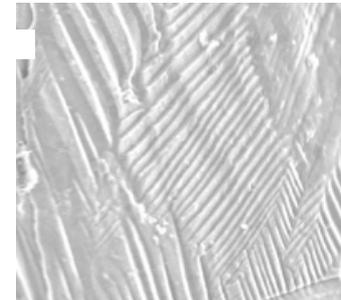
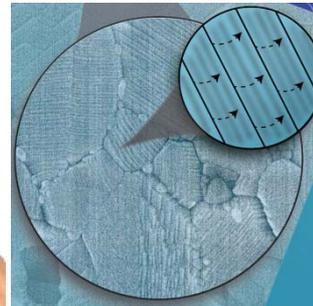
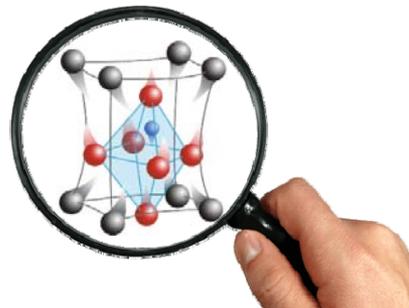
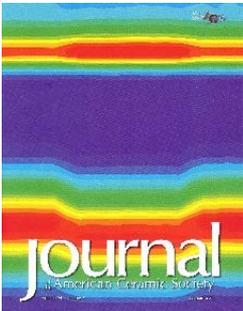


Origins of Electromechanical Strain Ascertained from *in situ* X-ray and Neutron Diffraction

Jacob L. Jones

*Department of Materials Science and Engineering
North Carolina State University*



This work is the compilation of results from a number of students, postdocs, and collaborators, who are acknowledged in the next few slides...

Origins of Electromechanical Strain Ascertained from *in situ* X-ray and Neutron Diffraction

Contributors and Collaborators on this Topic

Jones Research Group

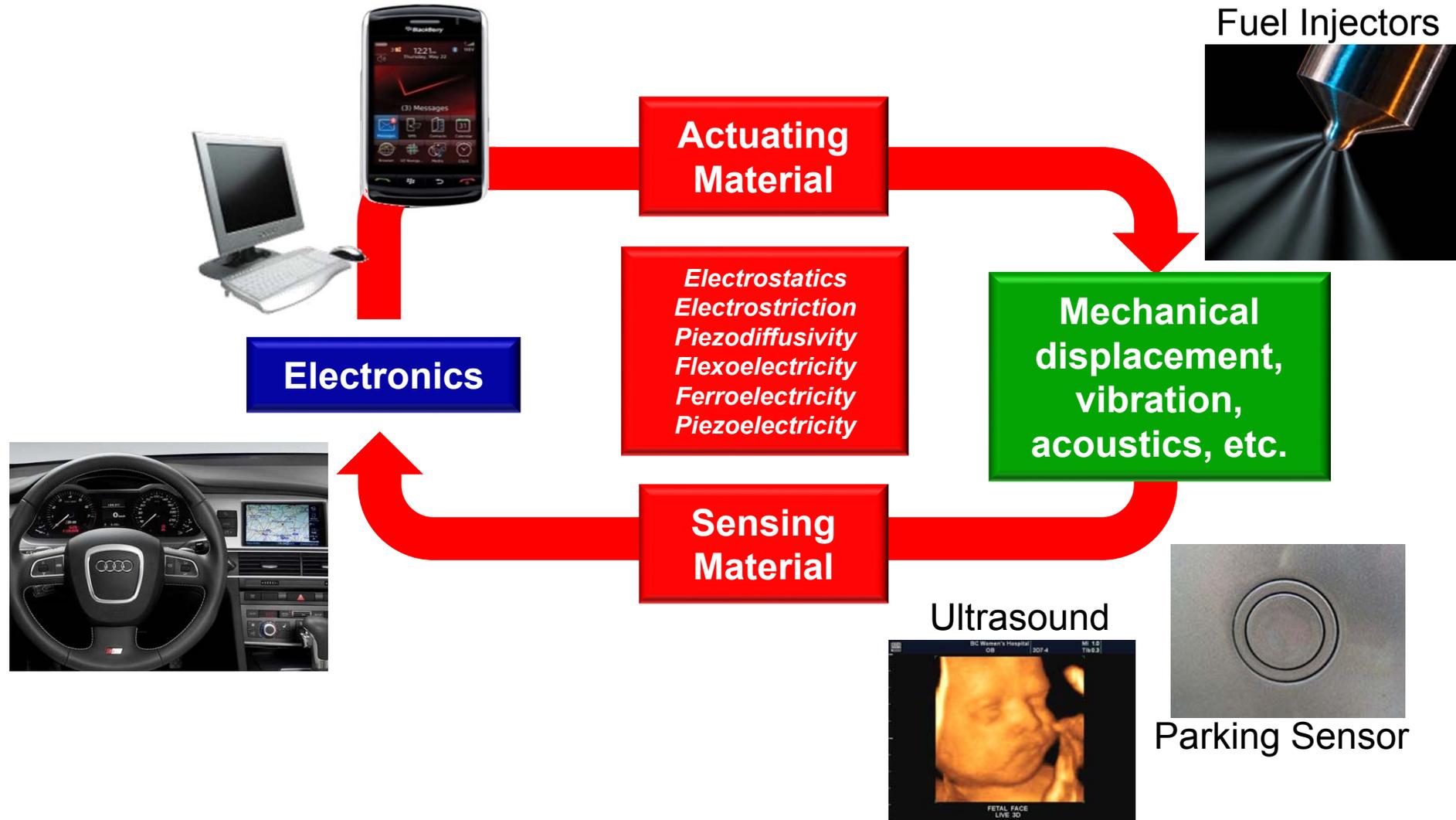


- *John Daniels, UNSW*
- *Dragan Damjanovic, EPFL*
- *Susan Trolier-McKinstry, Penn State*
- *Pam Thomas, Warwick*
- *J. Chen, Univ. Sci. Tech. Beijing*
- *K. Bowman, IIT*
- *S. Pojprapai, Suranaree University of Technology, Thailand*
- *Y. Ren, APS*
- *A. J. Studer, ANSTO*

Origins of Electromechanical Strain Ascertained from *in situ* X-ray and Neutron Diffraction

1. Introduction to **Electromechanical Strain Mechanisms**
2. *In situ* **X-ray Diffraction** experiments
3. Applications
 1. Polycrystalline $Pb(Zr,Ti)O_3$ (**PZT**) (dominant commercial material)
 2. High-temperature $BiScO_3 - PbTiO_3$ piezoelectric
 3. Pb-free $Ba(Zr_{0.2}Ti_{0.8})O_3 - x(Ba_{0.7}Ca_{0.3})TiO_3$ as a function of ***c/a***
 4. Classical $BaTiO_3$ as a function of ***grain size***
 5. Field-induced ***phase transitions*** in $Na_{0.5}K_{0.5}NbO_3$
 6. Polycrystalline ***PZT thin films*** for piezoMEMS, etc.

Applications of Piezoelectrics & Ferroelectrics



Applications of Piezoelectrics & Ferroelectrics

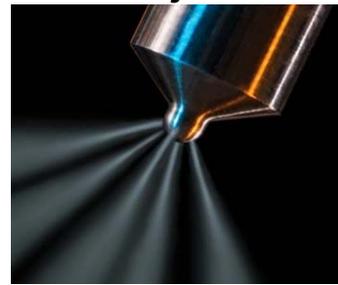


Electronics

**Ferroelectrics
and
Piezoelectrics**

**Mechanical
displacement,
vibration,
acoustics, etc.**

Fuel Injectors

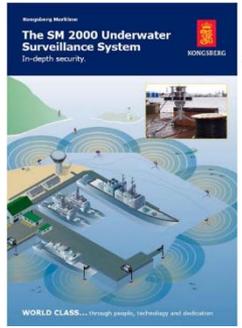


Ultrasound



Parking Sensor

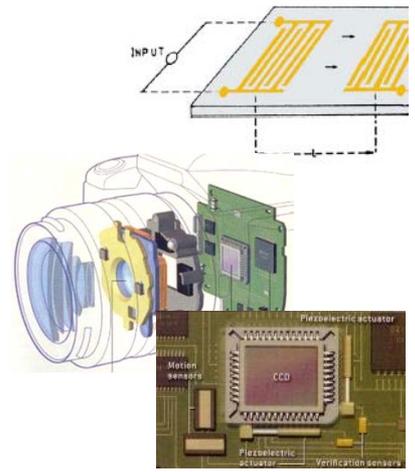
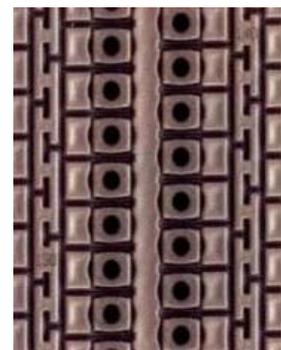
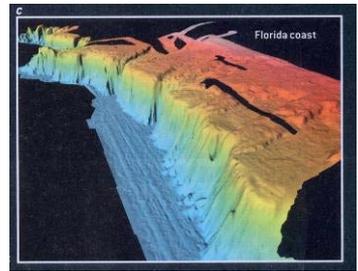
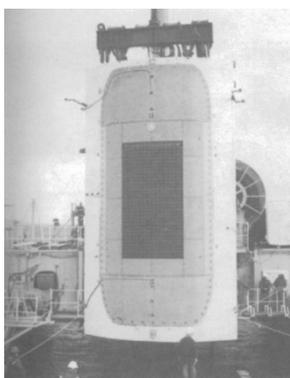
Applications of Piezoelectrics & Ferroelectrics



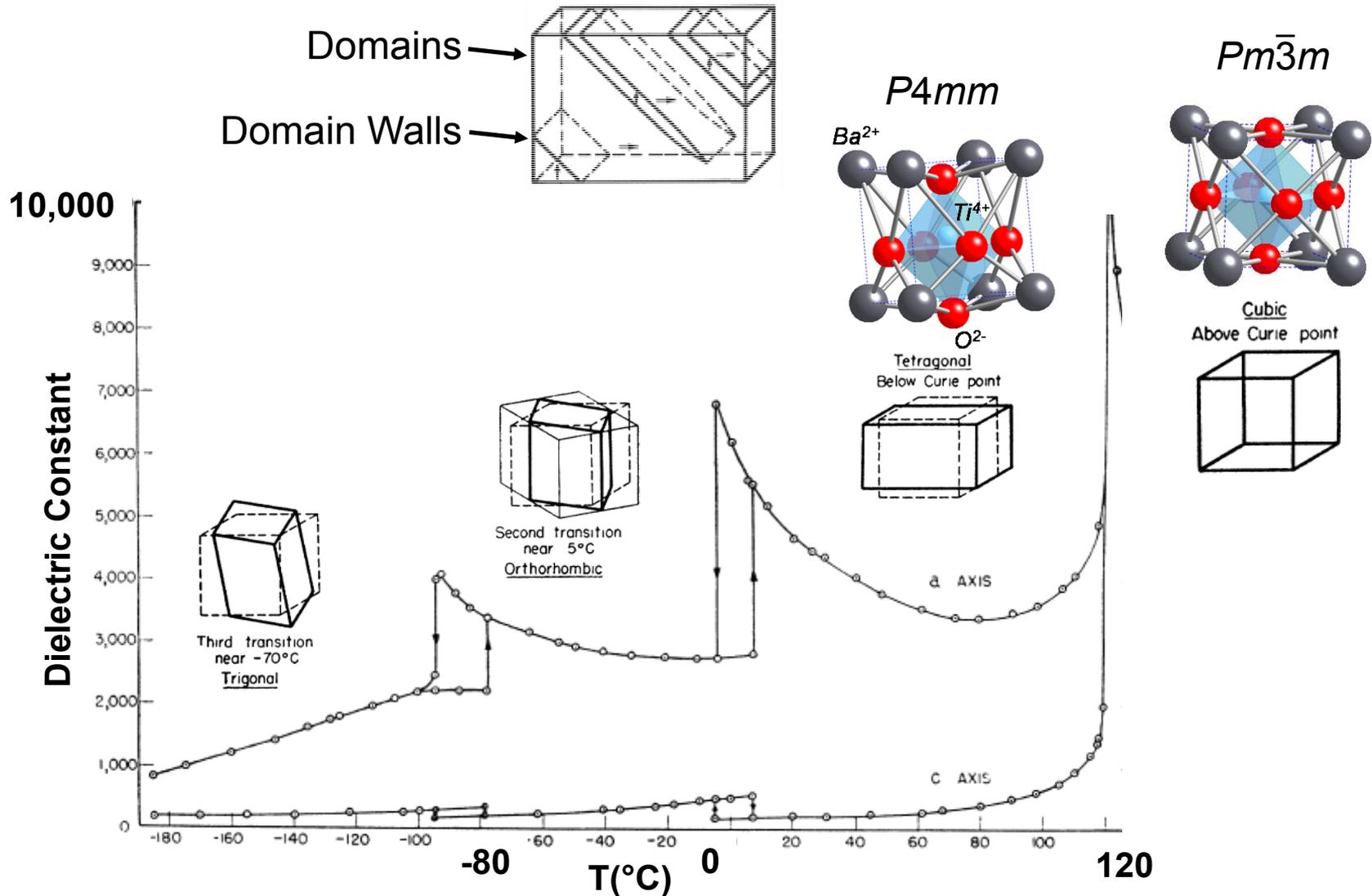
Electronics

Ferroelectrics and Piezoelectrics

Mechanical displacement, vibration, acoustics, etc.

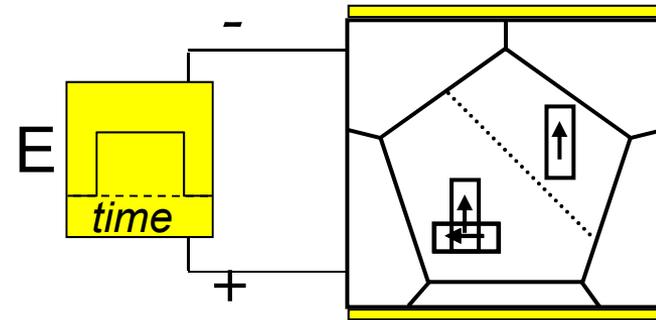
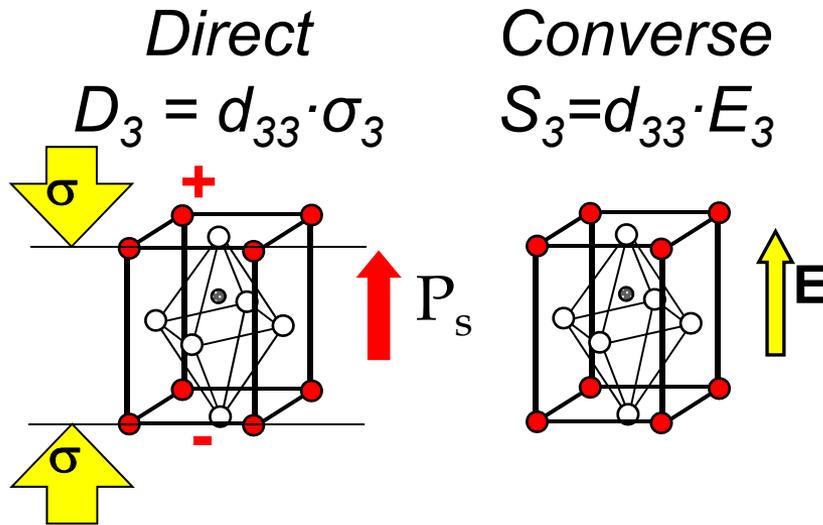


BaTiO₃ – a Classical Example



Adapted from A. von Hippel, Rev. Modern Physics, 22, 221 (1950).

Mechanisms of Electromechanical Strain



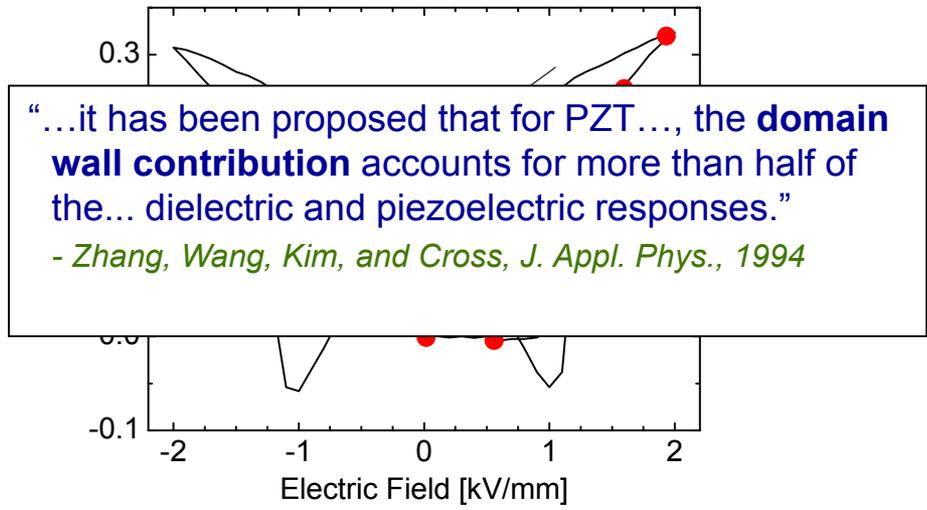
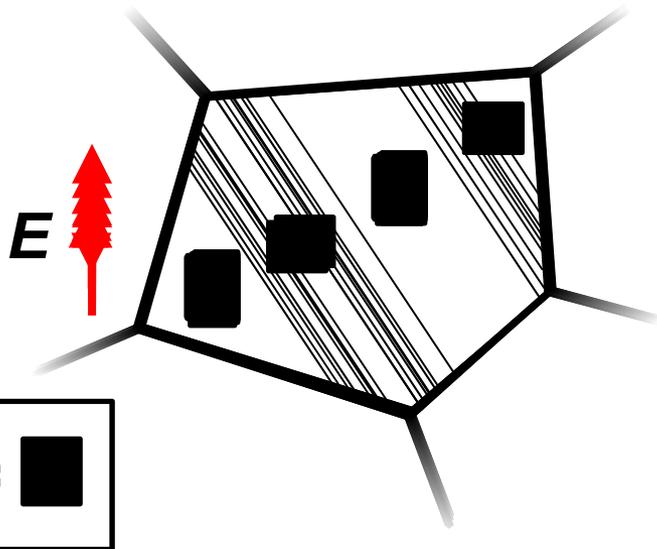
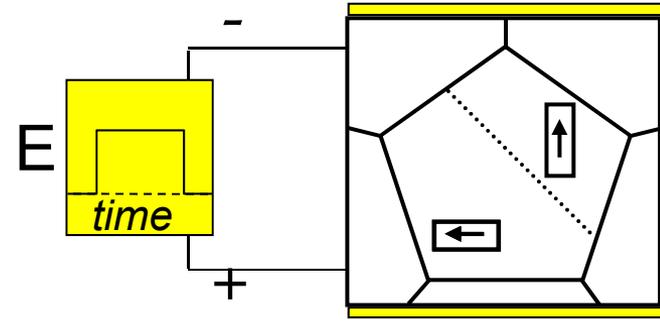
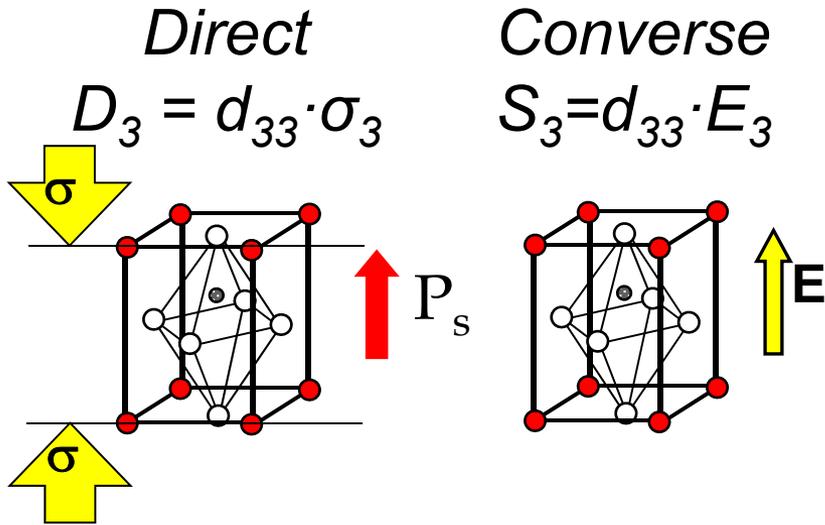
Piezoelectric strain

- **Intrinsic** effect of polar structures
- Expect **linear** response of strain to field amplitude, or
- d_{33} being a “constant”

Domain wall motion (ferroelectric effect)

- **Extrinsic** effect
- Expect strain coefficient (strain/field) to be **nonlinear** and dependent upon many variables.

