

Core-shell Domain Structure Investigation of Lead-free Incipient Piezoceramic by Piezoresponse Force Microscopy



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Outline



1. Motivation

- $\text{Bi}_{1/2}\text{Na}_{1/2}\text{TiO}_3$ -25SrTiO₃ lead-free incipient piezoceramic
- Core-shell microstructure

2. Piezoresponse Force Microscopy

- Domain imaging
- Domain switching

3. Results

- Visualization of non-ergodic core and ergodic shell domain structure
- Heterogeneous nucleation of domains at core/shell interface
- Topographical difference between the core and shell region

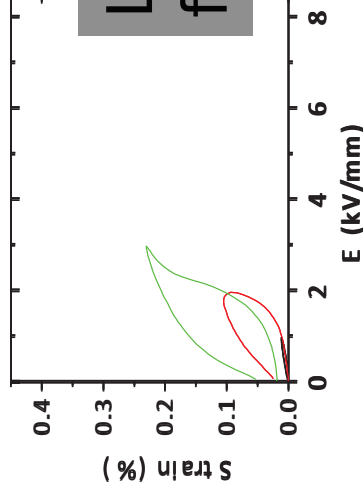
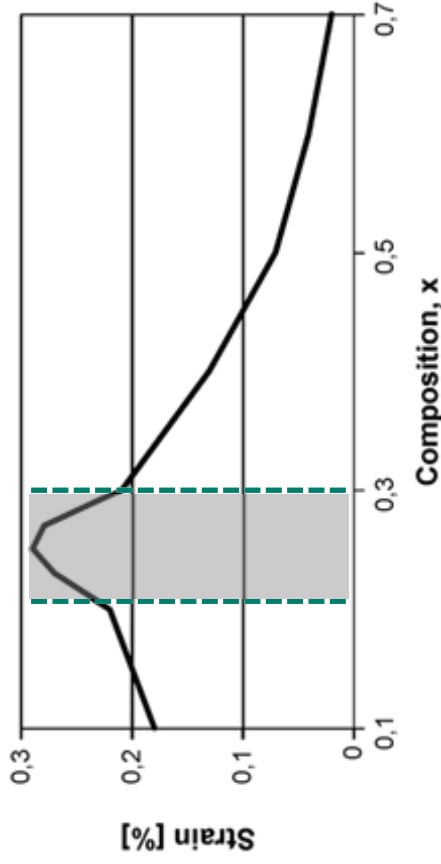
4. Conclusions

Motivation



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Lead-free incipient piezoceramic



Large electric-field-induced strain

Poster: P 01 by Matias Acosta

Jo, W., et al., Journal of Electroceramics 29(1), 71-93 (2012).
Eu-Directive 2011/65/Eu: (RoHS), Off. J. Eur. Union, L 174, 88-110 (2011).
Krauss et al. JECerS 30 (2010)



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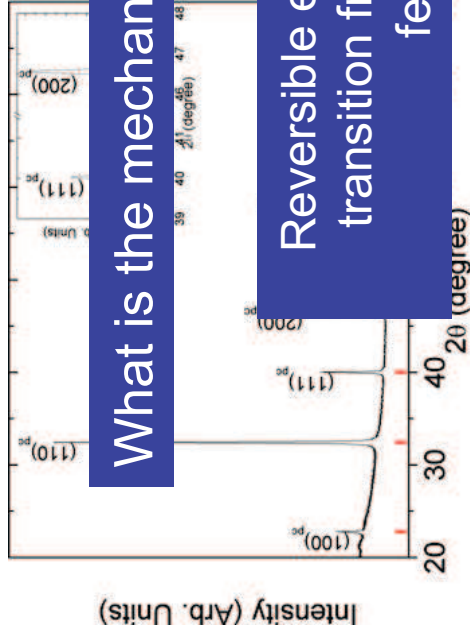


