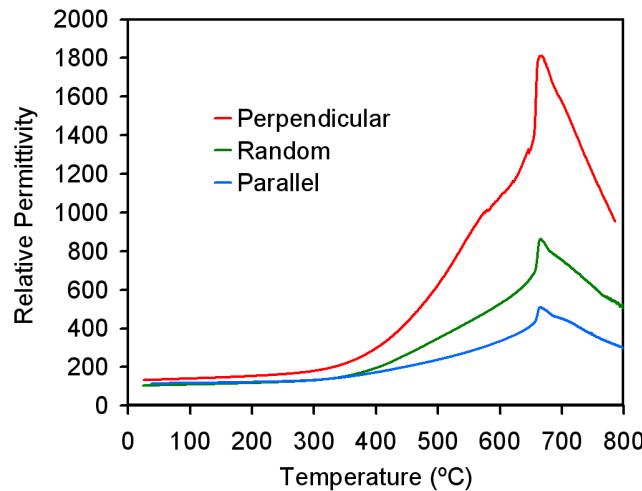
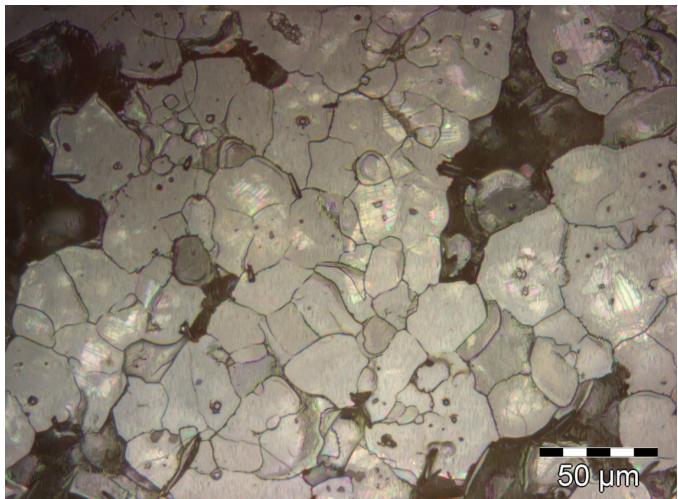


# Thick Film Texturing to Enhance the Properties of Lead-Free Ferroelectric Materials

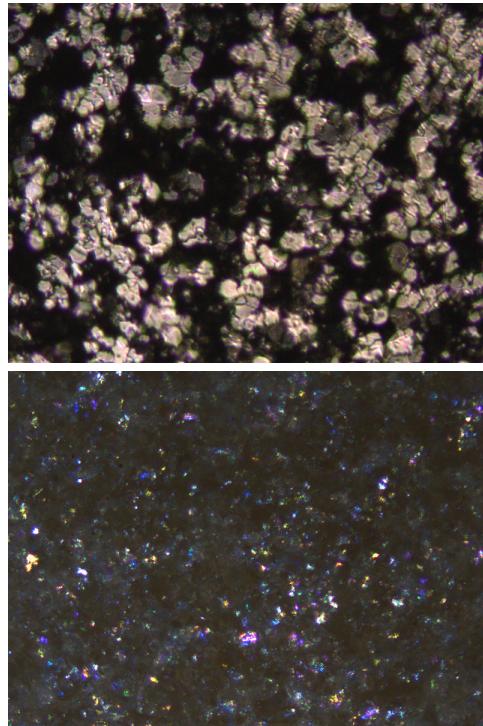


Michael R. Winter

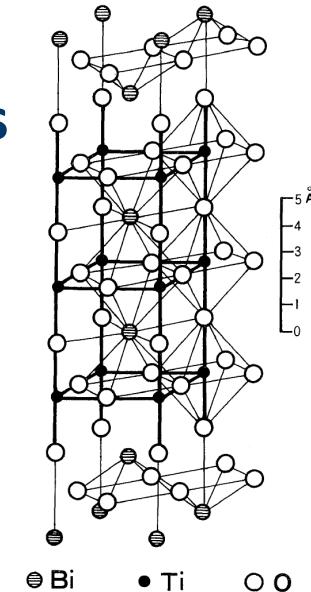
Christopher B. DiAntonio   Tom Chavez   Mark Rodriguez

Sandia National Laboratories

This presentation focuses on improving the dielectric properties of lead-free materials textured by screen printing