Mechanisms of Electromechanical Strain



- Polarization rotation (intrinsic)

letters to nature

Polarization rotation mechanism for ultrahigh electromechanical response in single-crystal piezoelectrics

Huaxiang Fu & Ronald E. Cohen

Carnegie Institution of Washington, 5251 Broad Branch Road, NW, Washington DC 20015, USA

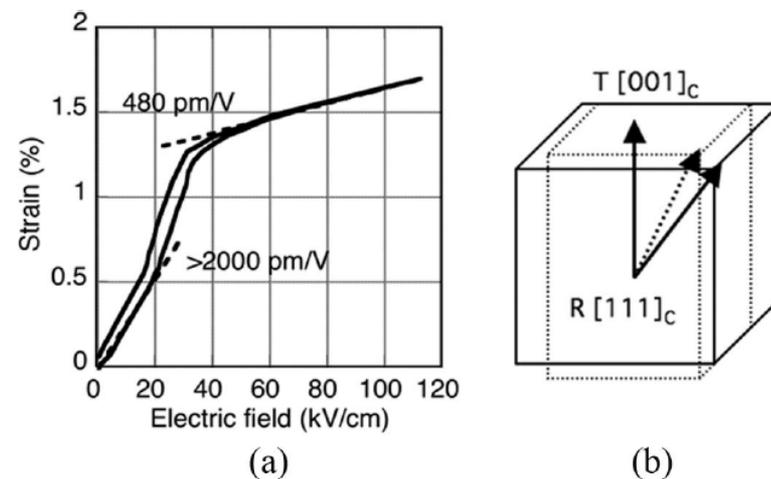
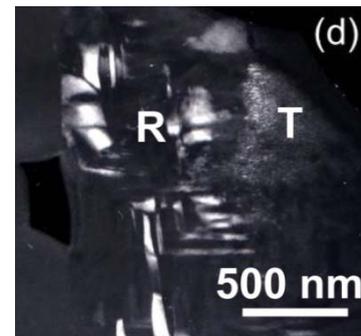
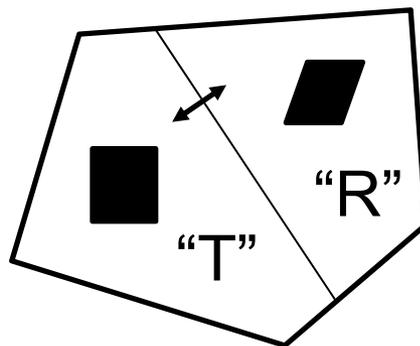
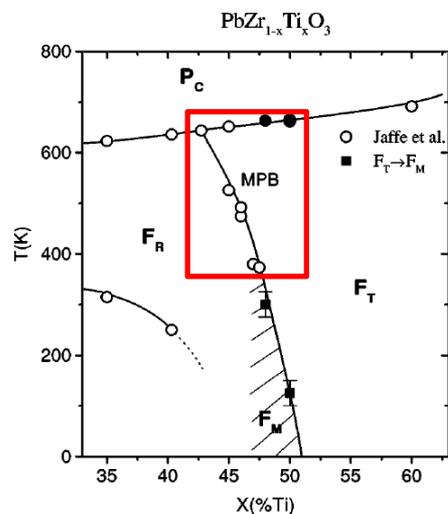
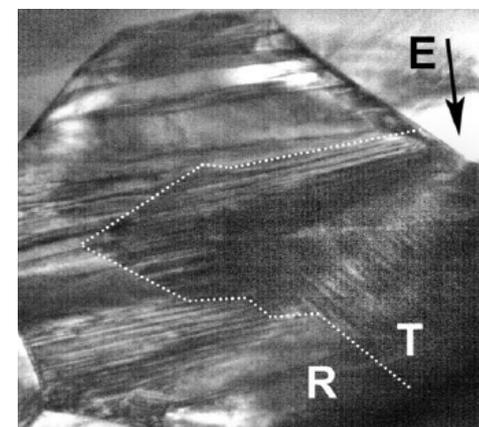


Fig. 1. (a) The field induced strain in $0.92\text{Pb}(\text{Zn}_{1/3}\text{Nb}_{2/3})\text{O}_3 - 0.08\text{Pb-TiO}_3$ crystal and (b) schematic presentation of the polarization “inclination” under electric field from rhombohedral $[111]_C$ toward tetragonal $[001]_C$ polar axis. The field is applied along $[001]_C$. After Ref [1].

• **Interphase boundary motion (extrinsic)**



Levin et al., *Adv. Funct. Mater.* (2012).



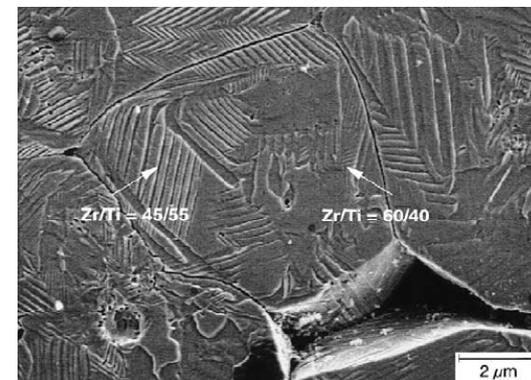
Ma, Tan et al., *PRL* (2012)

“the observed behavior of the electromechanical properties of the ceramics is based on the model of the domain wall and **interphase boundary motion.**”

- Kugel and Cross, *J. Appl. Phys.*, 1998

“It is tempting to speculate that in ferroelectric compositions close to the morphotropic phase boundary, ... the **interfaces between different phases can be moved by external fields** and contribute to the properties in a similar way as the moving domain walls. At present, **there is no direct evidence that this interphase boundary displacement is actually happening.**”

- Damjanovic, *Chapter 4, The Science of Hysteresis*, 2005



Hoffmann et al., *Acta Mater.* (2001).

Mechanisms of Electromechanical Strain

Intrinsic Effects

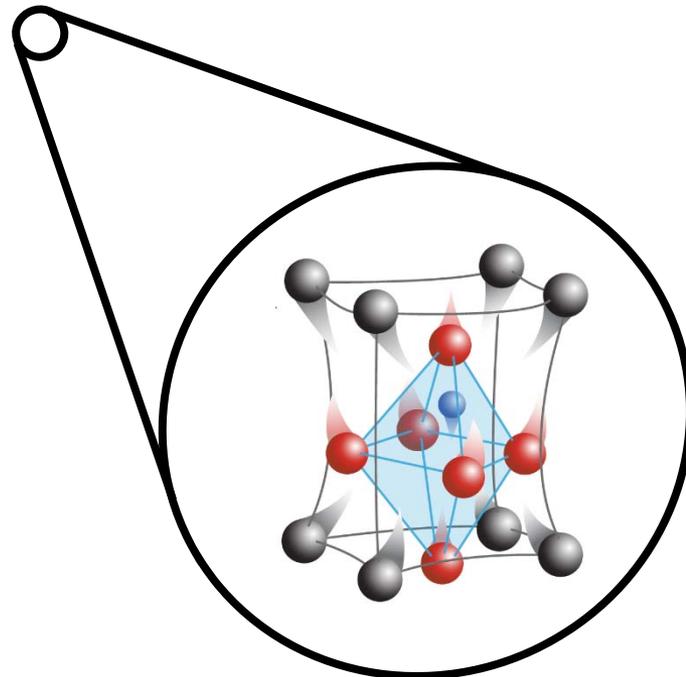
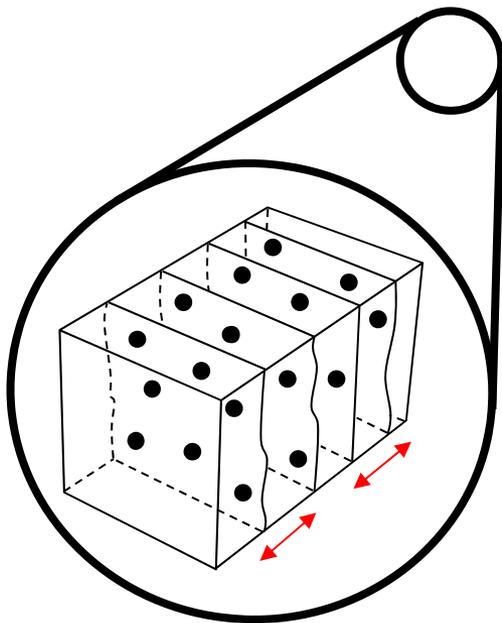
- Piezoelectric effect
- Polarization rotation

Extrinsic Effects

- Domain wall motion
- Interphase boundary motion

Mechanisms of Electromechanical Strain

Not so simple: **Emergence** and **Complexity** at length scales between 0.4 nm and $\sim 10 \mu\text{m}$



Mechanisms of Electromechanical Strain

Intrinsic Effects

- Piezoelectric effect
- Polarization rotation

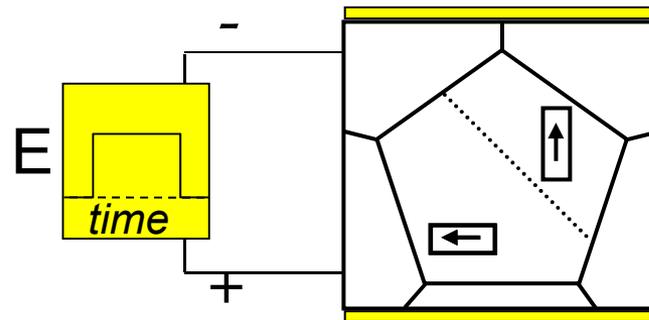
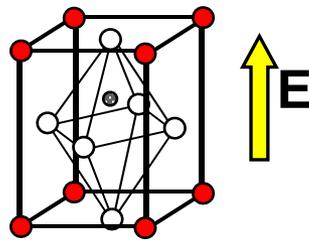
Extrinsic Effects

- Domain wall motion
- Interphase boundary motion

- **Domain wall pinning/depinning** with point and line defects
- **Domain wall – domain wall** interactions (i.e., ρ_{DW})
- **Competing mechanisms** of domain wall motion and intrinsic piezoelectricity
- **Domain wall interactions** with grain boundaries
- Effect of **spontaneous strain** on domain wall contributions
- **Intergranular** (grain-grain) interactions and constraint or compliance
- **Intrinsic elasticity** affects this coupling
- **Grain size** effects
- **Domain size** effects (e.g., polar nanoregions)
- Effect of **stress** (i.e., thin films)

Motivation

- When *electric fields are applied to insulating materials*, several different physical phenomena are known to create mechanical strain (e.g. **piezoelectricity**, **electrostriction**, **ferroelectric/ferroelastic domain wall motion**, and **flexoelectricity**).



Motivation

- When *electric fields are applied to insulating materials*, several different physical phenomena are known to create mechanical strain (e.g. **piezoelectricity**, **electrostriction**, **ferroelectric/ferroelastic domain wall motion**, and **flexoelectricity**).
- It is often difficult to *distinguish which mechanisms are active or dominant* in a particular material system, and how *the highest or designed material responses* can be achieved.

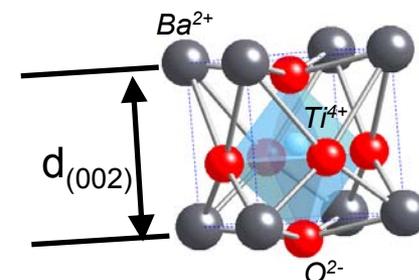
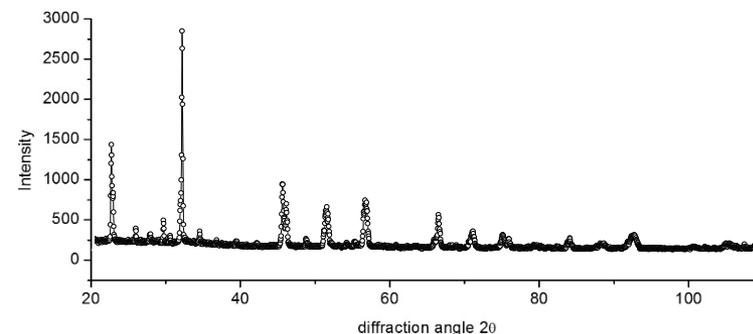
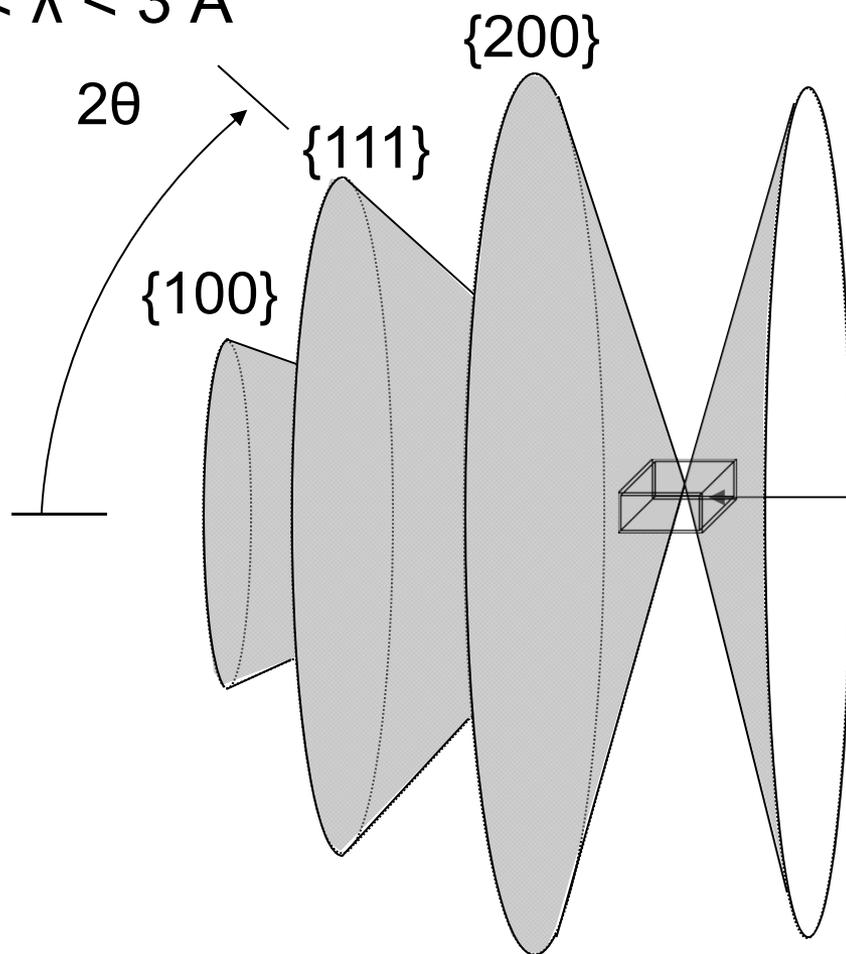
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- It is often difficult to *distinguish which mechanisms are active or dominant* in a particular material system, and how *the highest or designed material responses* can be achieved.
- *In situ characterization probes* (e.g., X-ray and neutron scattering) can deconvolute some of these effects and provide quantitative assessment of contributions.

Methodology: X-ray Diffraction

X-ray diffraction

$$0.1 \text{ \AA} < \lambda < 3 \text{ \AA}$$



Bragg's Law

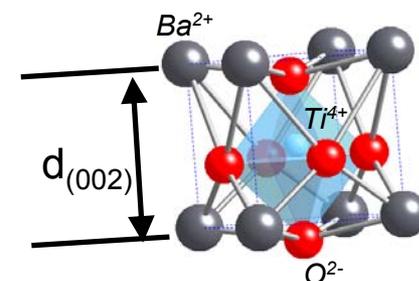
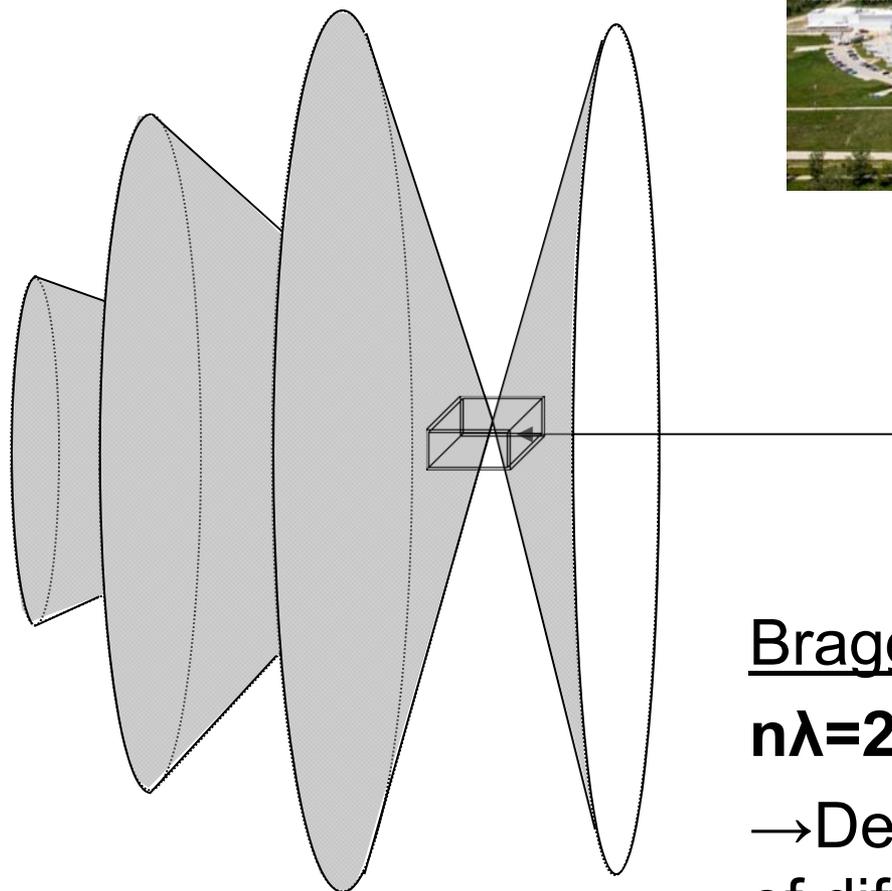
$$n\lambda = 2d \cdot \sin\theta$$