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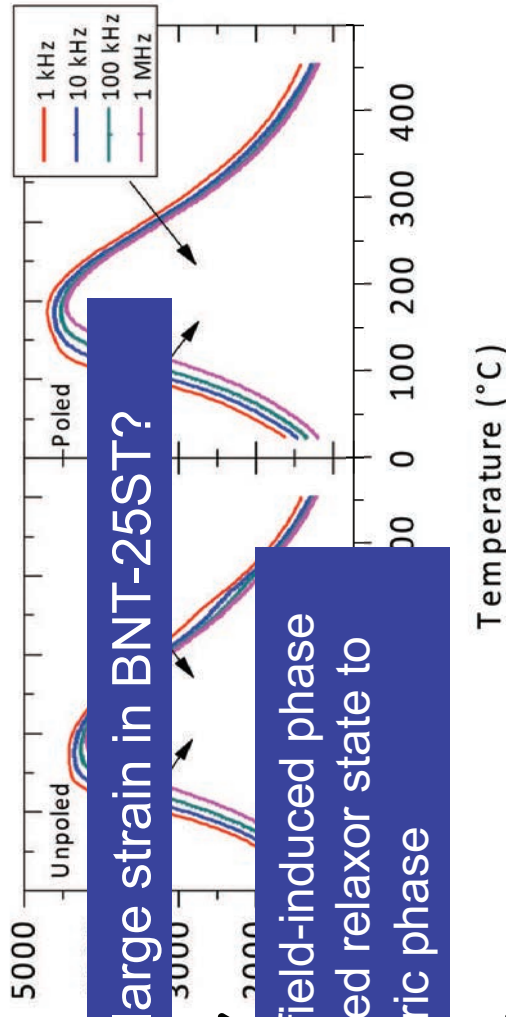
Motivation

$\text{Bi}_{1/2}\text{Na}_{1/2}\text{TiO}_3\text{-}25\text{SrTiO}_3$ lead-free incipient piezoceramic

XRD



Dielectric Properties



What is the mechanism of large strain in BNT-25ST?

Reversible electric-field-induced phase transition from mixed relaxor state to ferroelectric phase

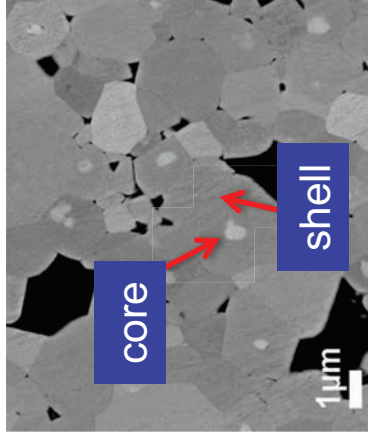
Core-shell microstructure
Macroscopically ferroelectric and Relaxor

Acosta, M., et al., *Journal of the American Ceramic Society* 97(6): 1937-1943. (2014)