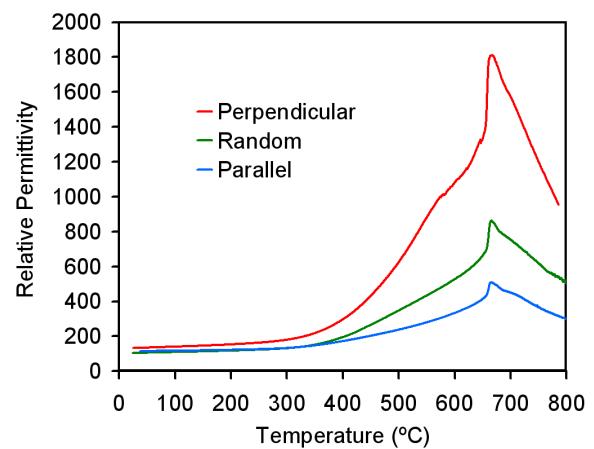


## Lead-free dielectrics

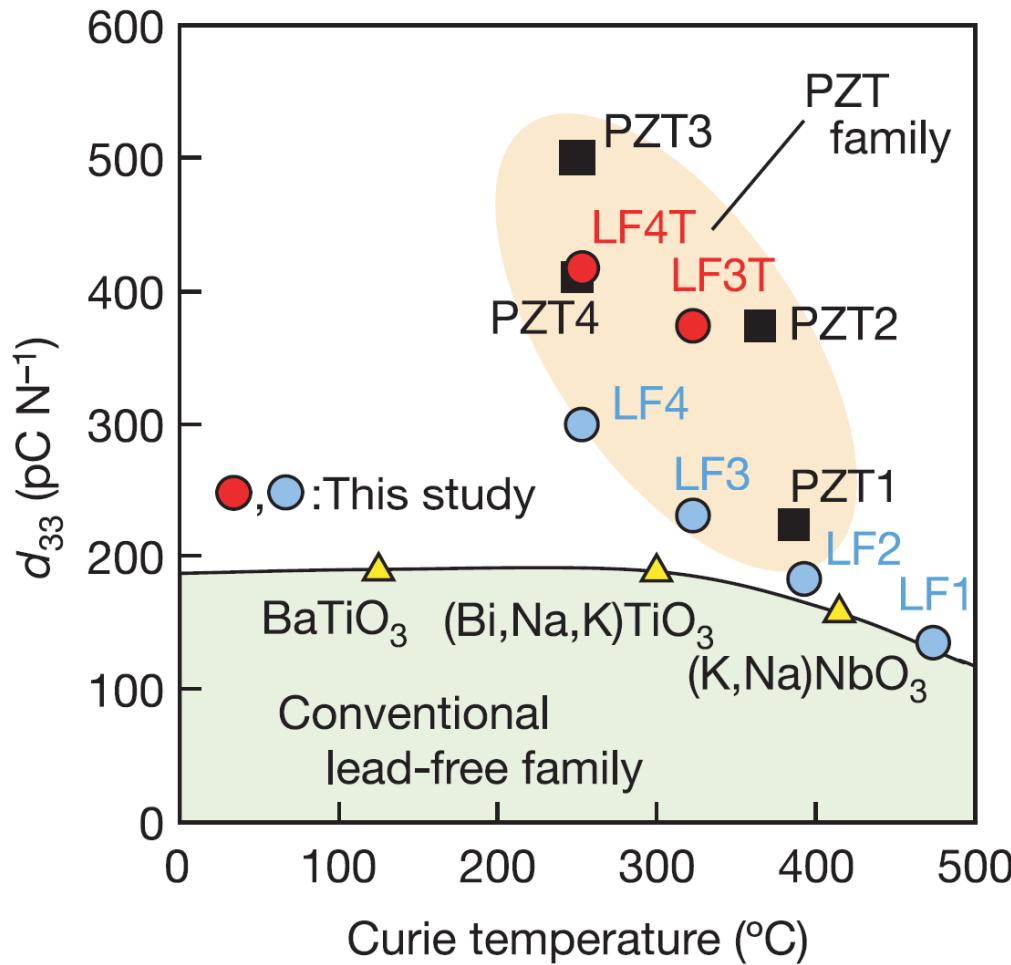


## Scalable industrial techniques

## Improved dielectric properties



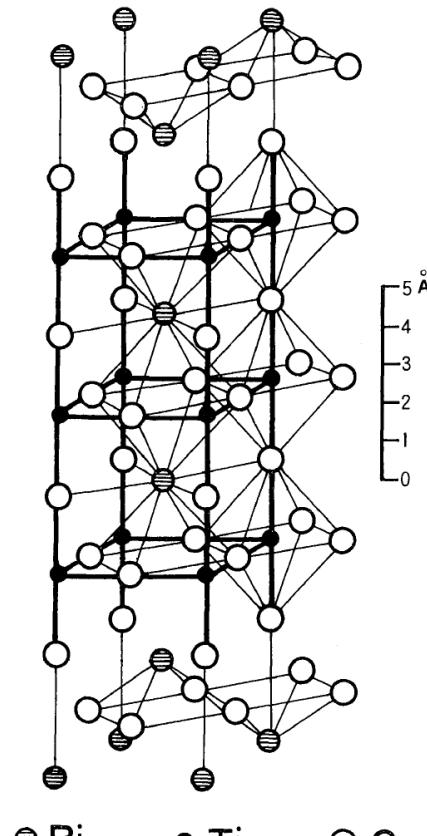
The motivation is to develop replacements for current lead-based systems that are rapidly scalable to industry