→ Debye-Scherrer cones of diffraction intensity

Methodology: X-ray Diffraction

High-energy X-ray diffraction

$$\lambda < 1 \text{ \AA}$$



Bragg's Law

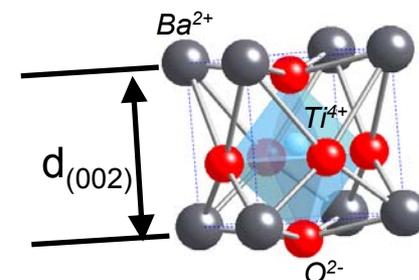
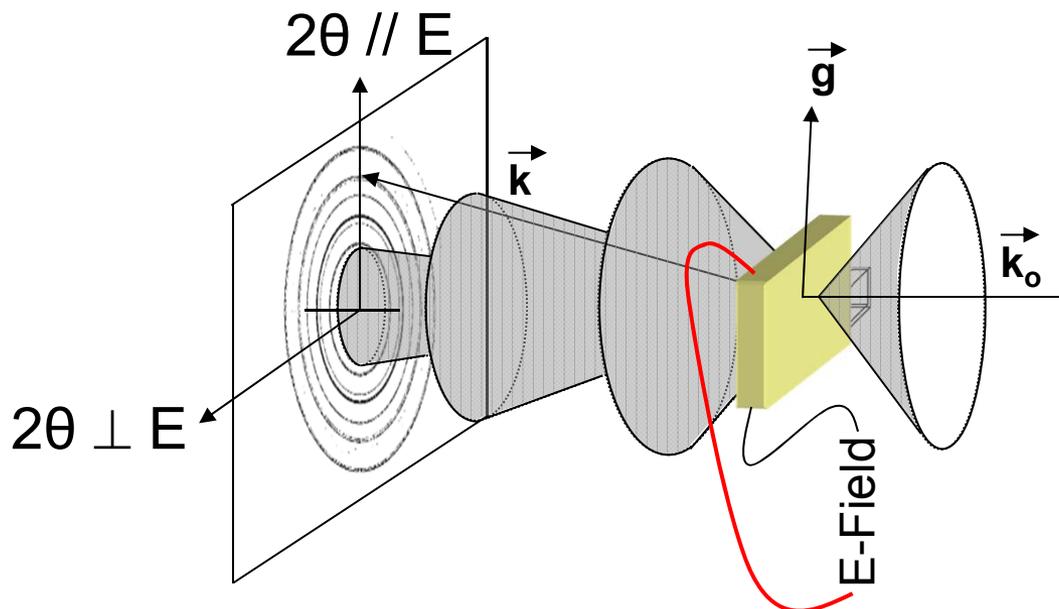
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$$\lambda < 1 \text{ \AA}$$



Bragg's Law

$$n\lambda = 2d \cdot \sin\theta$$

→ Debye-Scherrer cones of diffraction intensity

Methodology: X-ray Diffraction

Sophisticated *in situ* experiments

Analysis including profile fitting

Quantifying extrinsic and intrinsic effects

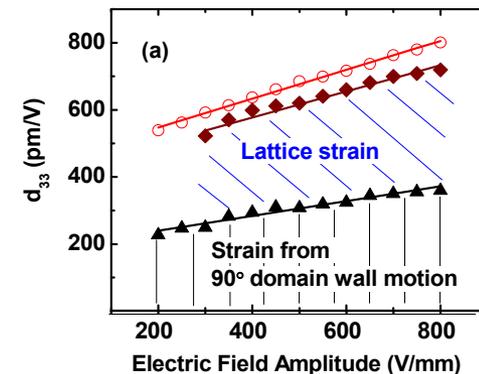
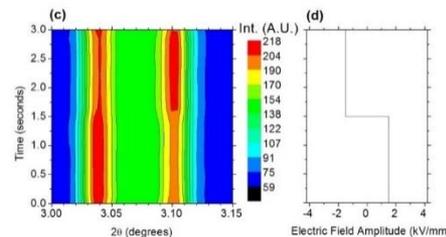
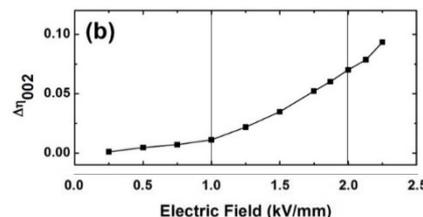
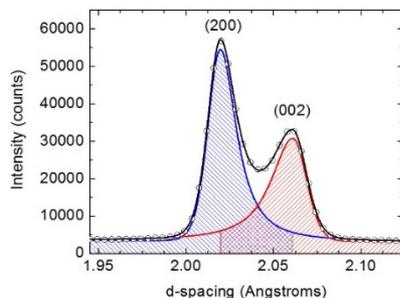
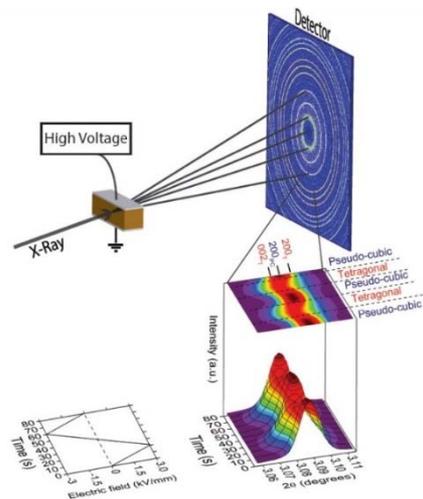
Relation to macroscopic properties

- Strong and weak fields
- Electric field synchronization
- Stroboscopic
- Dependence on field direction

- High intensities for resolving subtle effects
- Careful selection of profile shapes
- Automated for 1000s of datasets

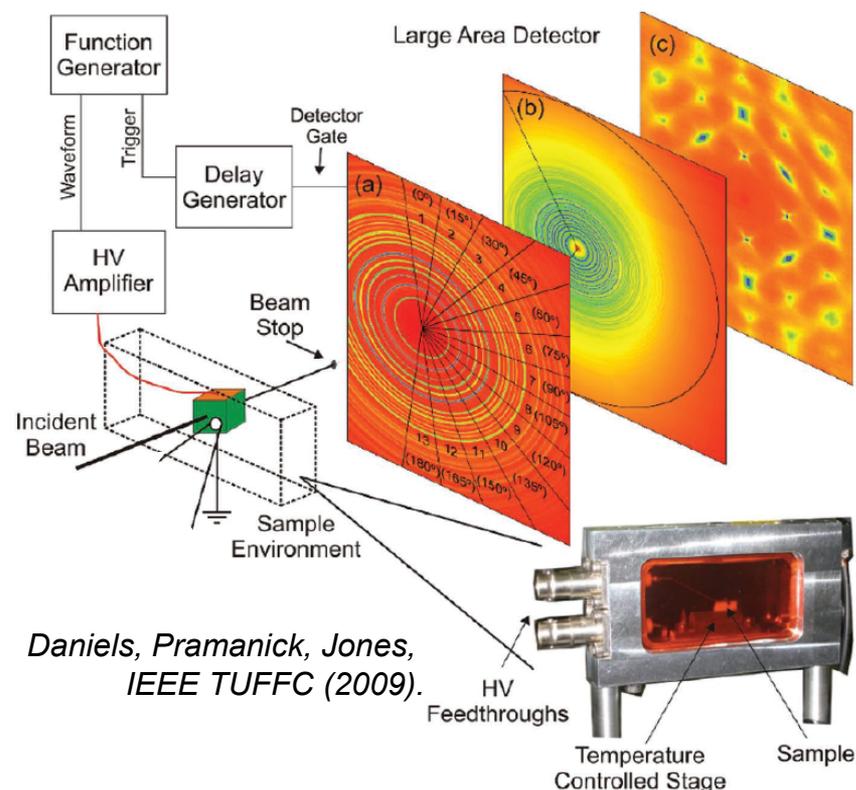
- Domain volume fraction calculations (η)
- Quantifying other effects, e.g. interphase boundary motion and lattice strain
- Field-amplitude dependence
- Frequency dependence

- Calculate effective contribution to macroscopic property coefficients (e.g., d_{33})
- Measure macroscopic property coefficients
- Interpret without presumption of superposition



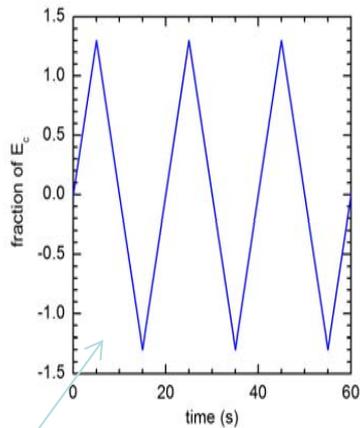
Methodology: X-ray Diffraction

- APS, Beamline 11-ID-C
- 114 keV energy
- through thin films (through the entire structure, including Si);
- or through polycrystalline samples of 1 mm x 1 mm x 5 mm sample.
- Perkin Elmer area detector, 1.8 m from sample
- Voltages up to 6 kV, often stroboscopic methods
- **High-energy (hard) X-ray scattering lends itself to investigation of sub-surface phenomena, and allow easy powder averaging to relate to macroscopic properties.**

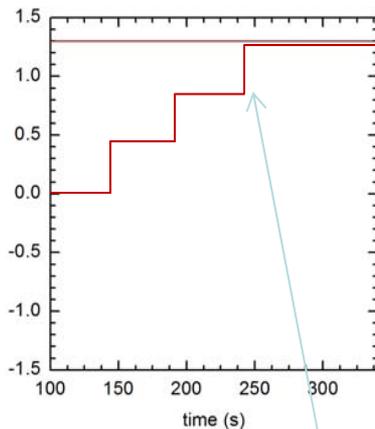


Methodology: X-ray Diffraction

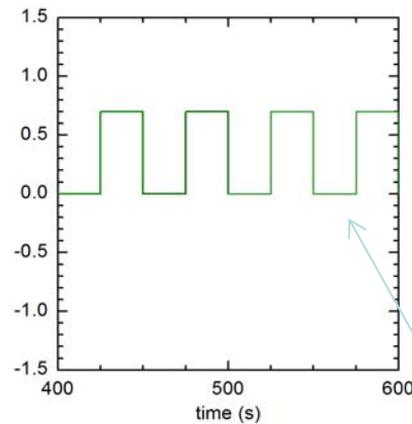
Strong bipolar fields – high field cycling



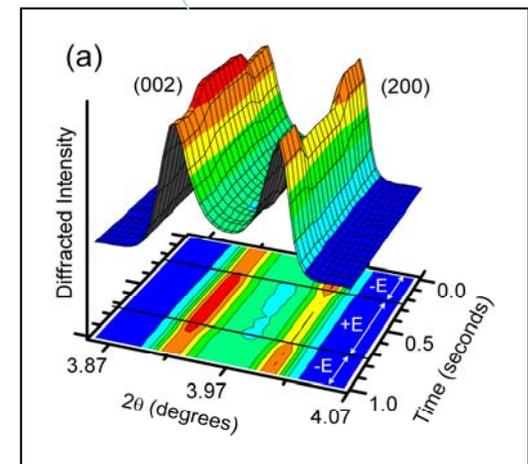
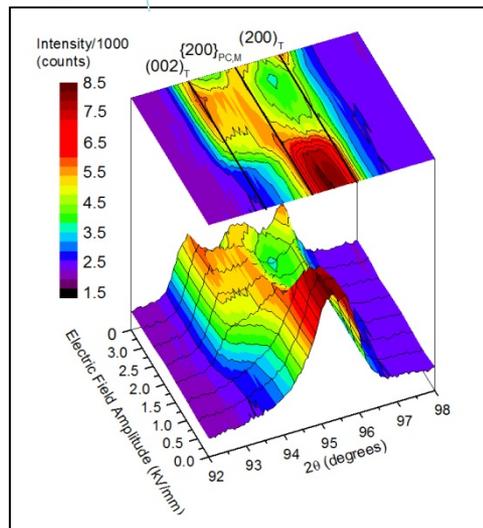
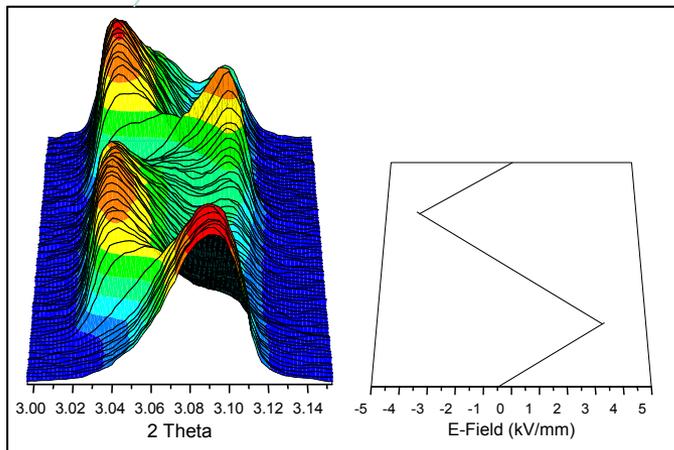
Strong DC fields - "poling"



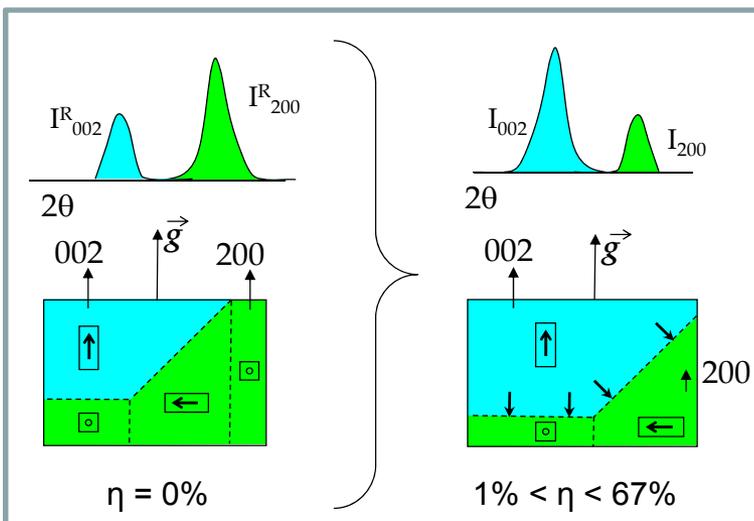
Weak unipolar or bipolar fields – property coefficients



As a function of field amplitude, cycle number, etc. – Aging; Rayleigh behavior, etc.



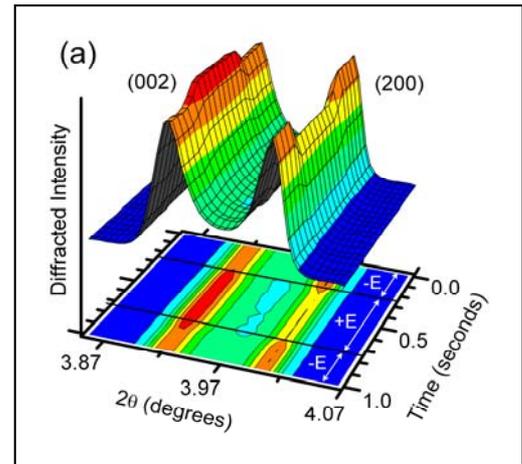
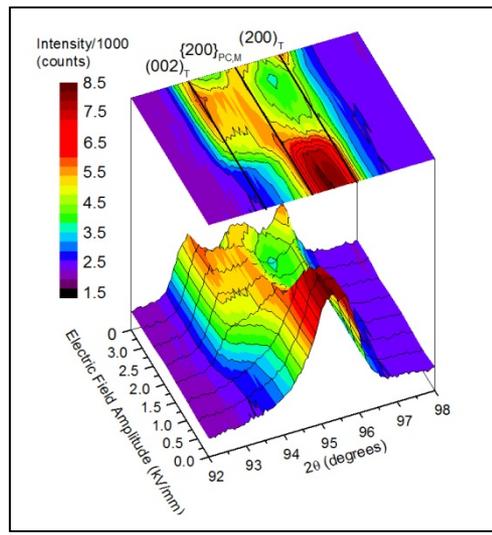
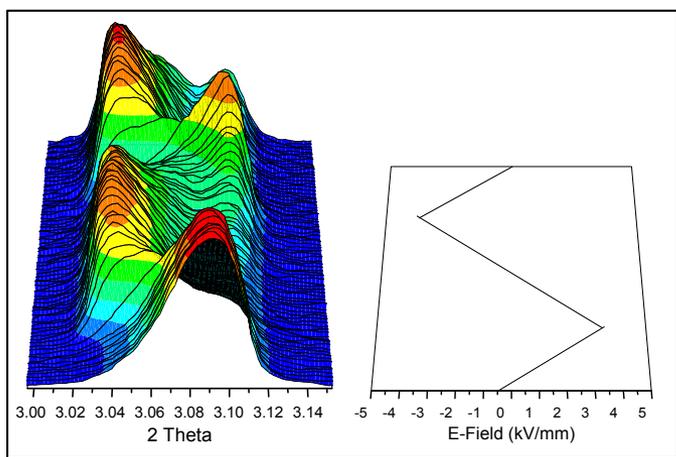
Methodology: X-ray Diffraction



- Quantifying ferroelastic domain wall motion: measured in diffraction by a change in the intensities of certain peaks

$$\eta_{002} (\%) = \frac{I_{002}/I_{002}^{\text{unpoled}}}{I_{002}/I_{002}^{\text{unpoled}} + 2 \cdot (I_{200}/I_{200}^{\text{unpoled}})} - \frac{1}{3}$$

Jones et al., J. Appl. Phys., vol. 97, 034113, 2005.



Methodology: X-ray Diffraction

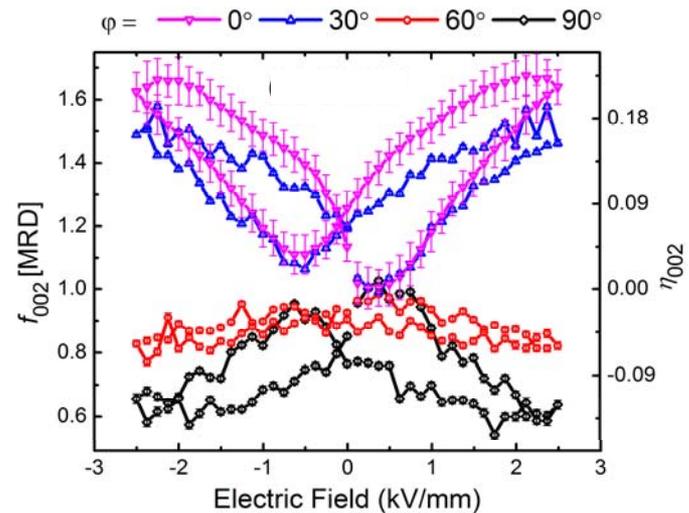
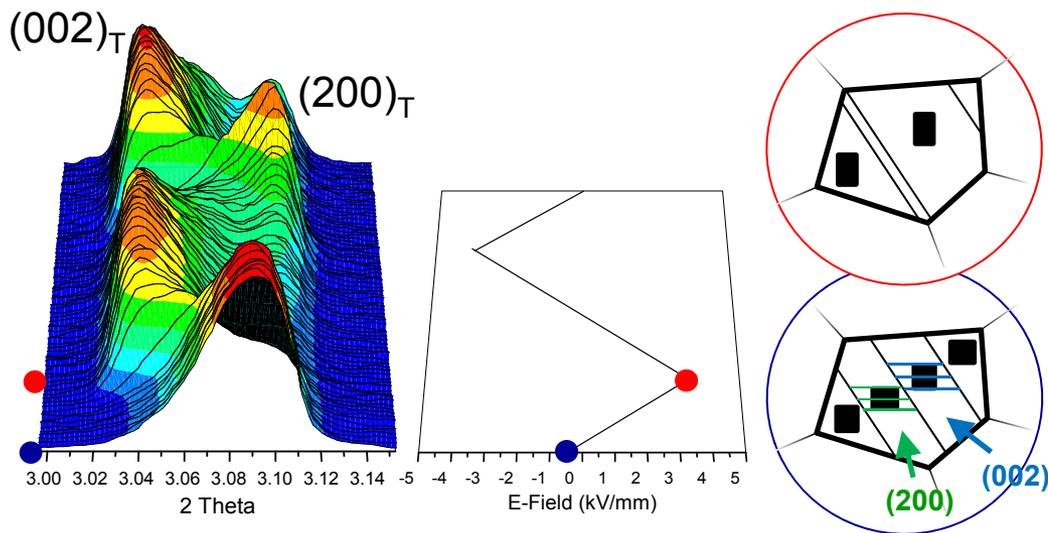
$\eta = 0\%$

$1\% < \eta < 67\%$

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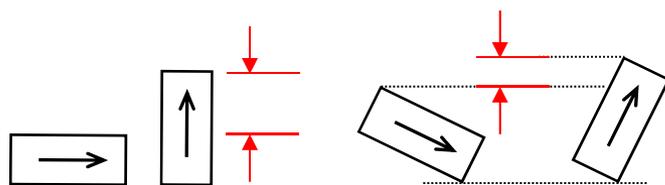
Methodology: X-ray Diffraction

Volume average of a tensorial quantity,¹ $\langle t \rangle$

$$\langle t \rangle = \frac{1}{V} \int_V t(x) dV$$

Arithmetic average of domain reorientation yields²

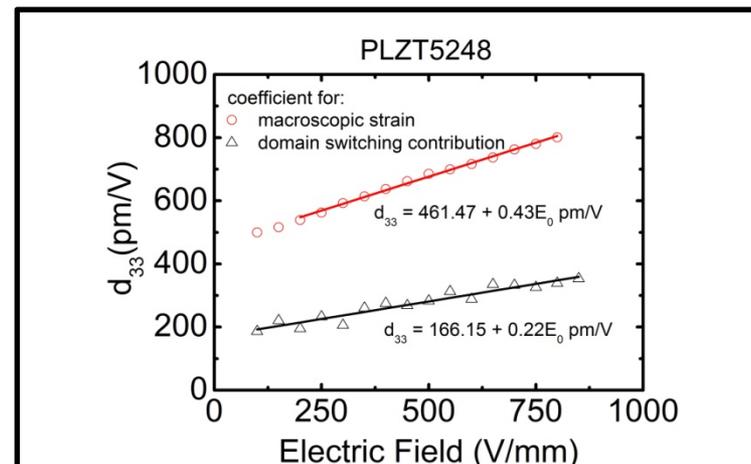
$$\langle \epsilon_{\text{non-180}} \rangle = \frac{(c-a)}{a} \int_0^{\pi/2} 3 \cdot \Delta\eta(\alpha) \cdot \cos^2 \alpha \cdot \sin \alpha \cdot d\alpha$$



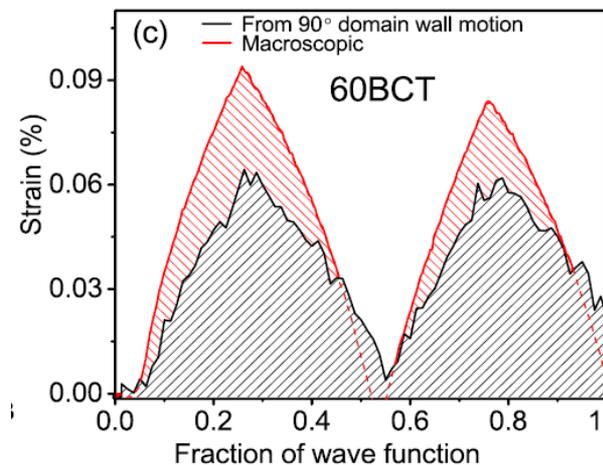
$$d_{\text{non-180}} = \frac{\langle \epsilon_{\text{non-180}} \rangle}{2 \cdot E_0}$$

¹e.g. see Kocks et al., *Texture and Anisotropy*, Cambridge Press (1998).