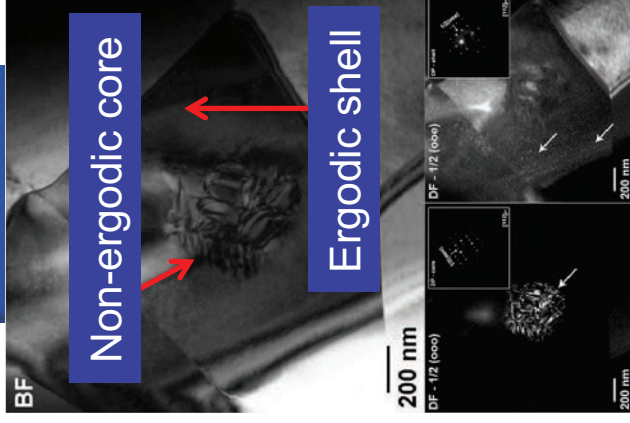
Motivation

Core-shell microstructure

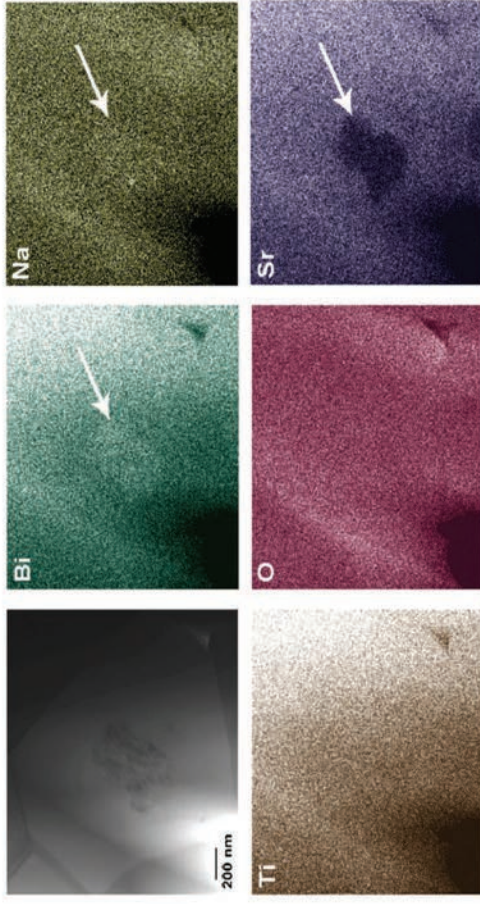
SEM-BSE



TEM



STEM



Nanoscale

PFM

Core: Sr-depleted/(Bi,Na)-rich
Shell: Sr-rich/(Bi,Na)-depleted

Courtesy of Acosta et al. and B3



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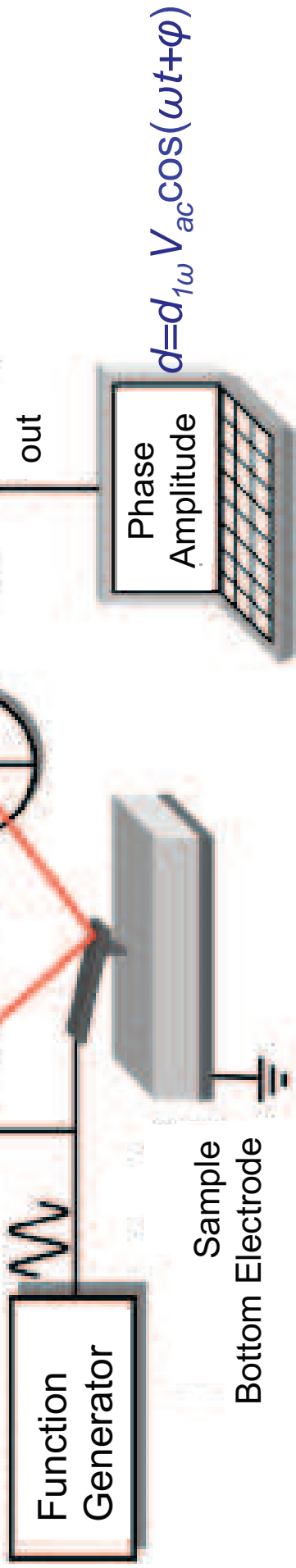
Piezoresponse Force Microscopy



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Domain imaging

$$V_{tip} = V_{ac} \cos(\omega t)$$



Domain switching

<http://www.ntmdt.com/spm-principles/>

Balke, N., et al., *Journal of the American Ceramic Society* 92(8): 1629-1647. (2009)