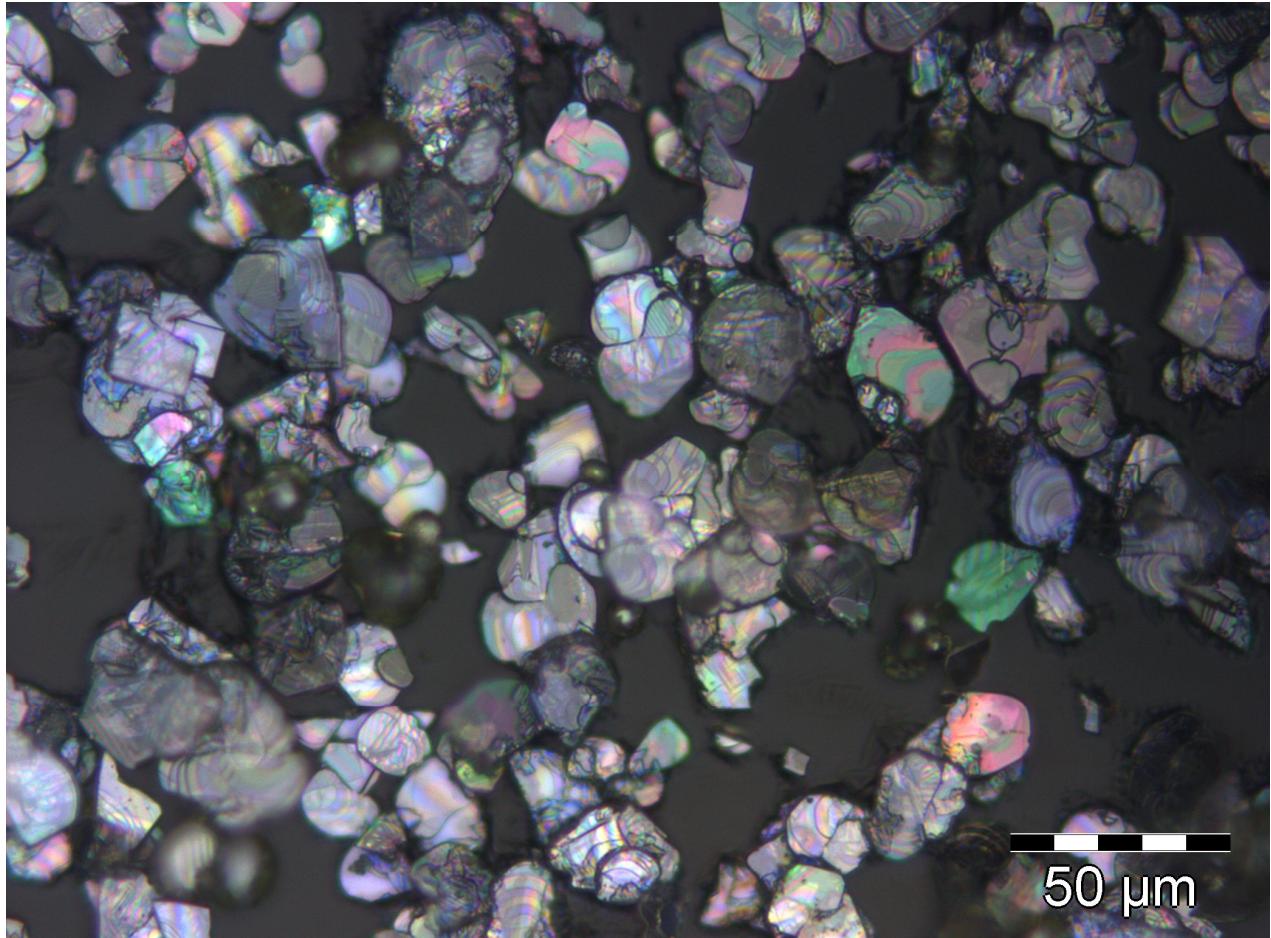
Y. Saito, Nature (2004)

When textured by tape casting, materials in the alkali niobate-based perovskite system have excellent dielectric properties