²Jones et al., *J. Appl. Phys.*, vol. 97, 034113, 2005.



Pramanick et al., *J. Am. Ceram. Soc.*, 92 2300 (2009)

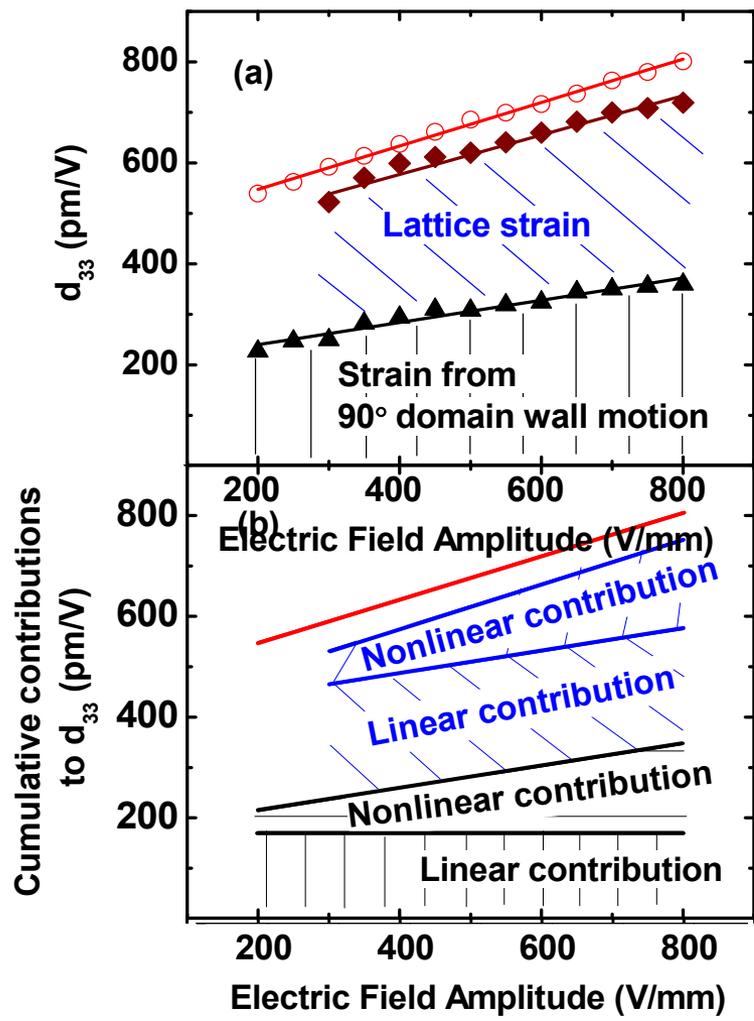


Tutuncu et al., *J. Appl. Phys.*, 115, 144104 (2014).

Application to Diverse Materials Systems

1. Polycrystalline $Pb(Zr,Ti)O_3$ (PZT) (dominant commercial material)
2. High-temperature $BiScO_3 - PbTiO_3$ piezoelectric
3. Pb-free $Ba(Zr_{0.2}Ti_{0.8})O_3 - x(Ba_{0.7}Ca_{0.3})TiO_3$ as a function of c/a
4. Classical $BaTiO_3$ as a function of *grain size*
5. Field-induced *phase transitions* in $Na_{0.5}K_{0.5}NbO_3$
6. Polycrystalline *PZT thin films* for piezoMEMS, etc.

2at% La-substituted $\text{Pb}(\text{Zr}_{0.52}\text{Ti}_{0.48})\text{O}_3$



Take-home Message:

- Lattice strains are only weakly piezoelectric, and are mostly attributed to intergranular strain coupling.
- Most of the piezoelectric property coefficients can be attributed to 90° domain wall motion.

Take-home Message:

- ~50% of piezoelectric coefficient in PZT results directly from 90° domain wall contributions.



High-Temperature $.36\text{BiScO}_3\text{-}.64\text{PbTiO}_3$

- BS-PT can exhibit $d_{33} > 460$ pC/N and a high T_c of 450°C near the morphotropic phase boundary (MPB).

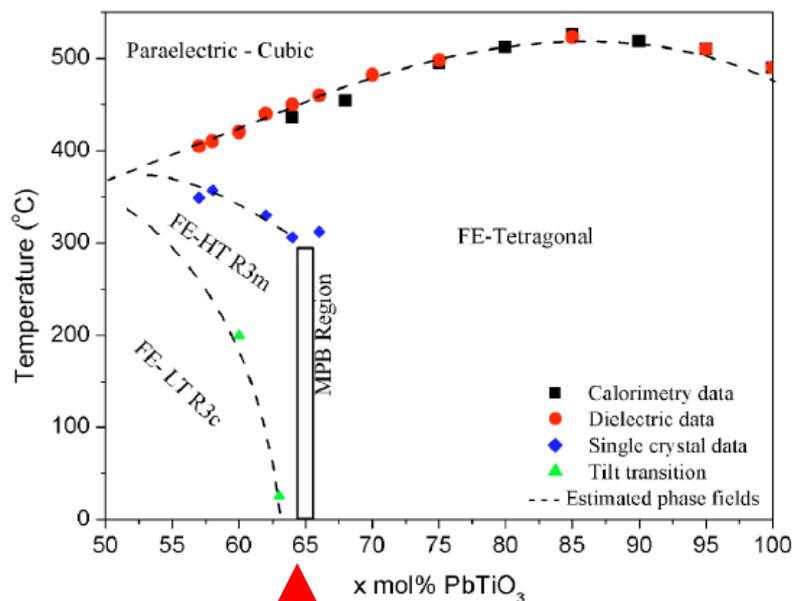
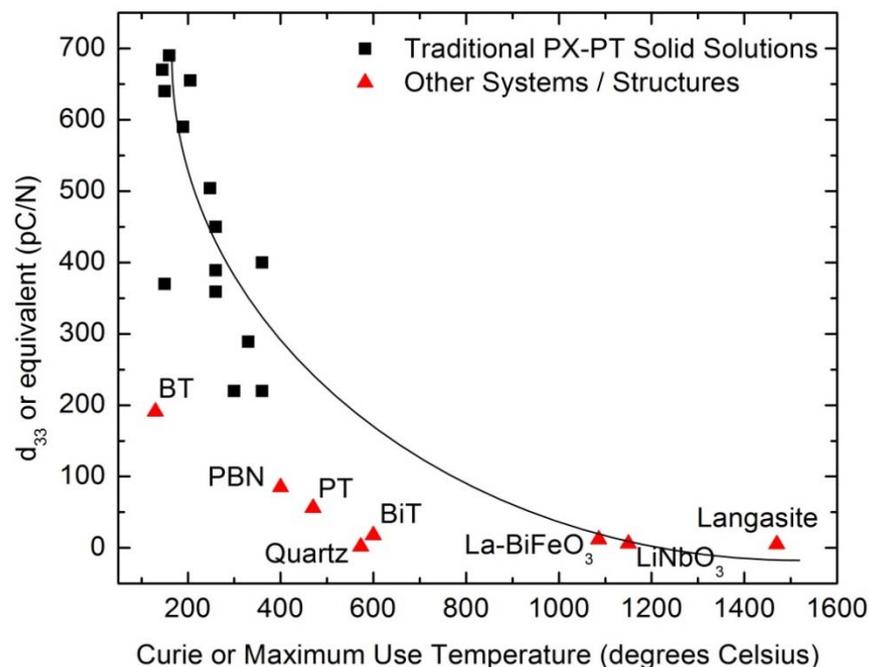


FIG. 3. Phase diagram for the perovskite $(1-x)\text{BiScO}_3\text{-}x\text{PbTiO}_3$ system representing revisions based on the TEM and single-crystal data.



High-Temperature $.36\text{BiScO}_3\text{-}.64\text{PbTiO}_3$

- BS-PT can exhibit $d_{33} > 460$ pC/N and a high T_c of 450°C near the morphotropic phase boundary (MPB).

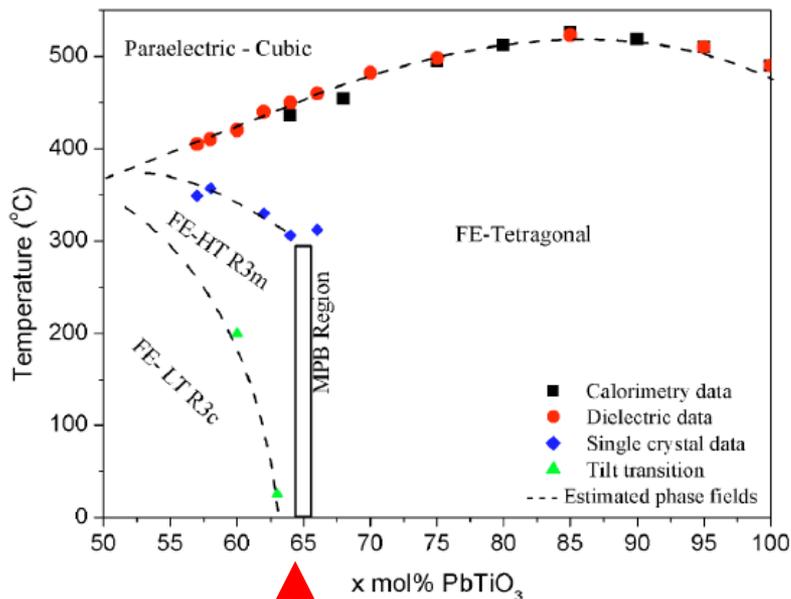
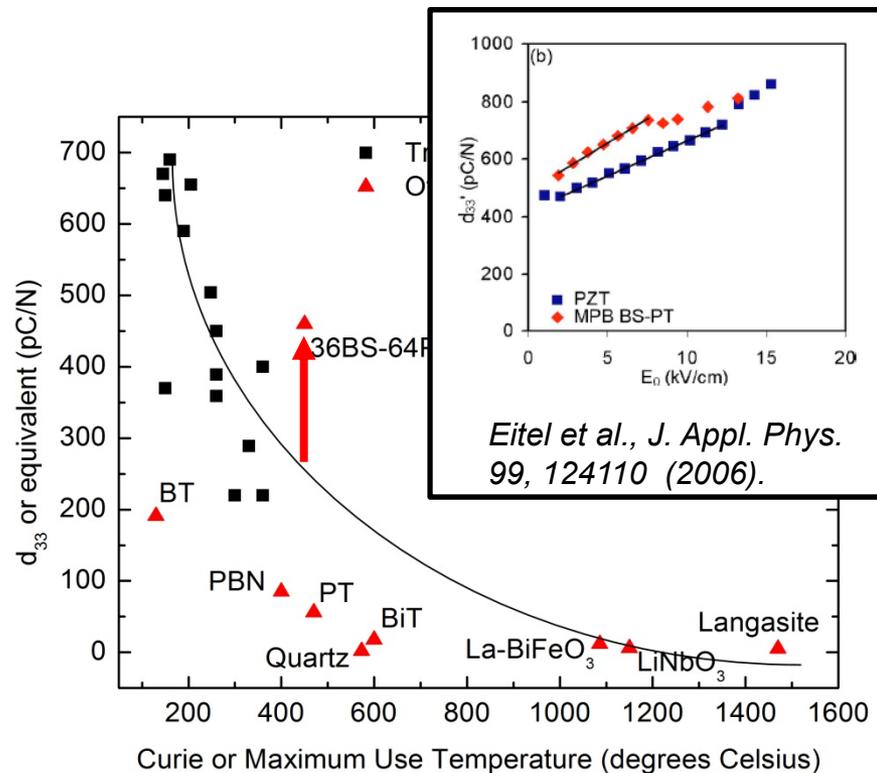
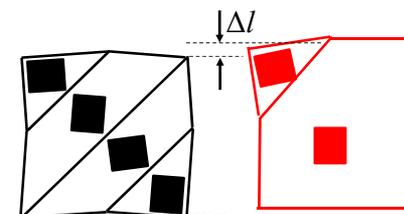
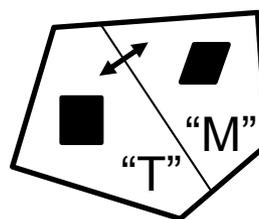


FIG. 3. Phase diagram for the perovskite $(1-x)\text{BiScO}_3\text{-}x\text{PbTiO}_3$ system representing revisions based on the TEM and single-crystal data.

*Mixture of monoclinic (Cm , ~50%) and tetragonal ($P4mm$, ~50%)



Eitel et al., *J. Appl. Phys.* 99, 124110 (2006).

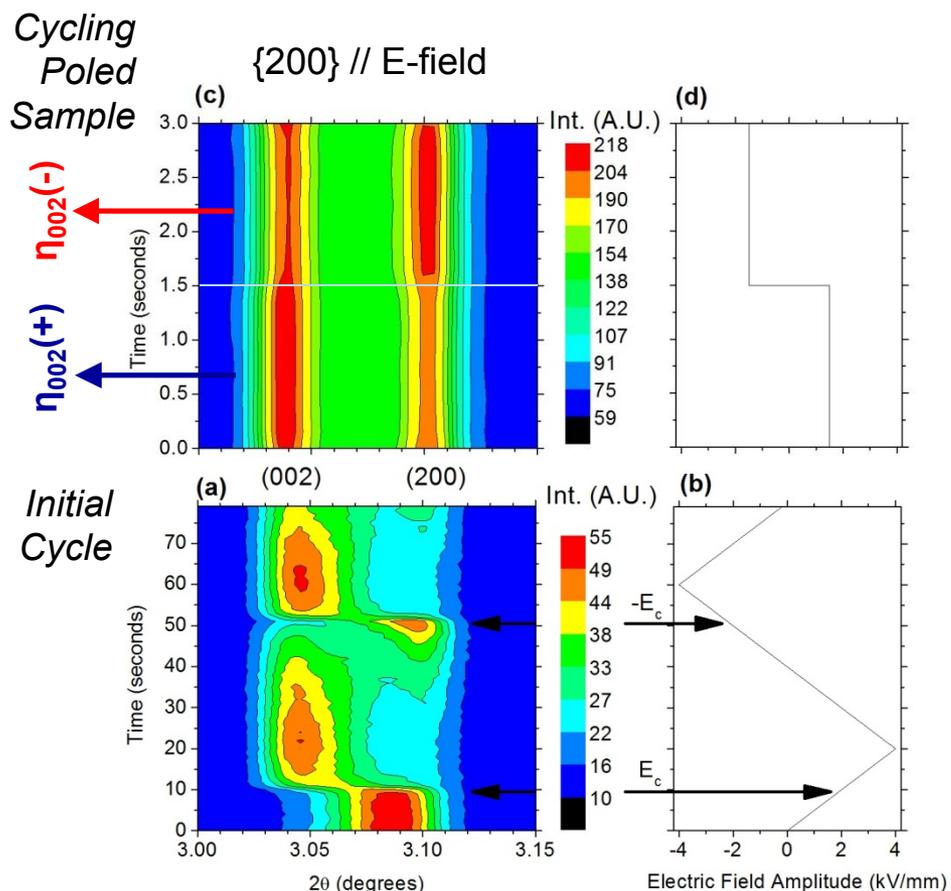
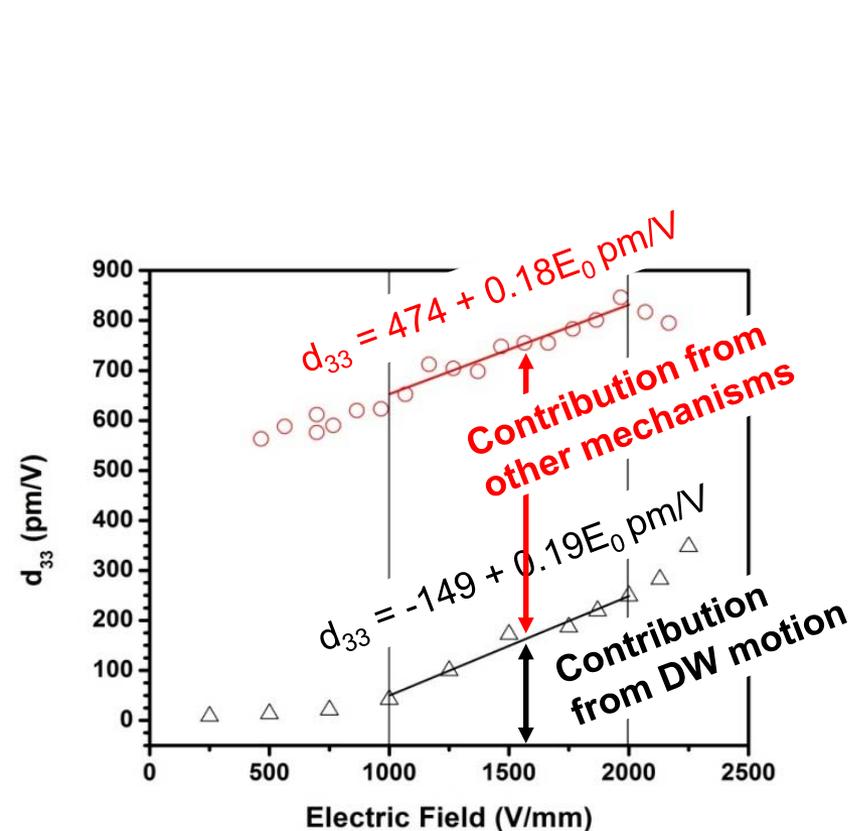


Eitel et al., *J. Appl. Phys.*, 96, 5, 2828+ (2004).

*Kim et al., *J. Appl. Phys.* 105, 114101 (2009).