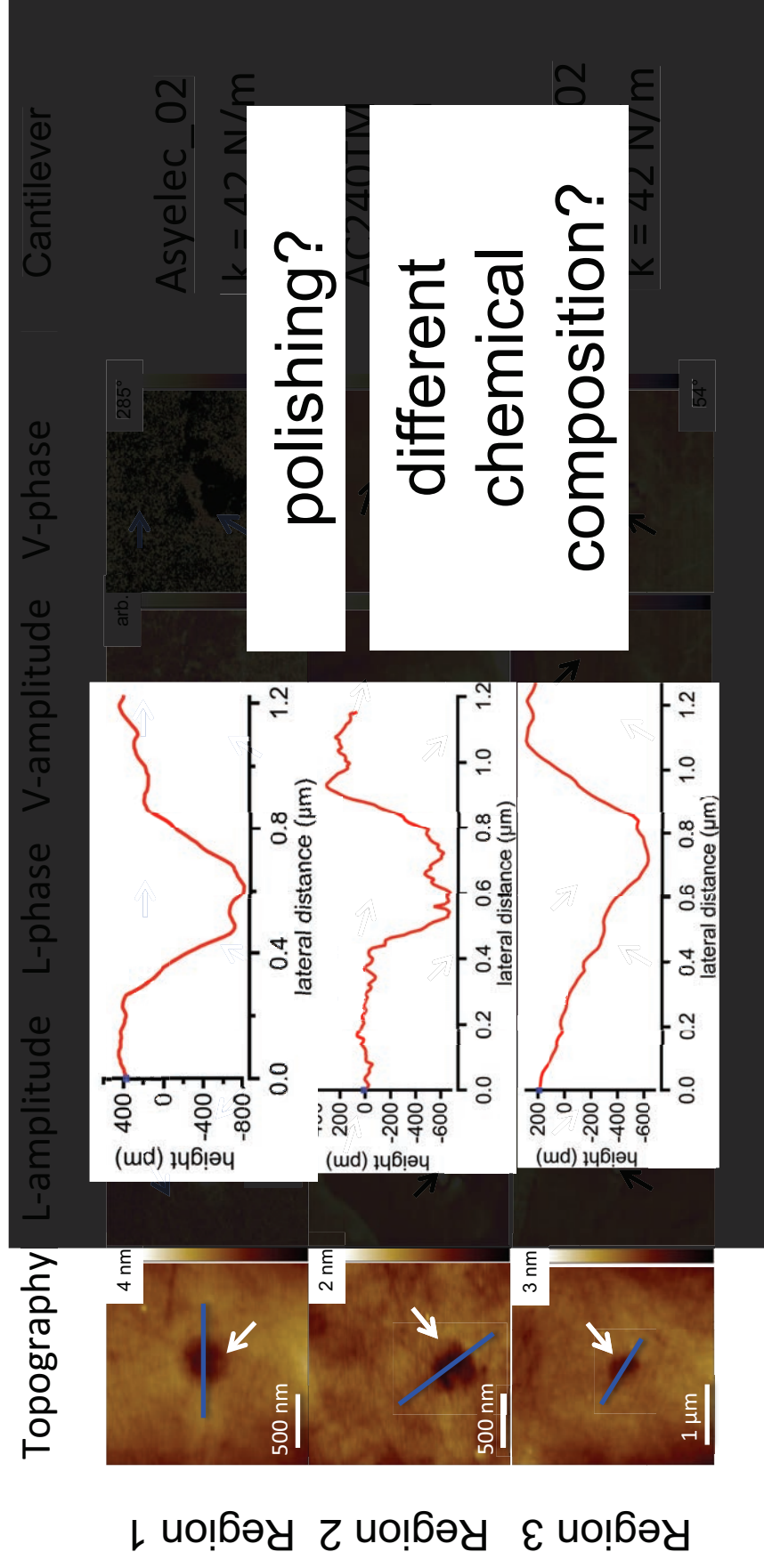


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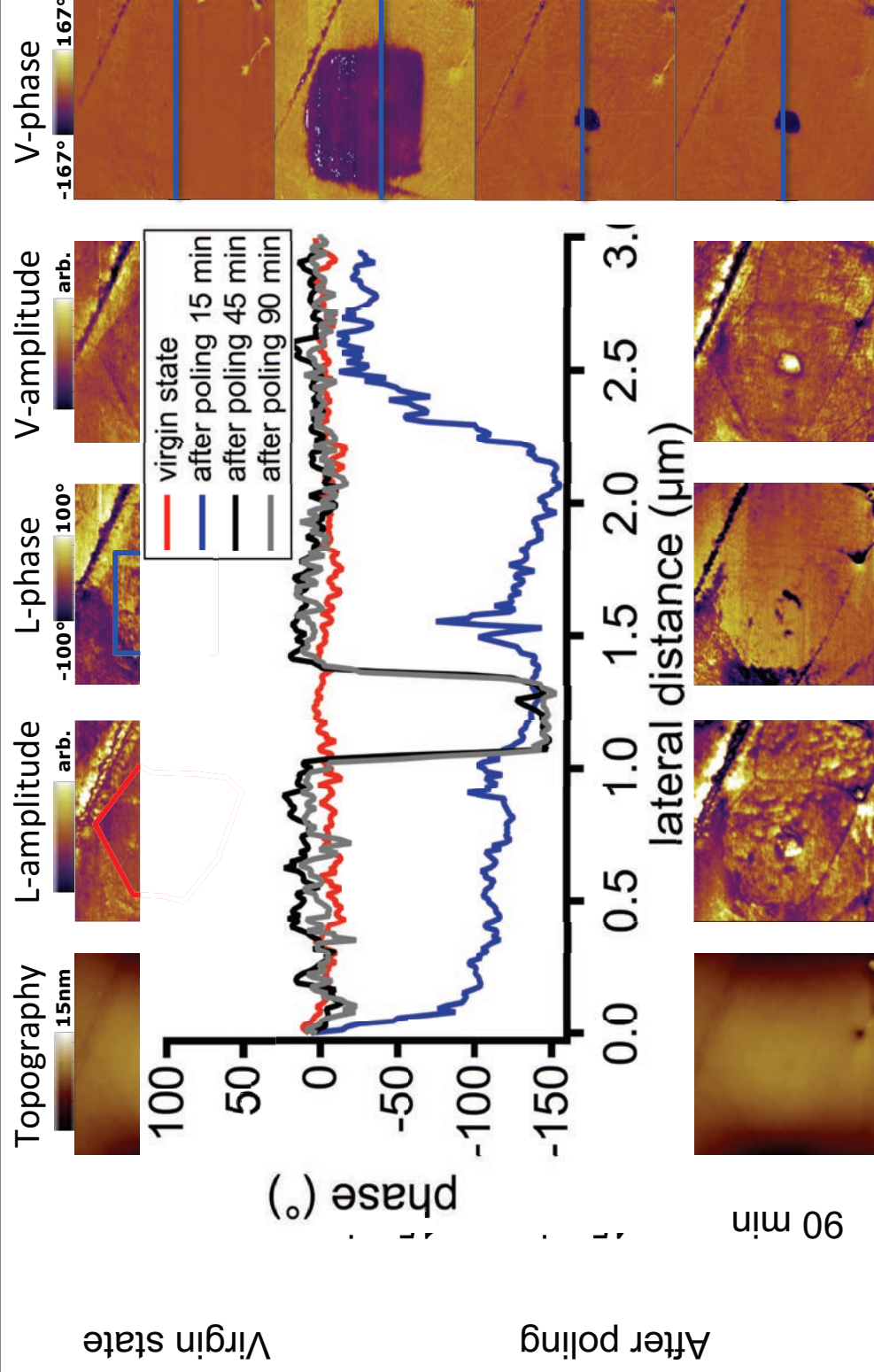


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Result: visualization of core-shell domain structure