# Layer-structure materials are excellent candidates for crystallographic texturing

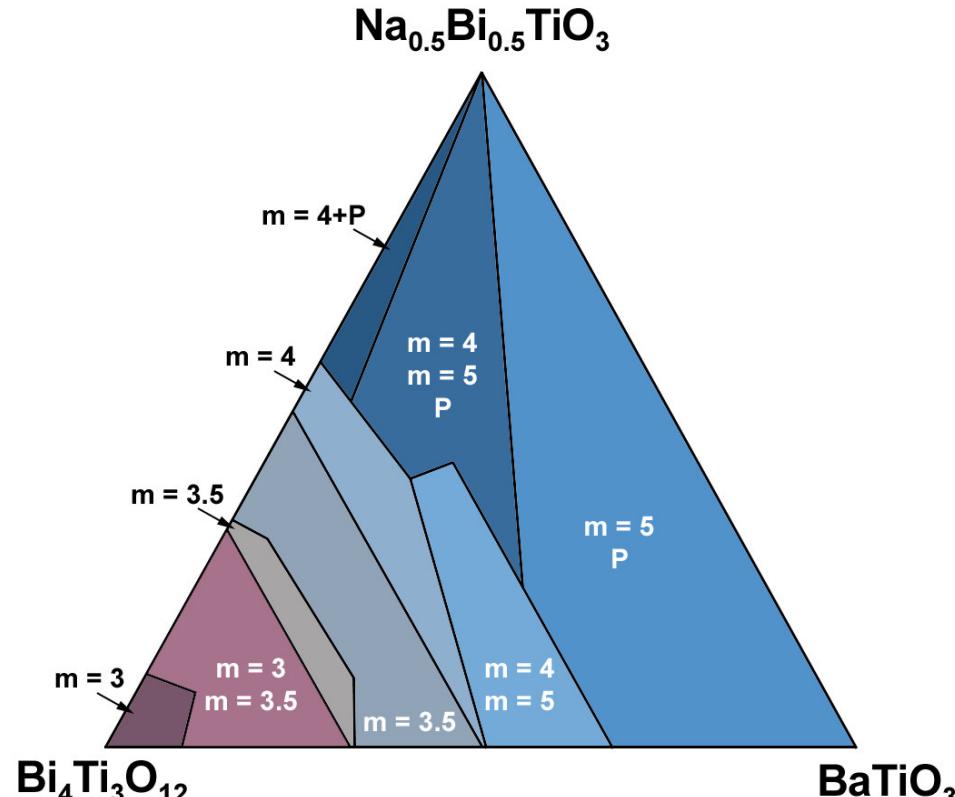


T. Takenaka, JJAP (1980)



High aspect ratio (~200:1) seeds of  $\text{Bi}_4\text{Ti}_3\text{O}_{12}$  can be grown through molten salt synthesis

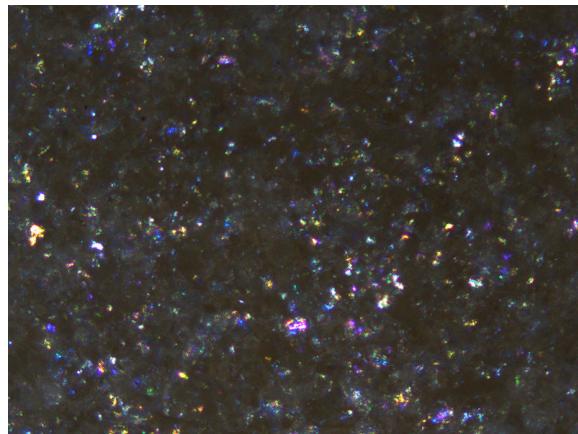
# Several compositions in the ternary are potential lead-free dielectrics



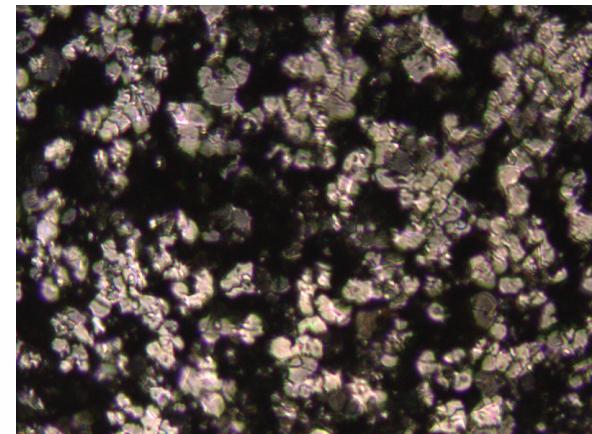
Bi <sub>4</sub> Ti <sub>3</sub> O <sub>12</sub>	Na <sub>0.5</sub> Bi <sub>0.5</sub> TiO <sub>3</sub>	BaTiO <sub>3</sub>
100%	0%	0%
0%	100%	0%
0%	0%	100%
33%	33%	33%
50%	50%	0%
8.55%	43.30%	48.15%

Materials in the Bi<sub>4</sub>Ti<sub>3</sub>O<sub>12</sub> - Na<sub>0.5</sub>Bi<sub>0.5</sub>TiO<sub>3</sub> - BaTiO<sub>3</sub> ternary have a variety of layer structures for different compositions