High-Temperature $.36\text{BiScO}_3\text{-}.64\text{PbTiO}_3$

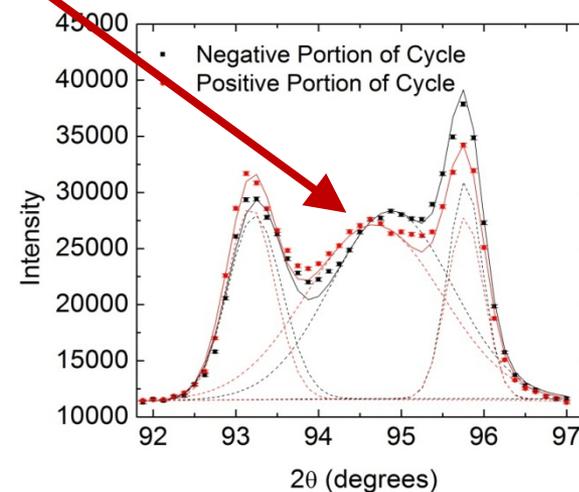
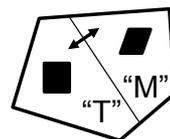
Domain wall motion in tetragonal phase



High-Temperature $.36\text{BiScO}_3\text{-}.64\text{PbTiO}_3$

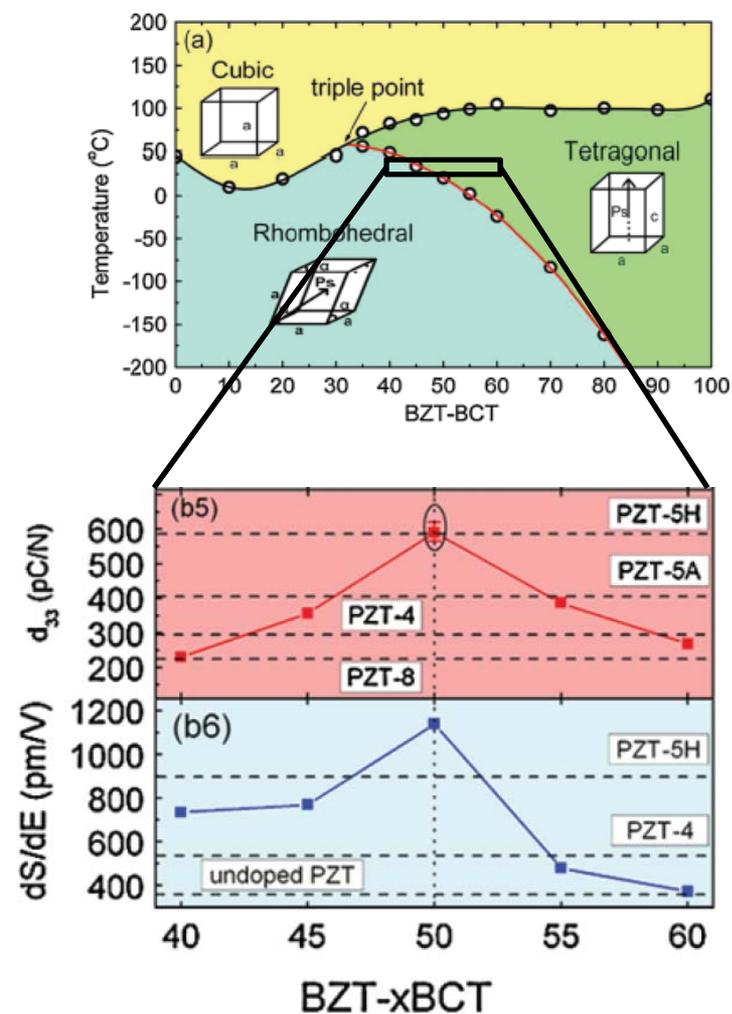
In related work,* we also showed:

- Domain wall motion in monoclinic phase of this two-phase mixture is *high*,
- Extent of interphase boundary motion (i.e., phase switching between M and T phases) is *low*, and
- Electric-field-induced piezoelectric strains are *frequency independent* and correlate with single crystal piezoelectric coefficients.



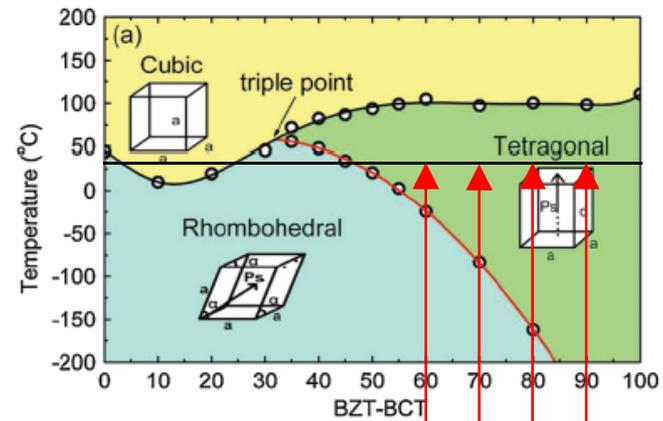
Ba(Zr_{0.2}Ti_{0.8})O₃-x(Ba_{0.7}Ca_{0.3})TiO₃

- Pb-free, with d_{33} up to 620 pC/N at room temperature
- (albeit with T_c of 93°C)

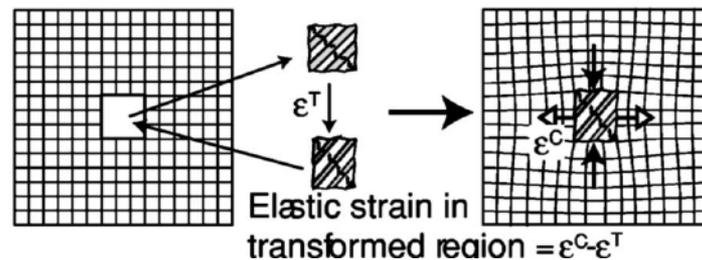
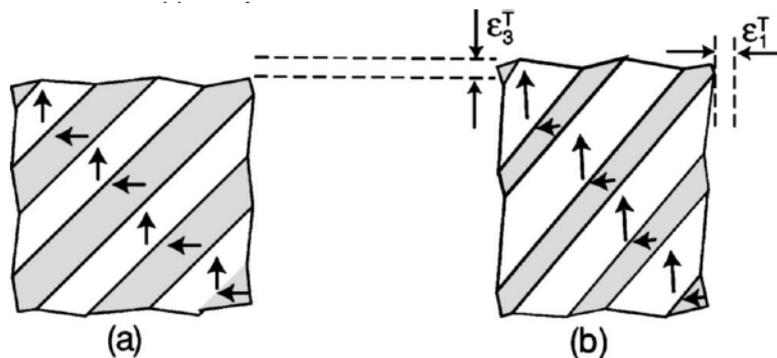


Ba(Zr_{0.2}Ti_{0.8})O₃-x(Ba_{0.7}Ca_{0.3})TiO₃

- Lattice aspect ratio decreases towards MPB
- **Test the developing hypothesis that domain wall motion *increases* with *decreasing* lattice aspect ratio due to *less* strain incompatibility (grain-grain interactions).**

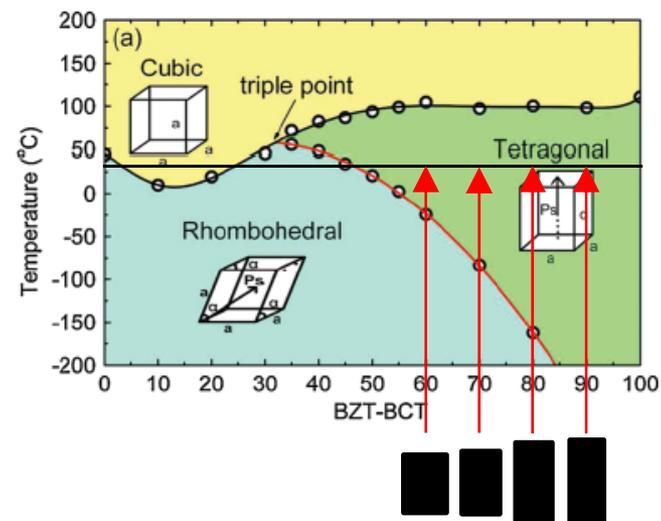


	60BCT	70BCT	80BCT	90BCT
c/a	1.0054	1.0067	1.0075	1.0079

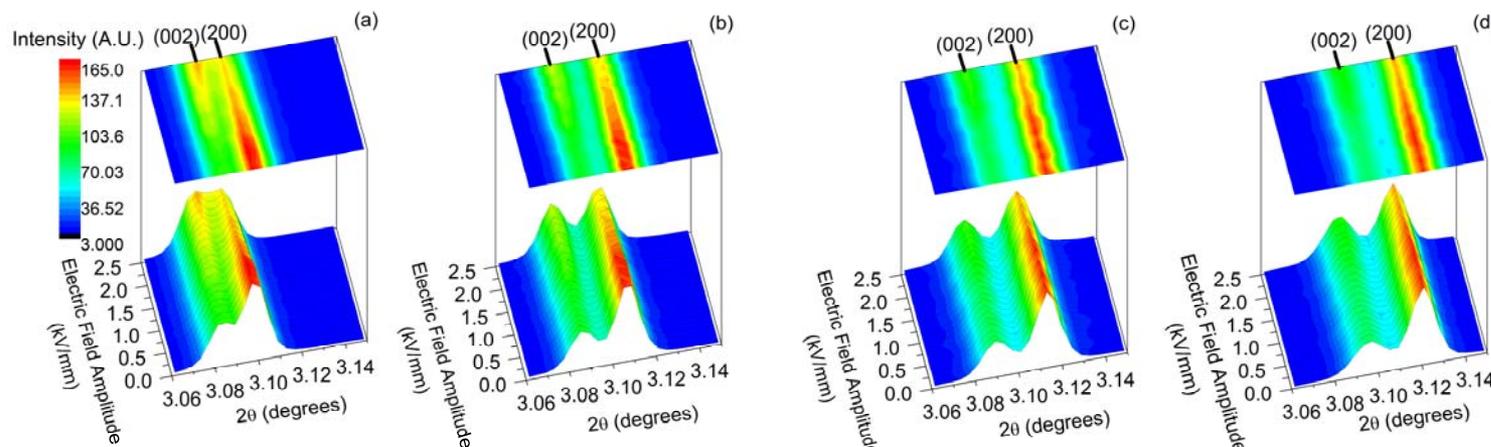


Ba(Zr_{0.2}Ti_{0.8})O₃-x(Ba_{0.7}Ca_{0.3})TiO₃

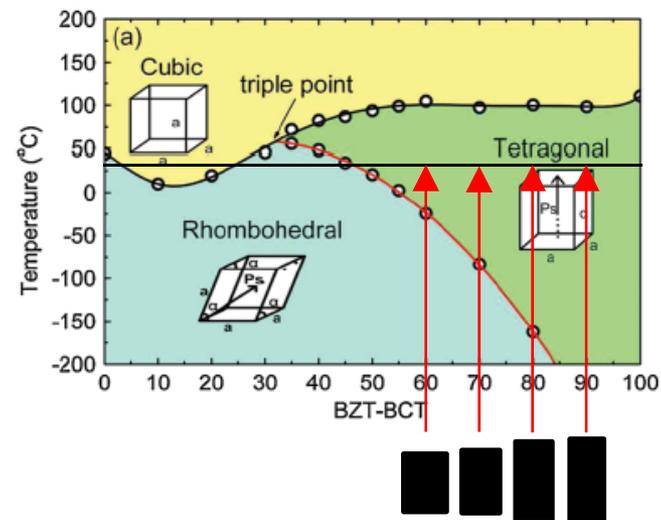
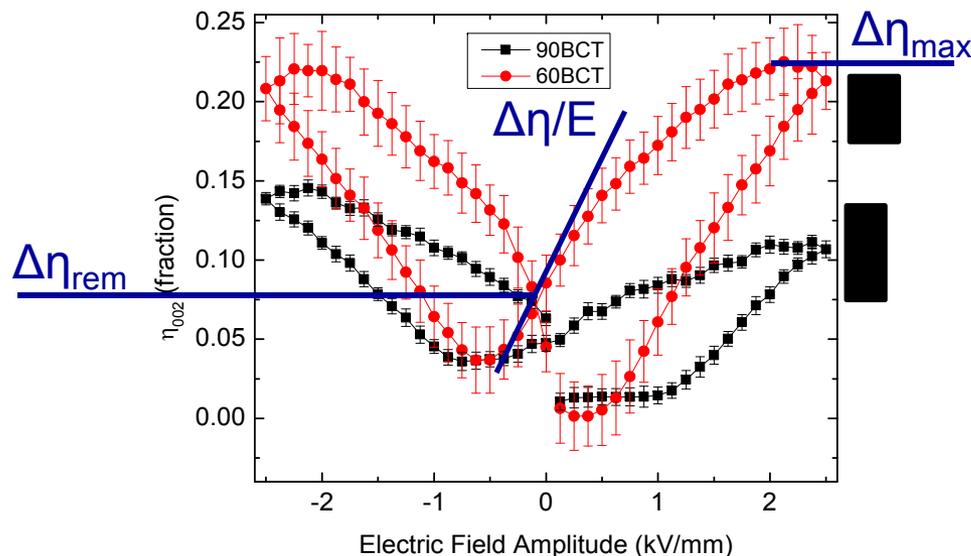
- Lattice aspect ratio decreases towards MPB
- **Test the developing hypothesis that domain wall motion *increases* with decreasing lattice aspect ratio due to less strain incompatibility (grain-grain interactions).**



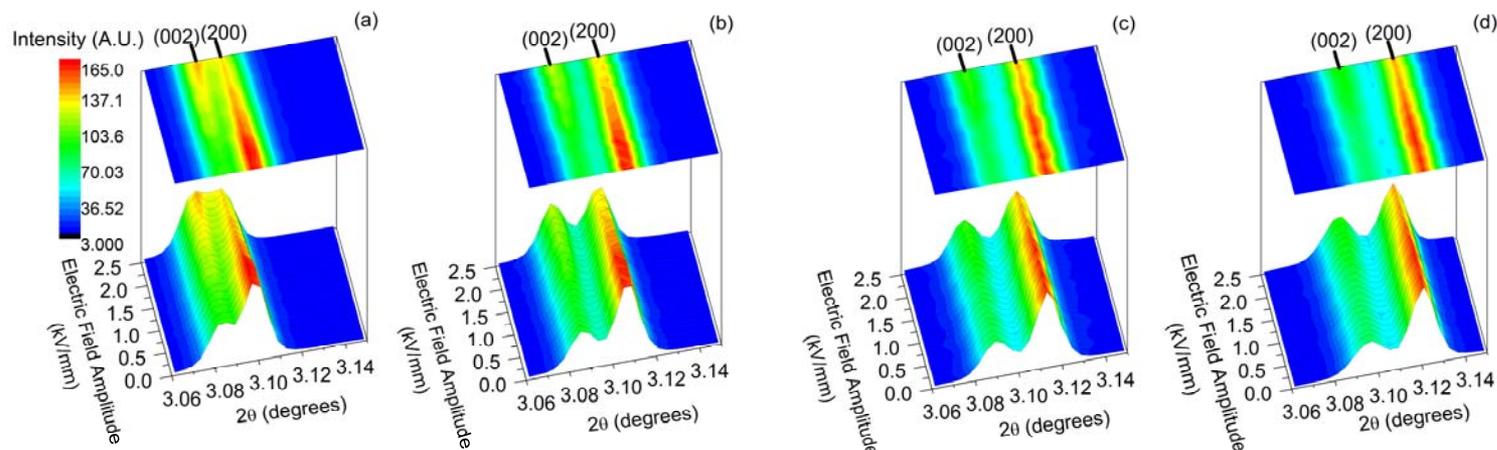
	60BCT	70BCT	80BCT	90BCT
c/a	1.0054	1.0067	1.0075	1.0079



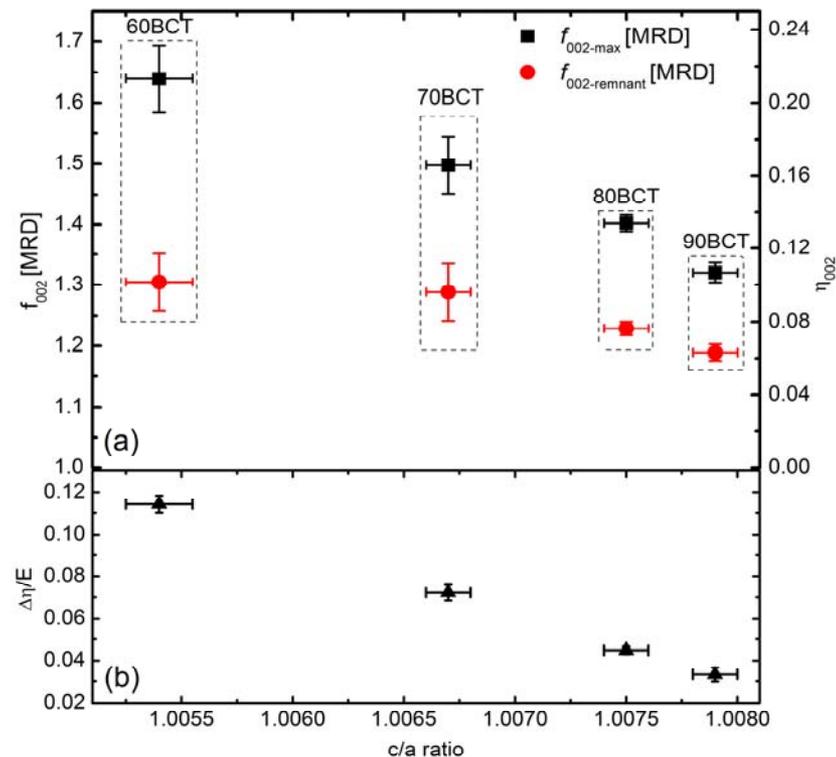
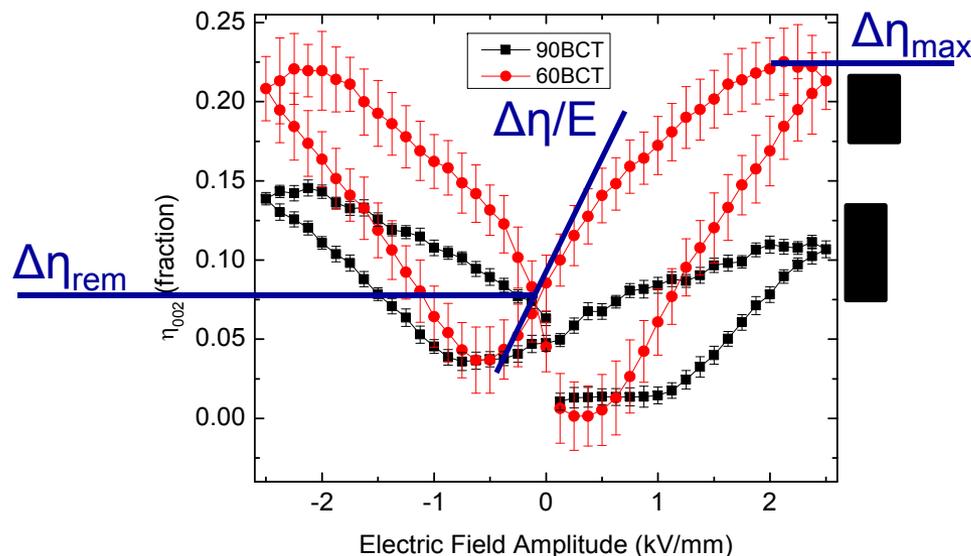
Ba(Zr_{0.2}Ti_{0.8})O₃-x(Ba_{0.7}Ca_{0.3})TiO₃



	60BCT	70BCT	80BCT	90BCT
c/a	1.0054	1.0067	1.0075	1.0079



Ba(Zr_{0.2}Ti_{0.8})O₃-x(Ba_{0.7}Ca_{0.3})TiO₃



Take-home Message:

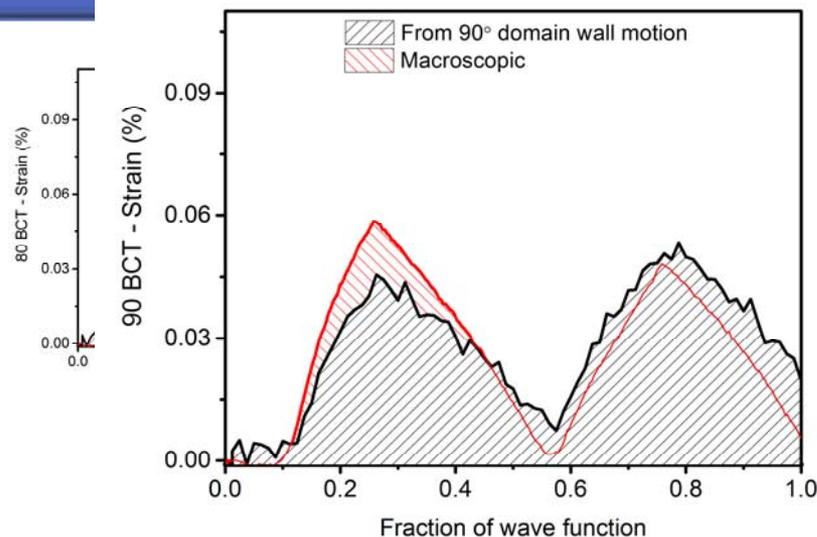
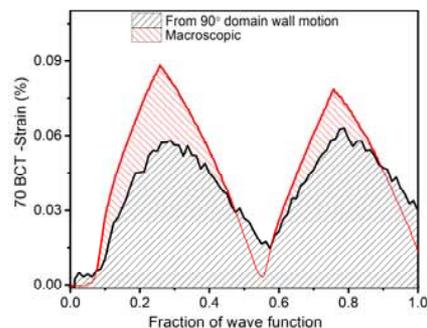
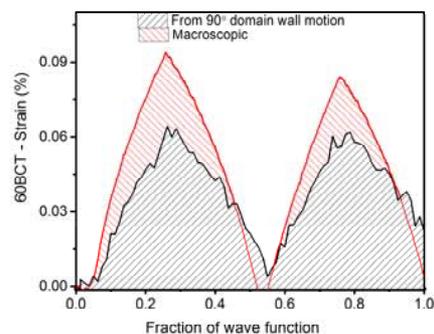
- Extent of domain wall motion inversely related to c/a .