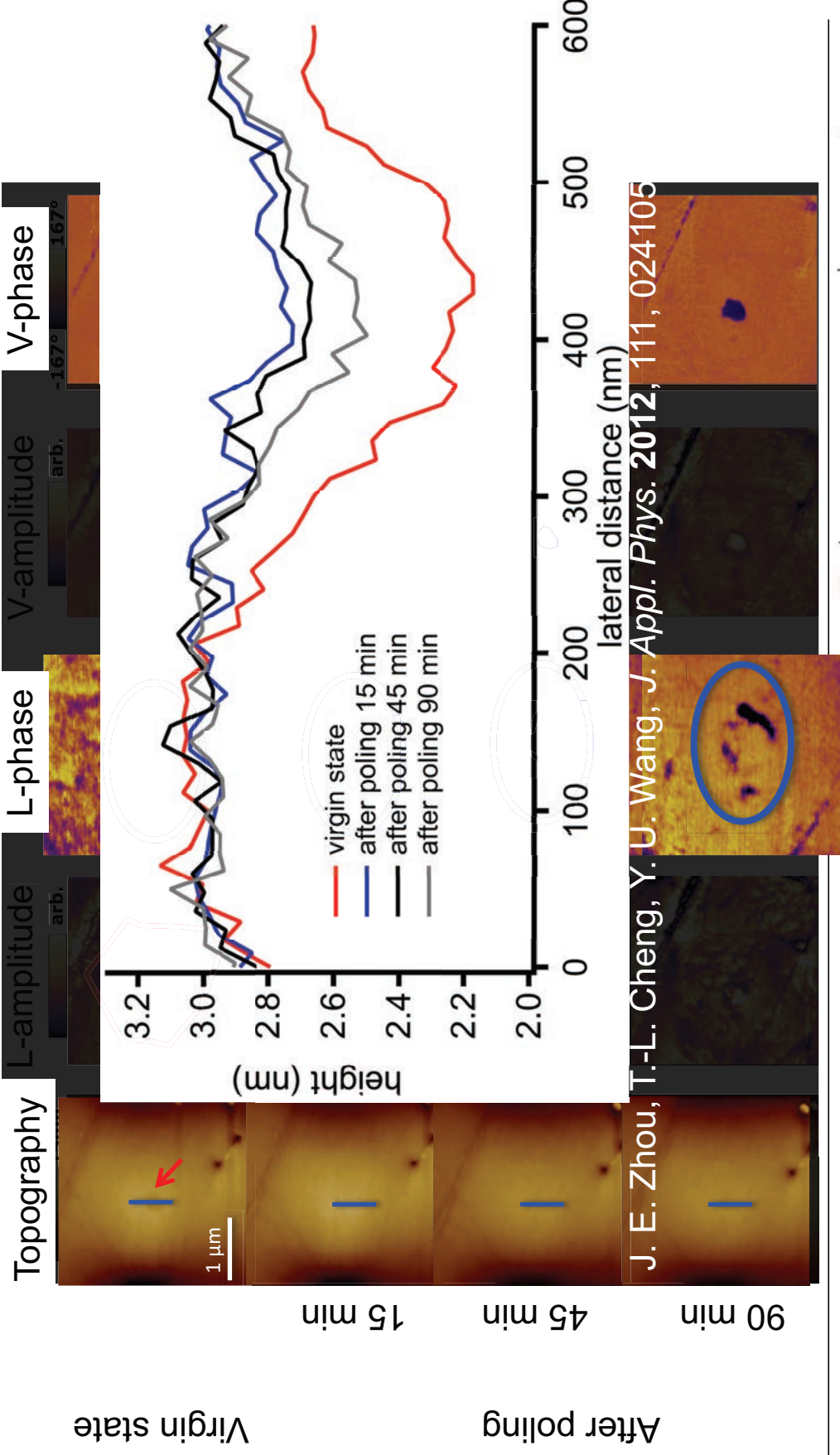
Result: electric-field-induced ferroelectric domain evolution



Result: electric-field-induced ferroelectric domain evolution



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J. E. Zhou, T.-L. Cheng, Y. U. Wang, *J. Appl. Phys.* **2012**, 111, 024105



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Conclusions



- non-ergodic core / ergodic shell domain structure
- heterogeneous nucleation of domains at the core/shell interface
- electric-field-sensitive topographical hollow in the core

Open questions:

- What is the mechanism behind the topographical change in the core region before/after applying electrical field?
- At core/shell interface, strain mismatch? Charge accumulation?

Thank you for the Attention!



Back row (f.l.t.r.): Limor Zemel, Marek Janko, Christian Dietz, Agnieszka Voß, Simon Schiwek, Marie-Christin Apfel; **Front row (f.l.t.r.):** Kim Phuong Lieu, Asma Siddique, Robert Stark, Suman Narayan, Na Liu; **(Missing:** Imke Murschel, Elke Kämmerer, Andreas Plog, Maximilian Köhn)