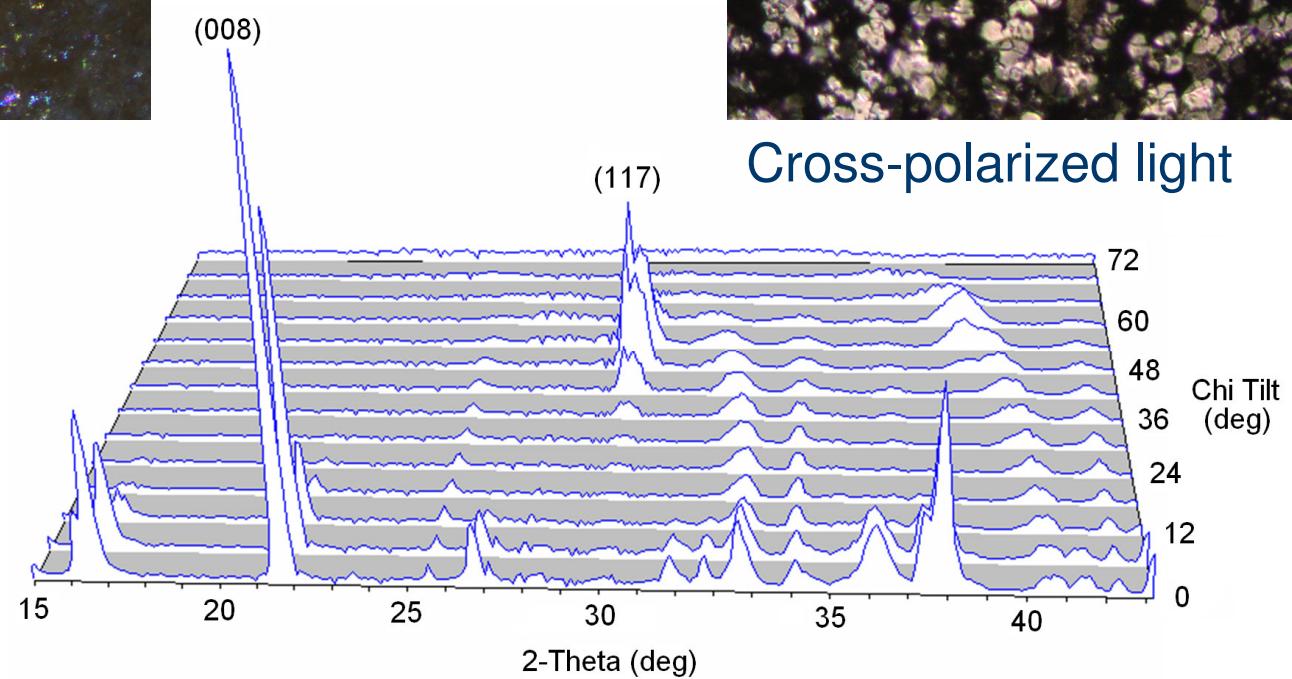
## Texture induced by high shear processing