$\text{Ba}(\text{Zr}_{0.2}\text{Ti}_{0.8})\text{O}_3 - x(\text{Ba}_{0.7}\text{Ca}_{0.3})\text{TiO}_3$

- Measured at all angles to the field direction, enabling a calculation of contribution of non-180° DW motion to macroscopic strain

Take-home Message:

- Non-180° domain wall motion constitutes majority of macroscopic response.*
- Absolute contribution from non-180° DW motion increases as MPB is approached.*
- Additional contributions from other effects also increase as MPB is approached.*



Grain size effects in BaTiO₃

Materials
Views

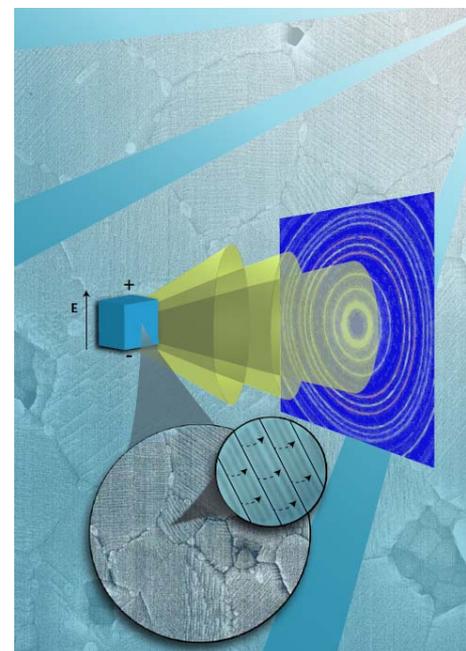
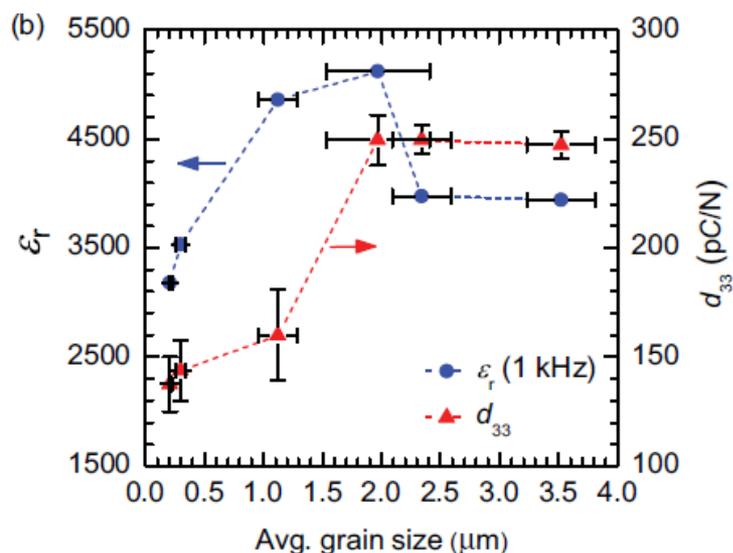
www.MaterialsViews.com

ADVANCED
FUNCTIONAL
MATERIALS

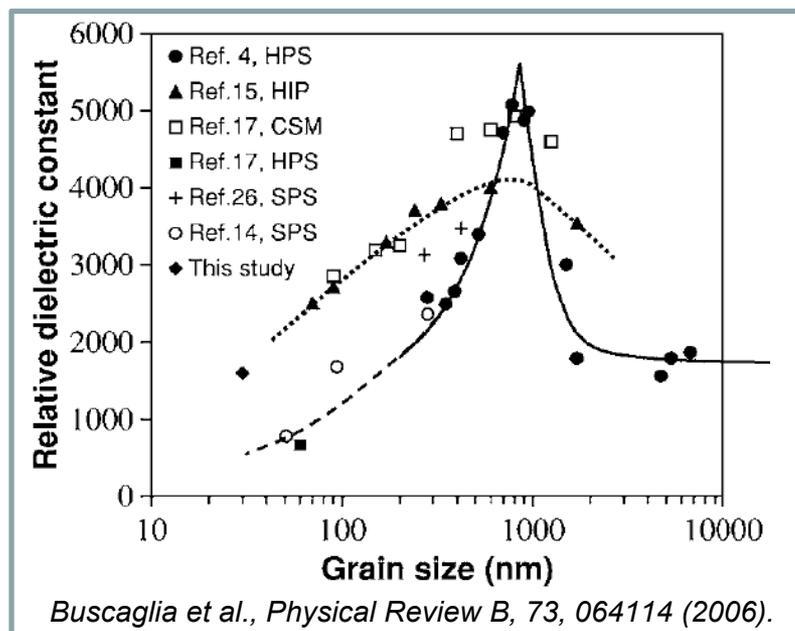
www.afm-journal.de

Domain Wall Displacement is the Origin of Superior Permittivity and Piezoelectricity in BaTiO₃ at Intermediate Grain Sizes

Dipankar Ghosh, Akito Sakata, Jared Carter, Pam A. Thomas, Hyuksu Han, Juan C. Nino, and Jacob L. Jones*

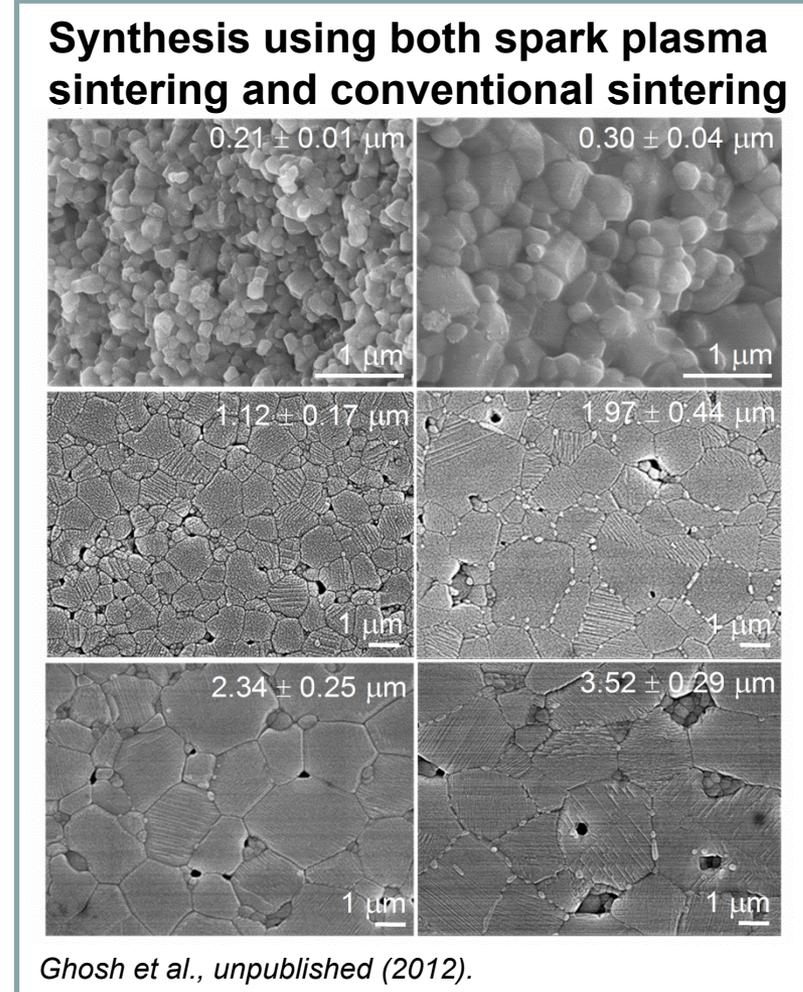
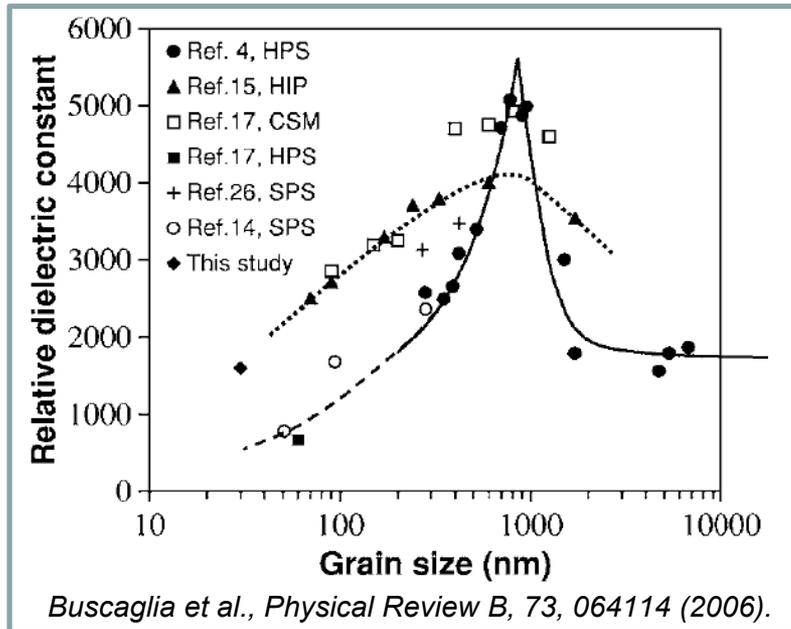


Grain size effects in BaTiO₃

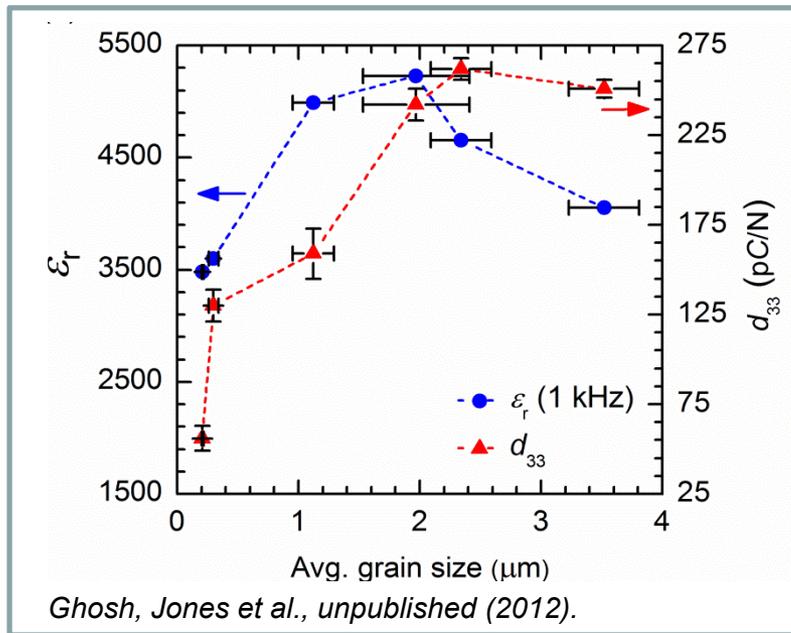


- Known since 1960's:
 - Peak in permittivity at $\sim 1 \mu\text{m}$
 - 90° domain sizes decrease down to $\sim 1 \mu\text{m}$, then single domain below $\sim 1 \mu\text{m}$.
- Competing theories:
 - Near $\sim 1 \mu\text{m}$, grains become single ferroelastic domain. This increases internal **residual stress** and pushes the structures towards the paraelectric phase with a commensurate increase in ϵ_r (Buessem *et al.*, 1966)
 - **Domain walls** exhibit greatest mobility at $\sim 1 \mu\text{m}$, leading to the large extrinsic contribution to ϵ_r (Arlt *et al.*, 1985)

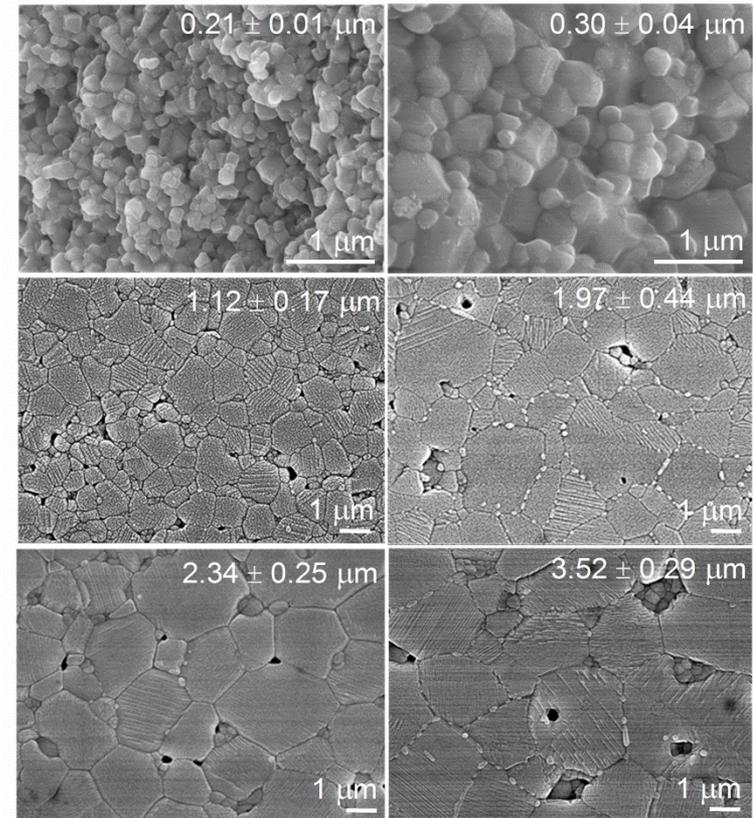
Grain size effects in BaTiO₃



Grain size effects in BaTiO₃

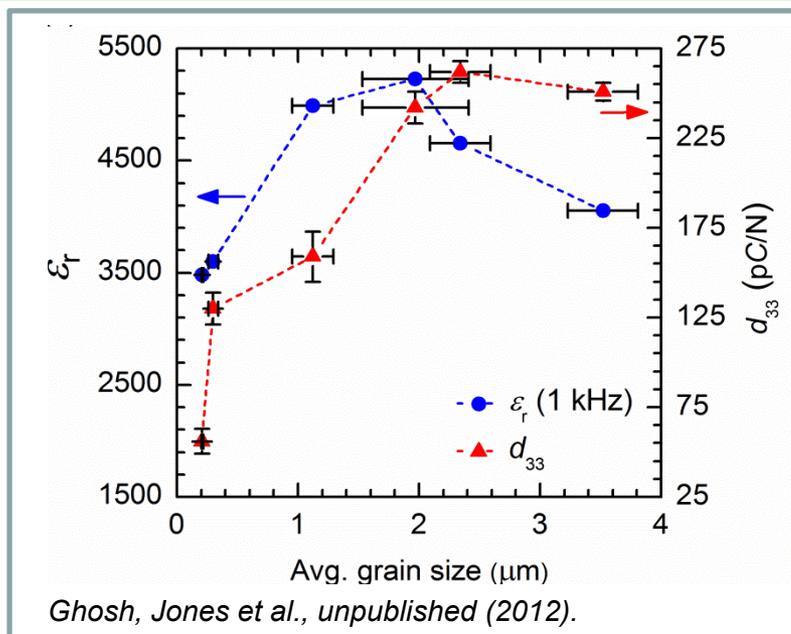


Synthesis using both spark plasma sintering and conventional sintering

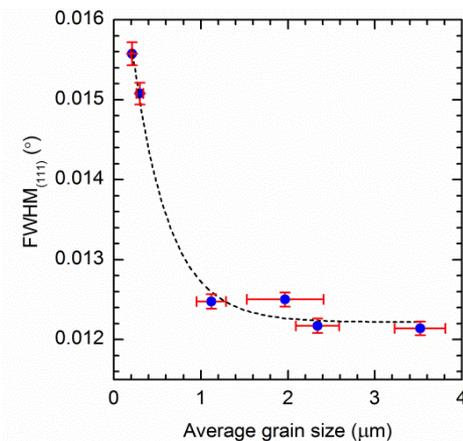
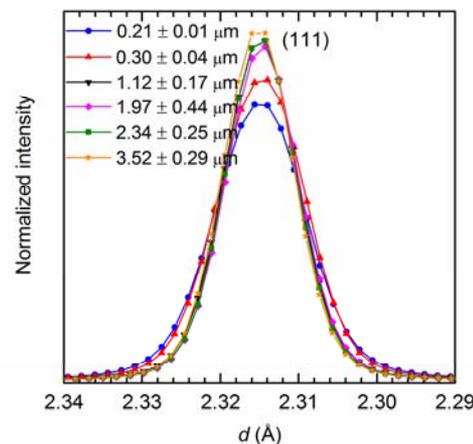


Ghosh et al., unpublished (2012).