Polarized light

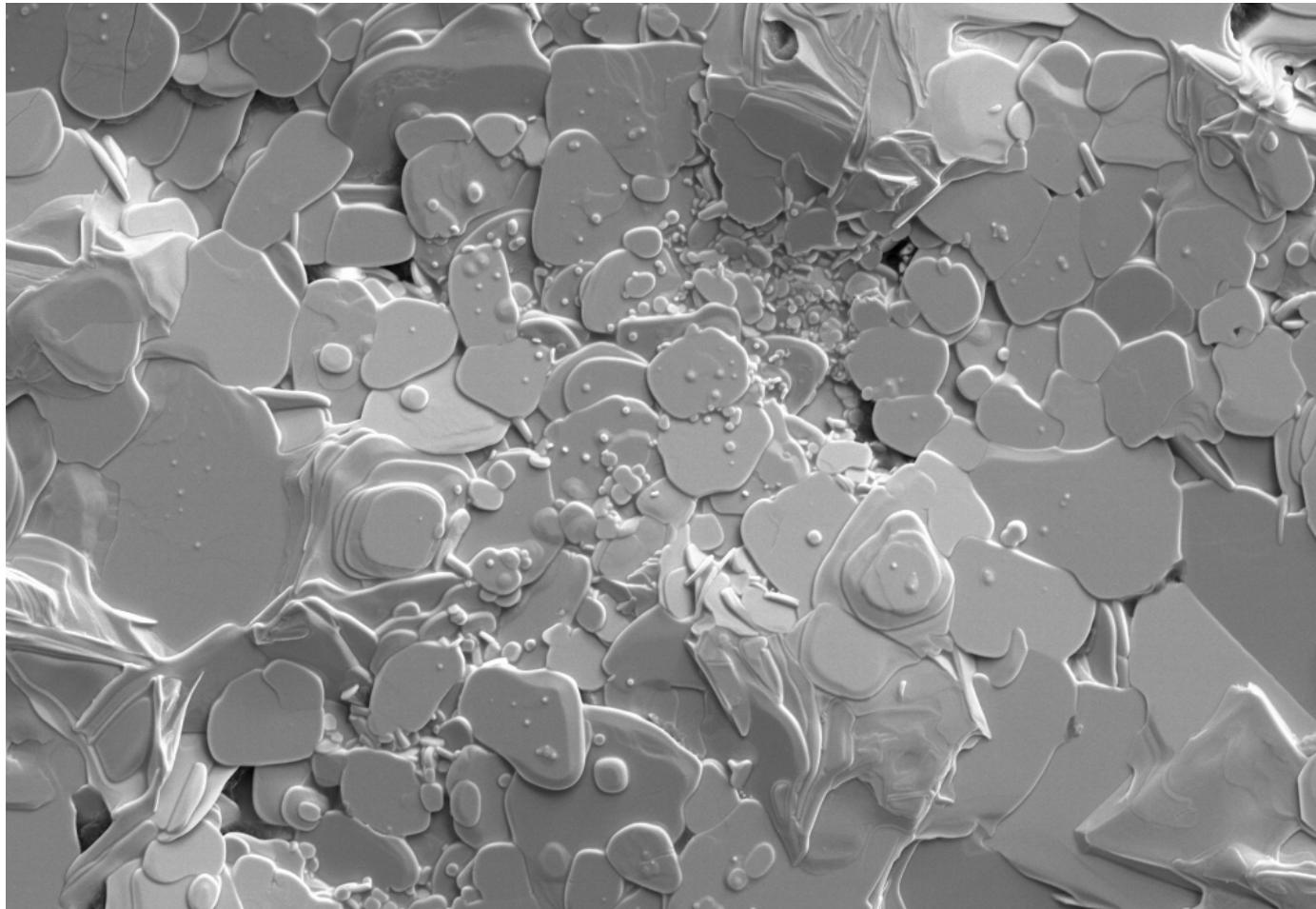


Cross-polarized light



The Lotgering factor is 0.93 for the green screen printed sample

Upon sintering, seeds grow at the expense of the equi-axial powder matrix



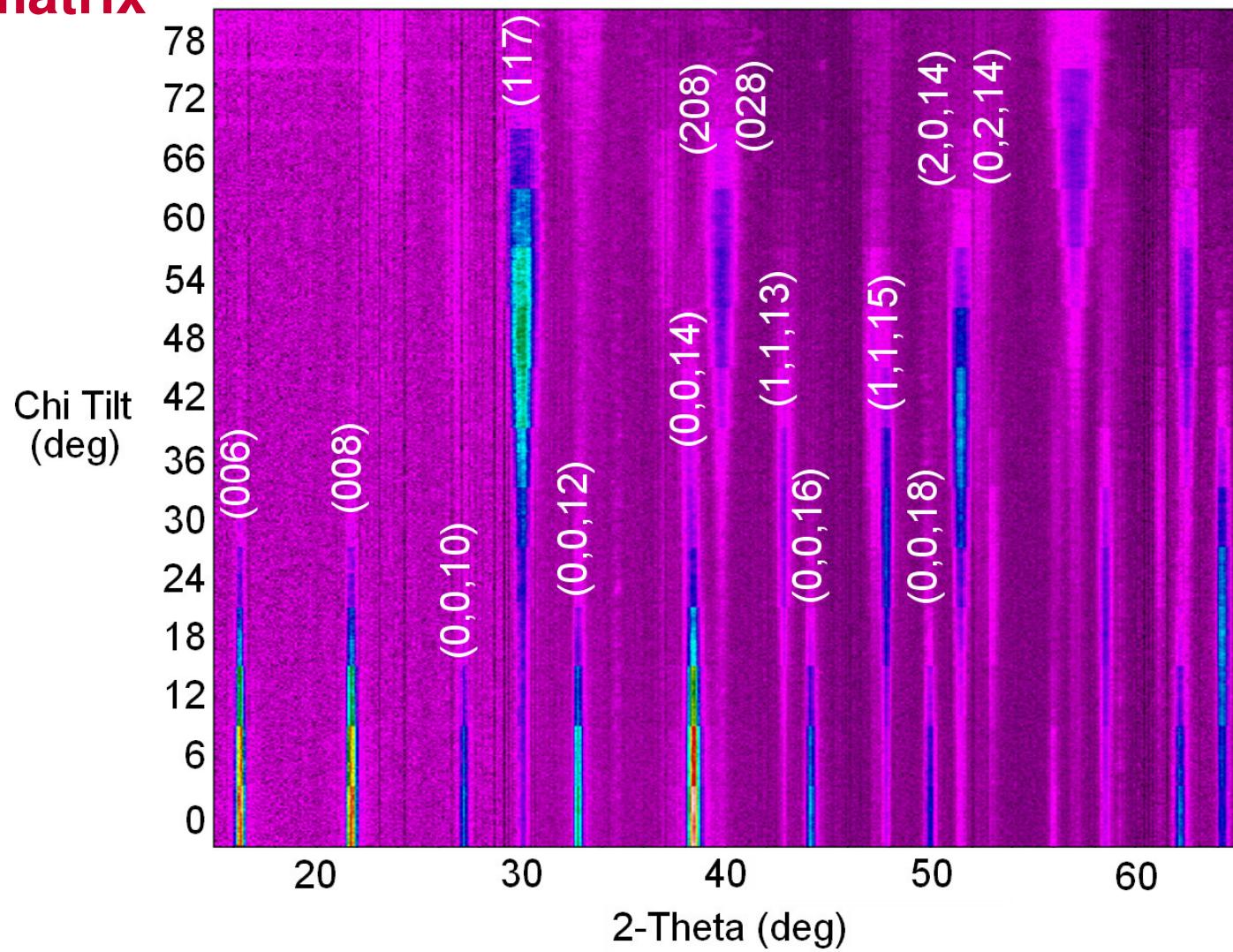
20  $\mu$ m

20 kV

15mm WD

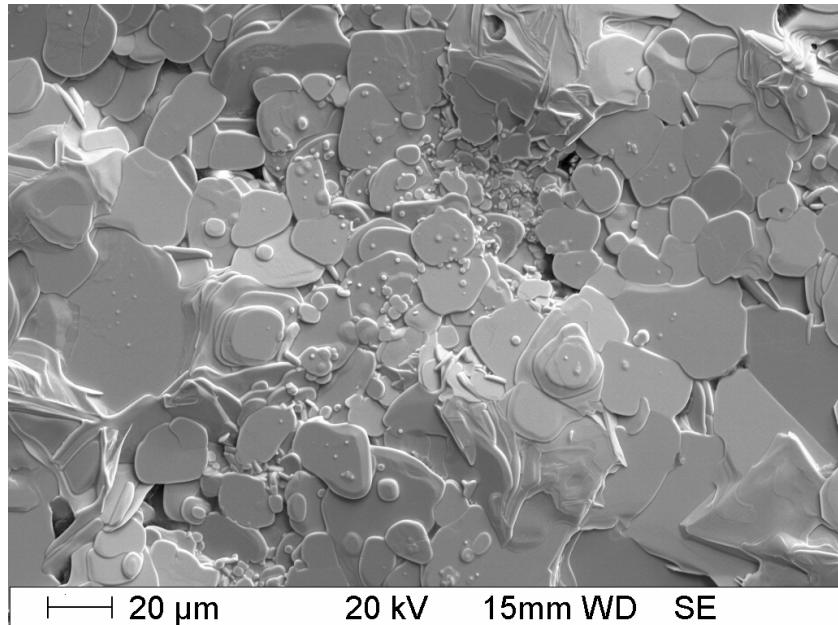
SE

Upon sintering, seeds grow at the expense of the equi-axial powder matrix

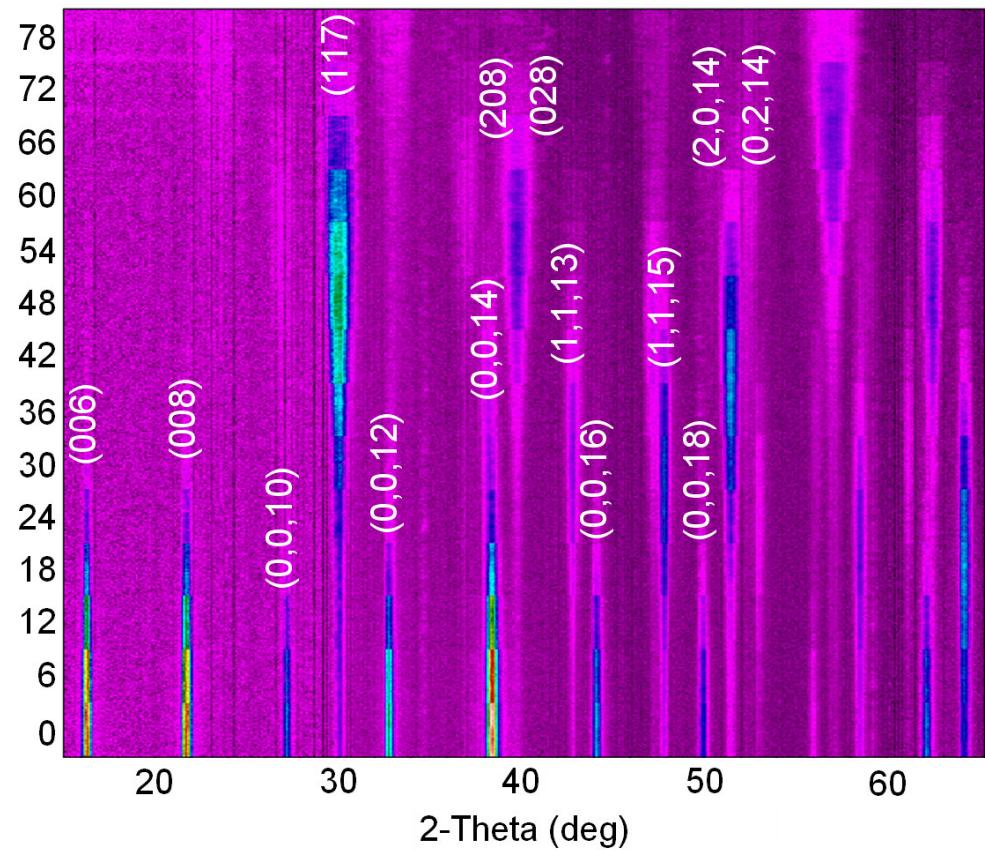


The Lotgering factor is 0.96 for the sintered screen printed sample

Upon sintering, seeds grow at the expense of the equi-axial powder matrix

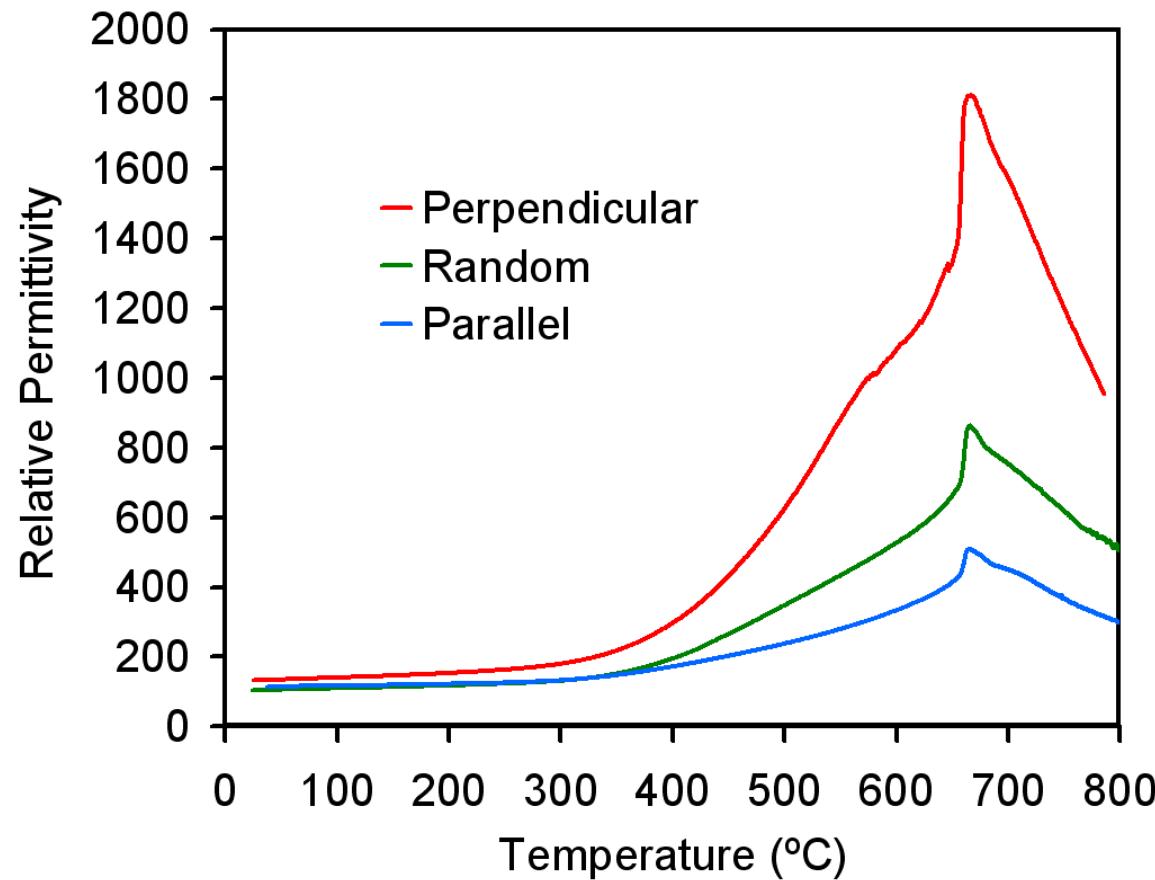


Chi Tilt  