Grain size effects in BaTiO₃



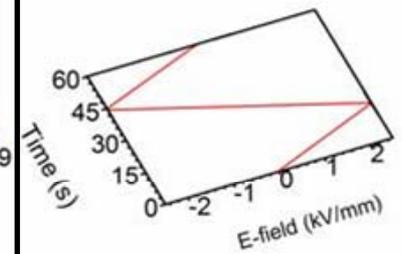
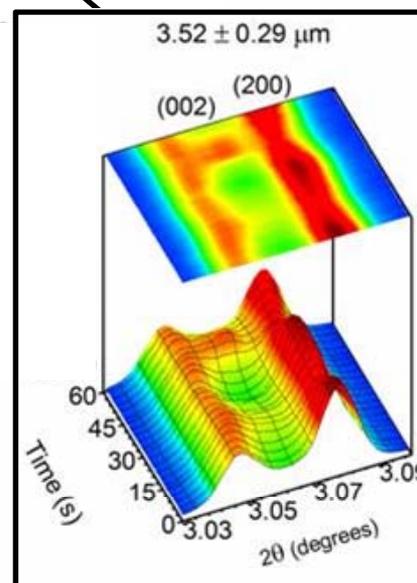
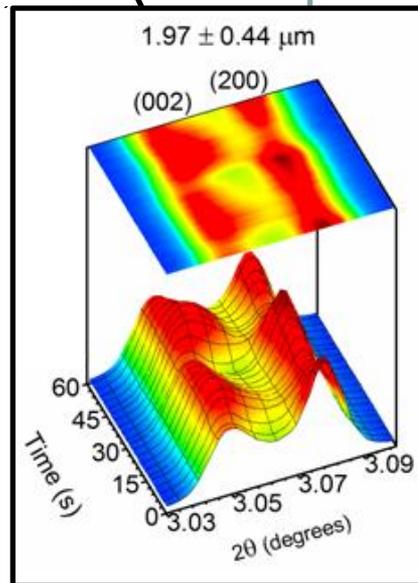
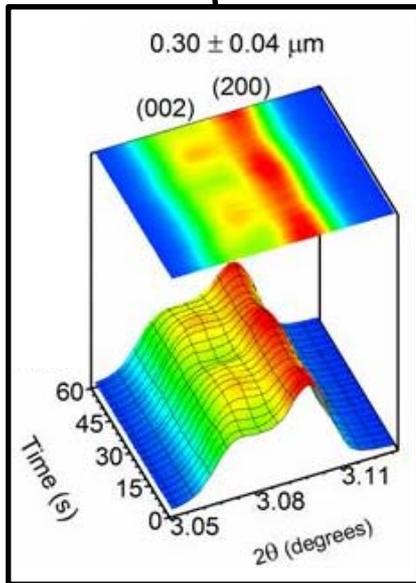
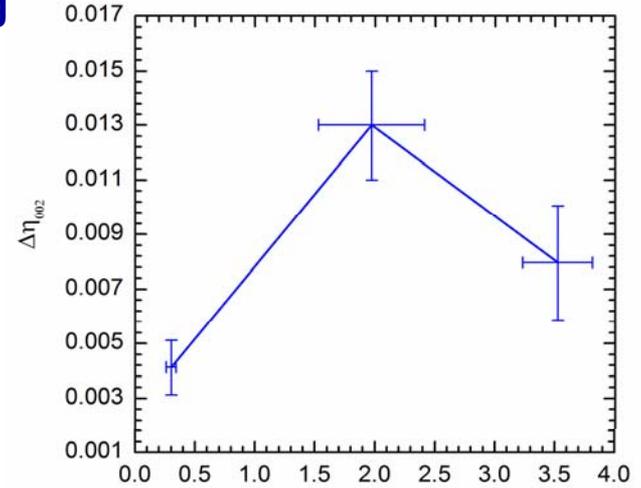
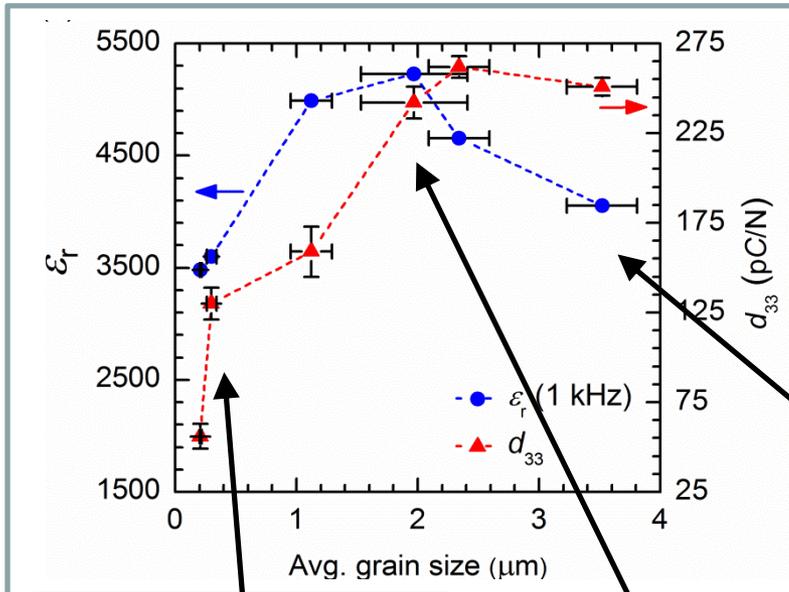
Residual strain and crystallite size effects



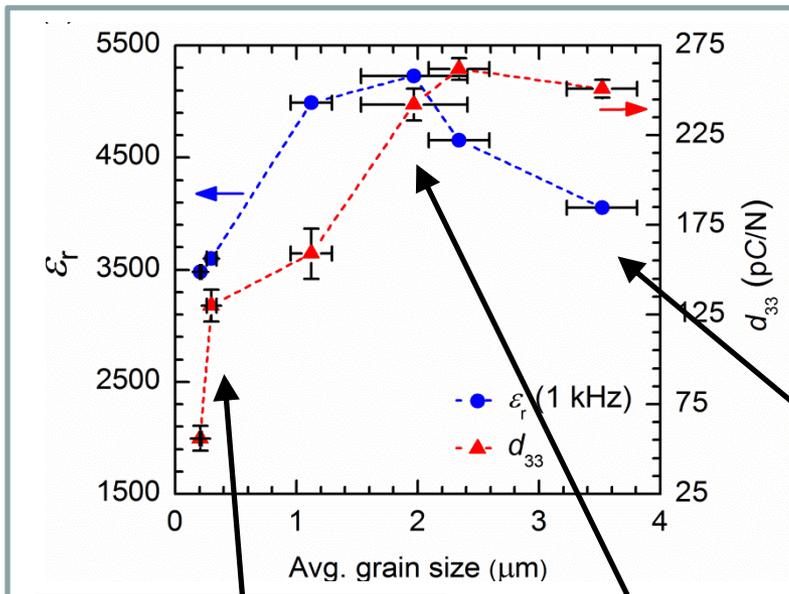
- No macrostrain (i.e., residual stress)
- Either microstrain (i.e., distribution of lattice spacings) or crystallite broadening.

Grain size effects in BaTiO₃

➤ Domain wall motion during weak field cycling

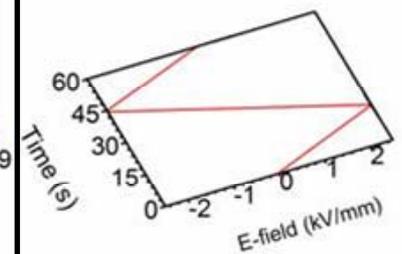
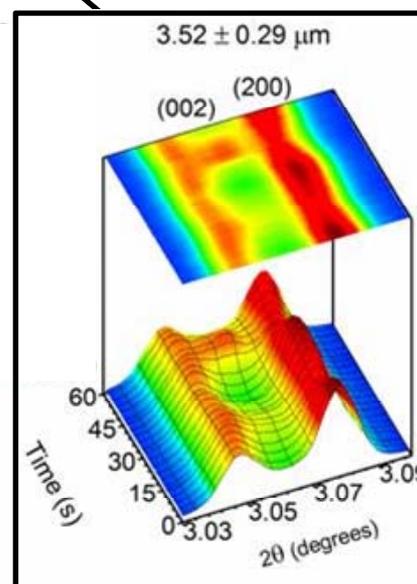
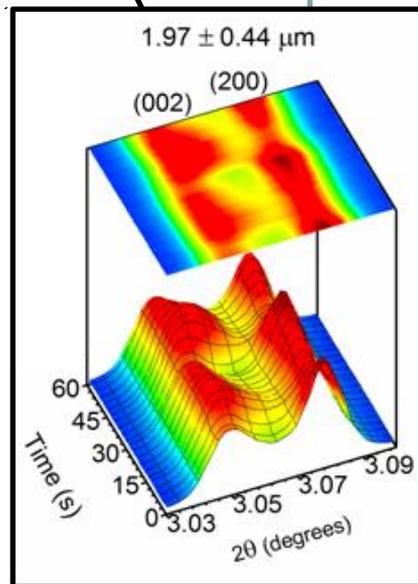
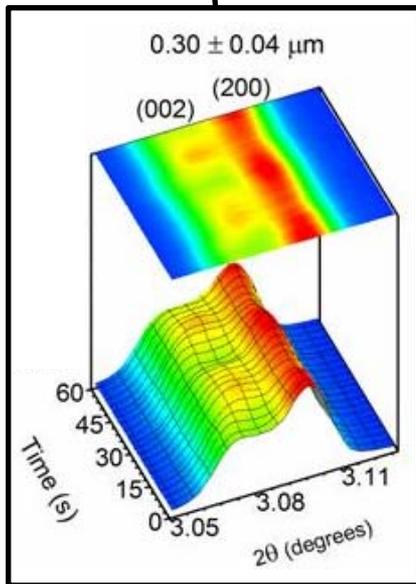


Grain size effects in BaTiO₃



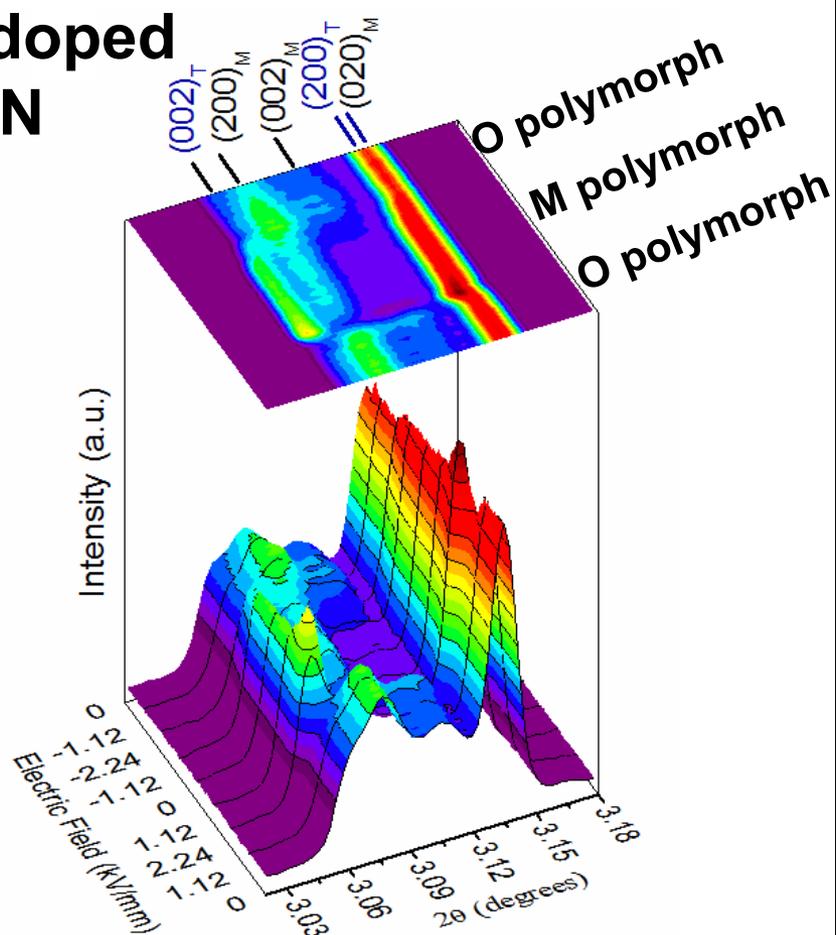
Take-home Message:

- Domain wall motion correlated with enhanced properties.
- Confirms domain wall motion theory is dominant origin of d_{33} and ϵ_r peaks at $\sim 1 \mu\text{m}$ grain size in BaTiO₃ (theory of Arlt *et al.*, 1985)



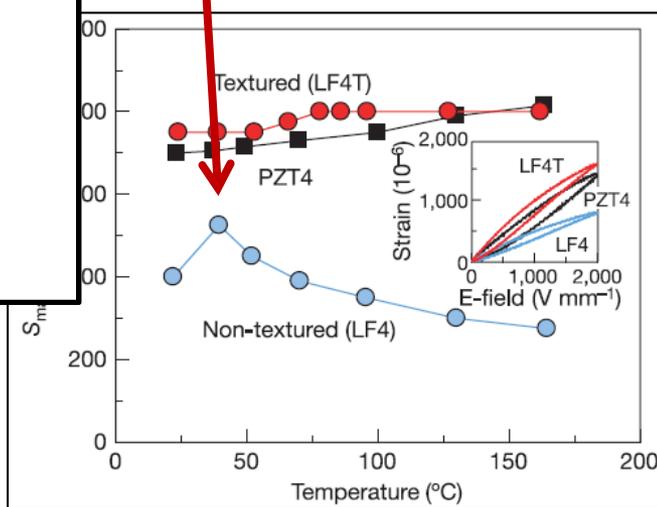
Phase transitions in $\text{Na}_{0.5}\text{K}_{0.5}\text{NbO}_3$

Li-doped
NKN



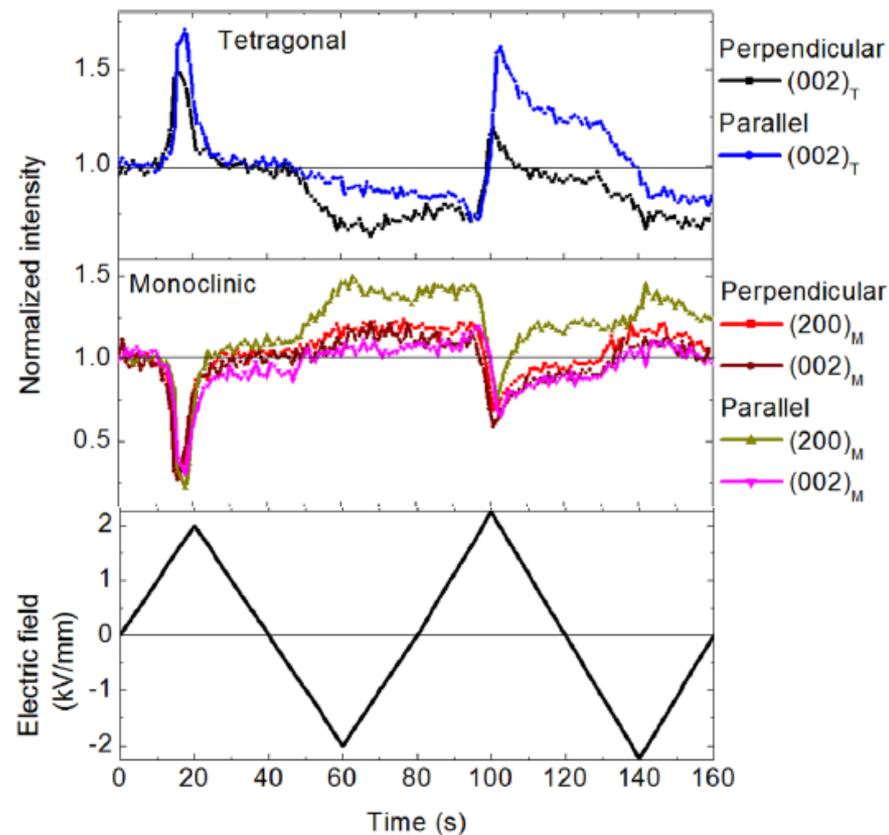
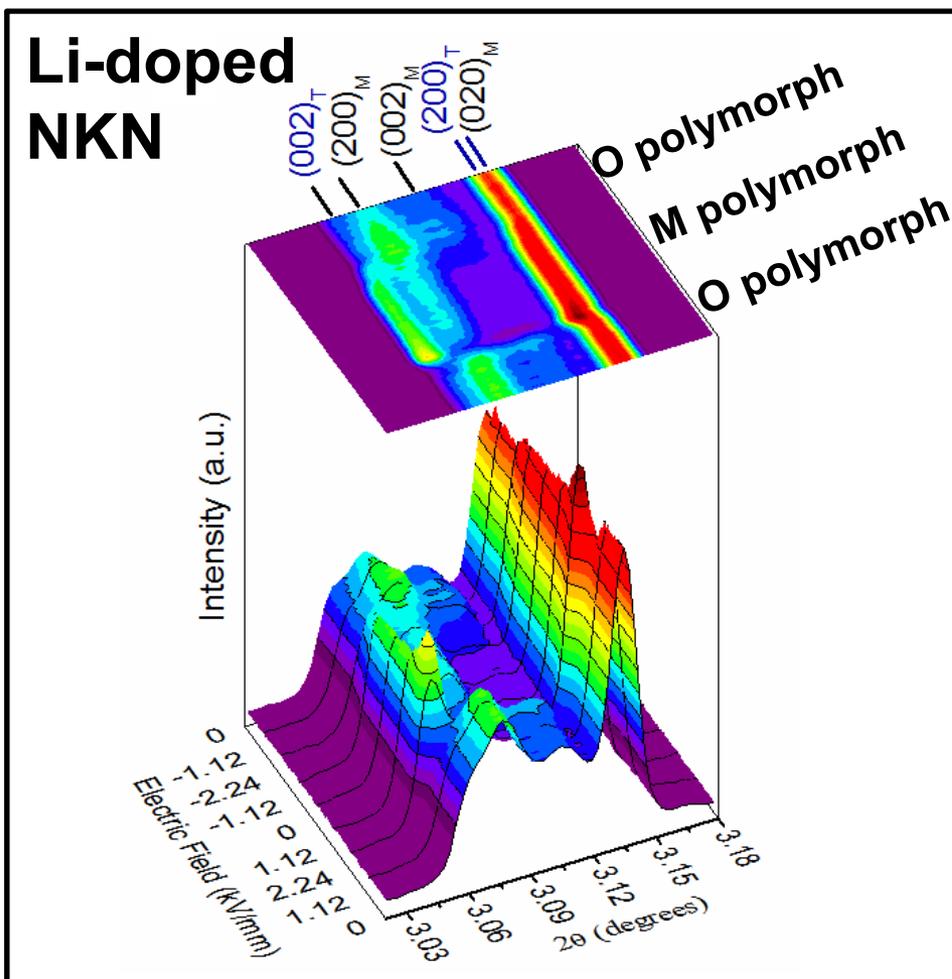
“It is considered that this anomaly is related to the **electric-field-induced tetragonal-to orthorhombic** phase transformation, shifting the phase transformation temperature from below 25°C in the unpoled specimen to 40°C under a high electric field. In order to clarify the origin of this anomaly, morphotropic phases need to be determined under high electric-field driving.”

- Saito et al., Nature (2004)



lamsasri, Jones et al., J. Appl. Phys., 117, 024101 (2015).

Phase transitions in $\text{Na}_{0.5}\text{K}_{0.5}\text{NbO}_3$



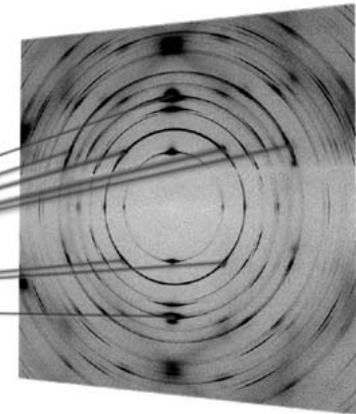
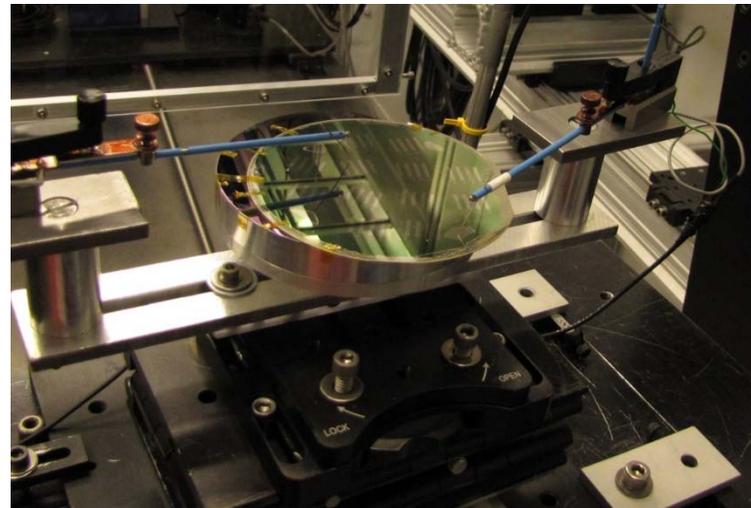
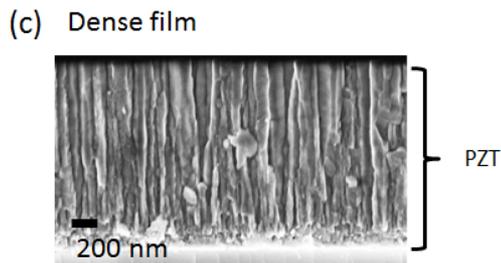
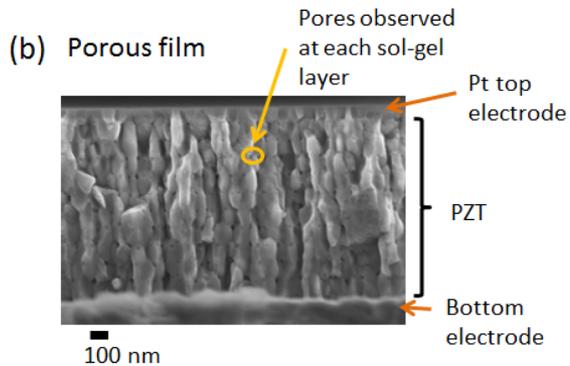
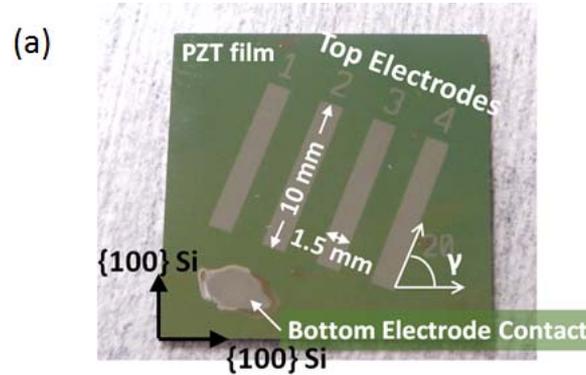
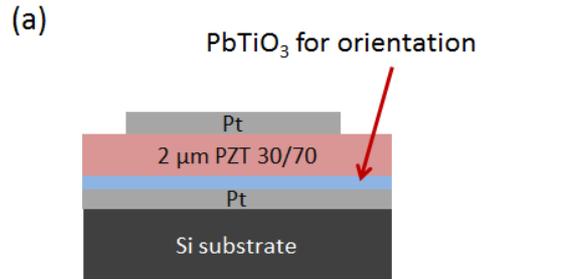
Take-home Message:

- Dramatic phase switching present, and field-dependent.

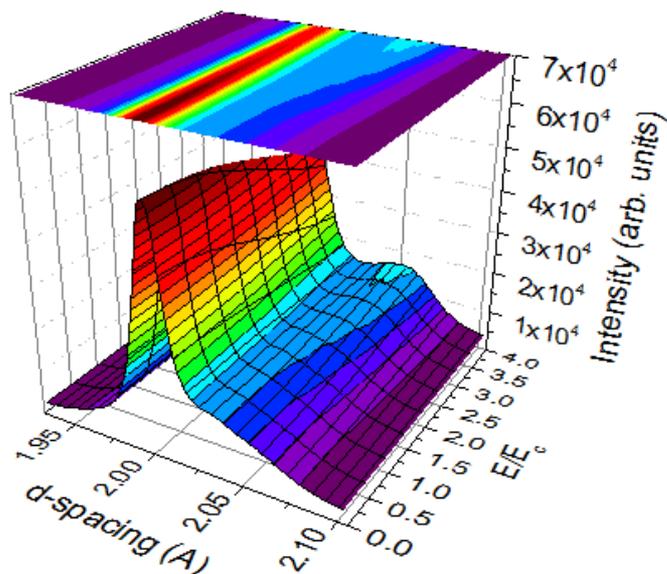


PbZr_{0.3}Ti_{0.7}O₃ Thin Films

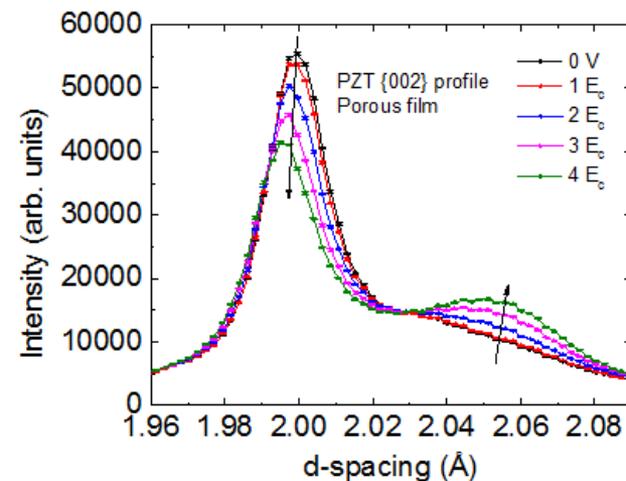
001-oriented, solution deposition from 2-MOE.



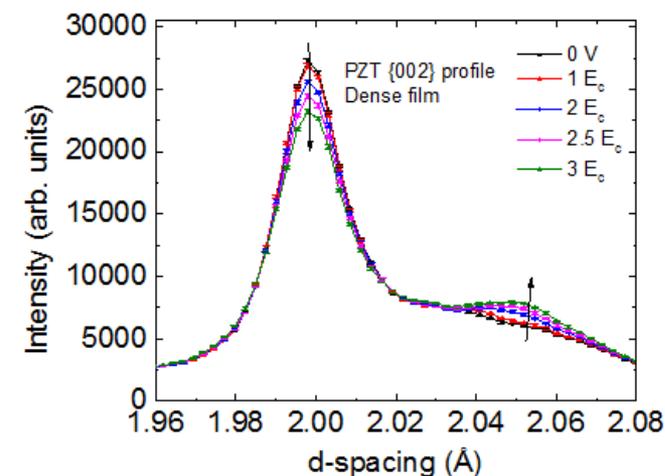
PbZr_{0.3}Ti_{0.7}O₃ Thin Films



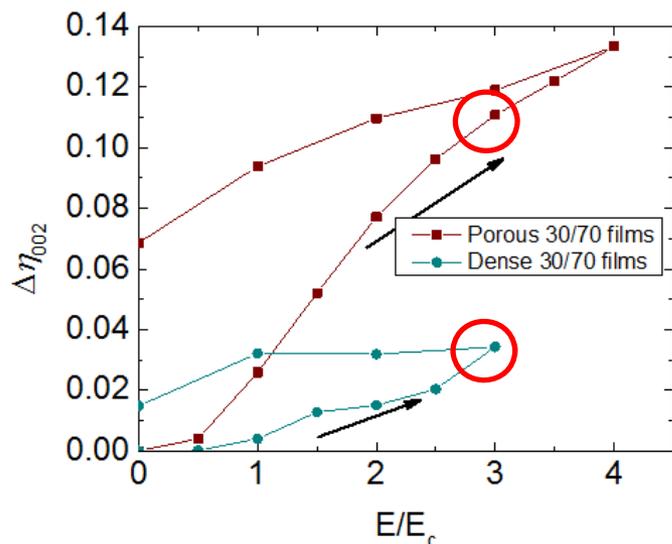
Porous:
More 90° domain wall motion and (002) lattice strain



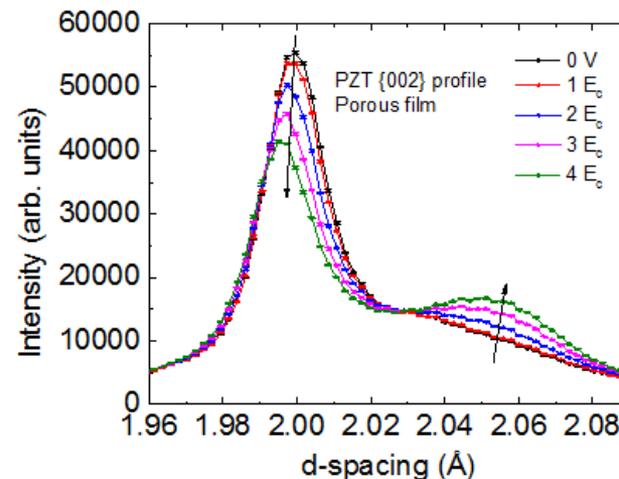
Dense:
Less 90° domain wall motion and (002) lattice strain



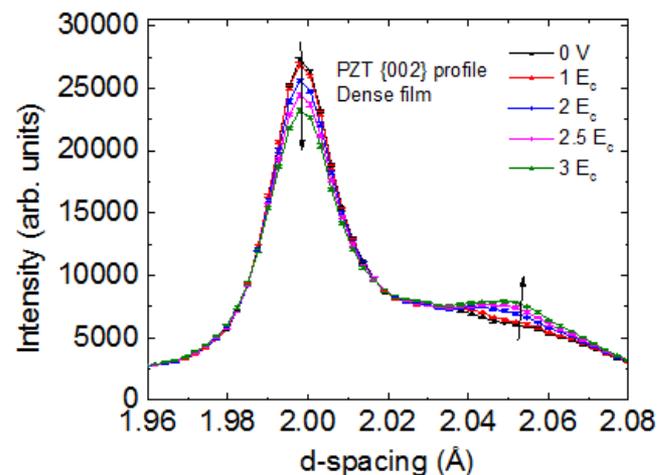
PbZr_{0.3}Ti_{0.7}O₃ Thin Films



Porous:
More 90° domain wall motion and (002) lattice strain



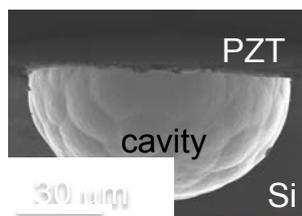
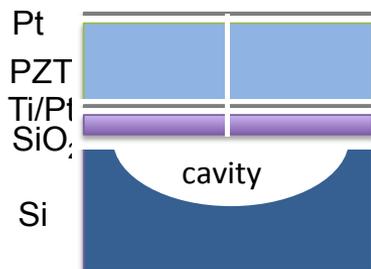
Dense:
Less 90° domain wall motion and (002) lattice strain



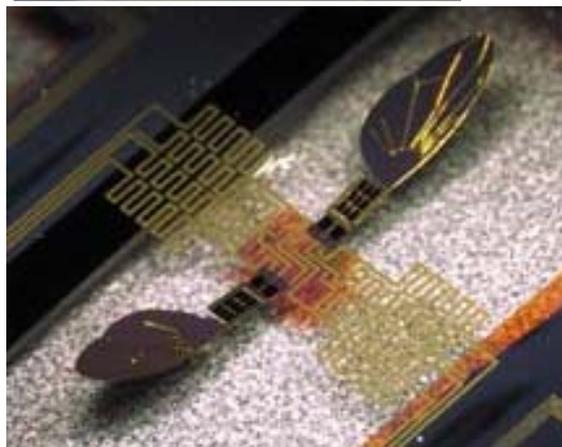
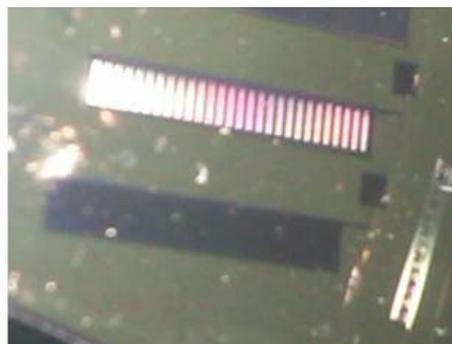
Take-home Message:

- Porous films have **~threefold** increase in DW motion relative to dense films.

PbZr_{0.3}Ti_{0.7}O₃ Thin Films

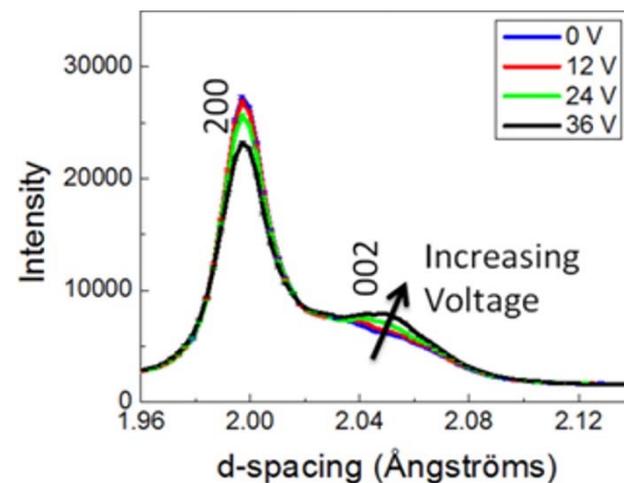


Cross-section of released PZT diaphragm



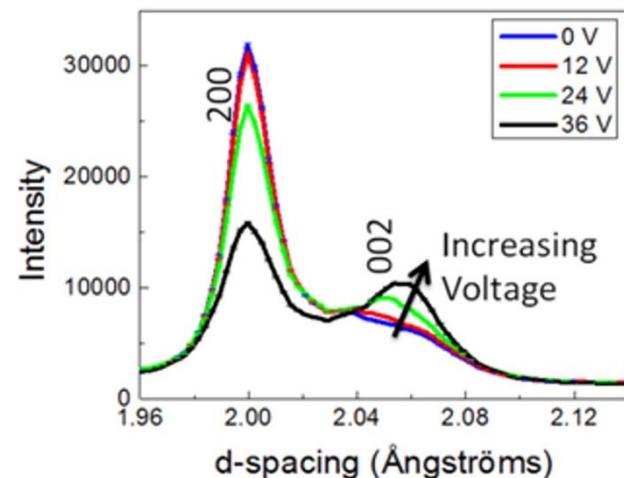
Clamped (control):

Less 90° domain wall motion and (002) lattice strain



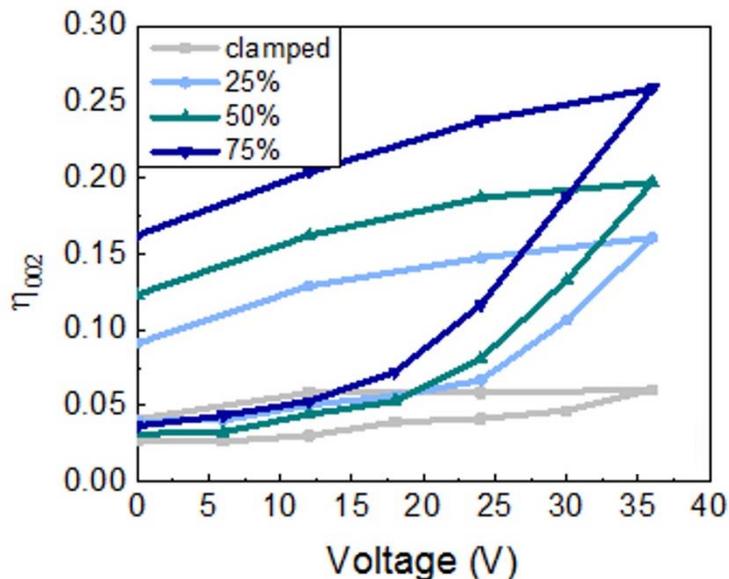
75% released:

More 90° domain wall motion and (002) lattice strain



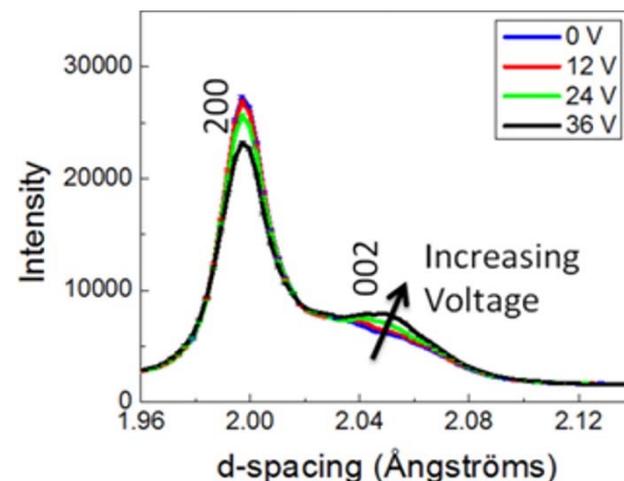


PbZr_{0.3}Ti_{0.7}O₃ Thin Films



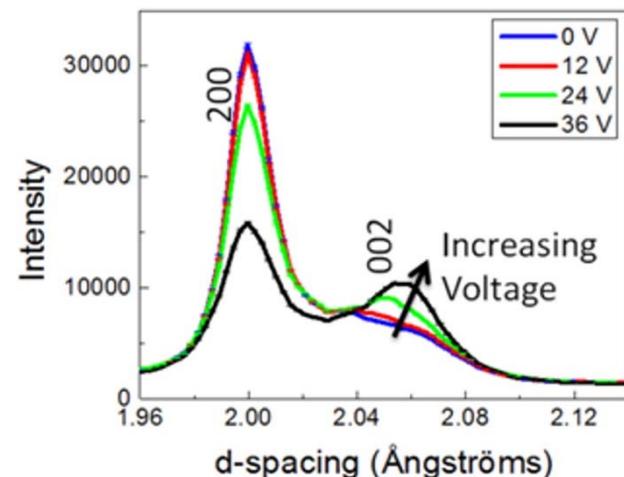
Clamped (control):

Less 90° domain wall motion and (002) lattice strain



75% released:

More 90° domain wall motion and (002) lattice strain



Take-home Message:

- Domain wall motion can be increased *~fivefold* if released from substrate.

Other experiments in this area (not shown today)

- **Much more** on polycrystalline **PZT**
- DW motion in **orthorhombic** $\text{Na}_{0.5}\text{K}_{0.5}\text{NbO}_3$
- $\text{Na}_{0.5}\text{Bi}_{0.5}\text{TiO}_3$ - $x\text{BaTiO}_3$ as a function of **x**
- **Frequency dispersion** of DW motion in $(1-x)\text{BaTiO}_3$ - $x\text{Bi}(\text{Zn}_{1/2}\text{Ti}_{1/2})\text{O}_3$ and **PZT**
- **Time-dependence** of domain switching and intermediate ferroelastic states during polarization reversal in **PZT**
- **Stress-dependence** of domain reorientation during stress and near crack tips in **PZT**

North Carolina State University



Main Campus

- *Raleigh, NC*
- *NC's main institution for Engineering*
- *Established 1887*
- *~35,000 students*

Centennial Campus

- *Built mostly within last 5-10 years*
- *1,227 acres*
- *65 Partners*
- *72 academic units*
- *34 buildings*

North Carolina State University



LeBeau

M. Dickey

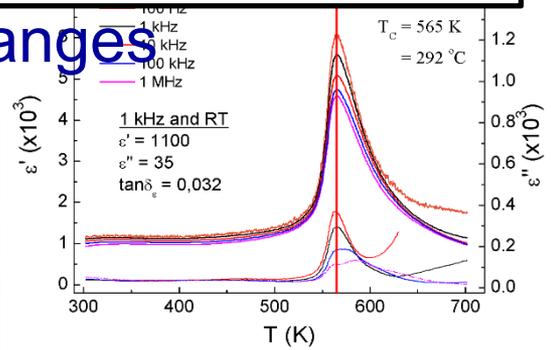
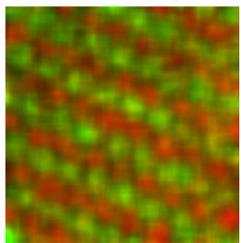
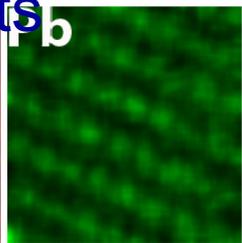
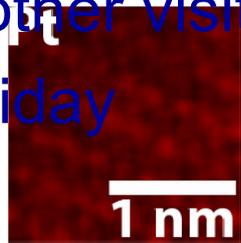


Consider Raleigh / NC State for:

- Conferences
- Sabbaticals, Research Exchanges or other visits



analytical instrumentation facility



Origins of Electromechanical Strain Ascertained from *in situ* X-ray and Neutron Diffraction

Jacob L. Jones

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