(deg)



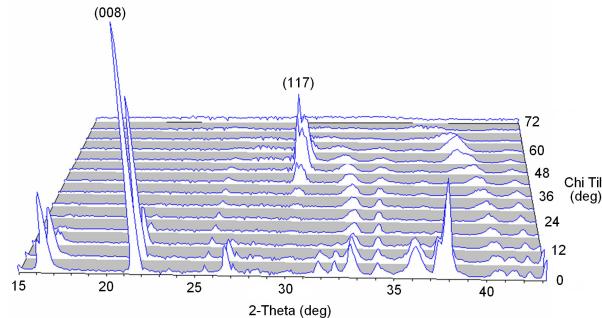
The Lotgering factor is 0.96 for the sintered screen printed sample

**The relative permittivity of the textured material is significantly better than that of the non-textured material**

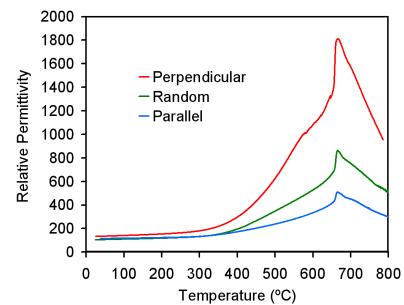


**Tape cast material with a similar Lotgering factor shows very different relative permittivity values dependent on orientation**

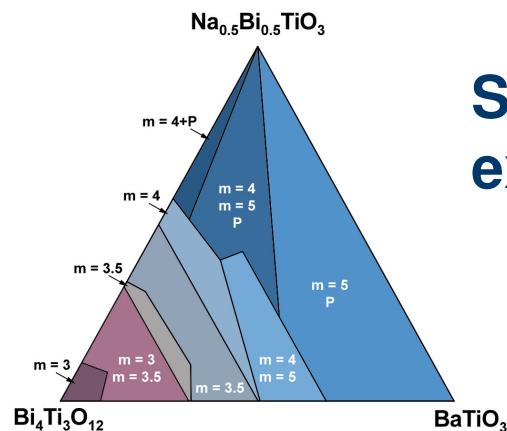
# Textured layer-structure, lead-free materials show considerable promise to replace lead-based dielectrics



Screen printing has been shown to result in highly textured layer-structure materials



Similarly textured materials result in a significant improvement in dielectric properties



Several layer-structure materials have yet to be examined as lead-free dielectrics

Questions?

**Lotgering Factor is calculated from the powder diffraction pattern**

$$F = \frac{P - P_0}{1 - P_0}$$

$$P = \frac{\sum I(h00)}{\sum I(hkl)}, P_0 = \frac{\sum I_0(h00)}{\sum I_0(hkl)}$$

## Processing conditions are continuously modified to achieve optimum density and microstructure

Seed Growth - 1100°C for 16 hours → slow cool at 10°C/h to 940°C

Ink - texanol-based ink with 30-50 v/o powder (10-50 w/o seeds)

Substrate - Pt barrier layer or Bi soaked alumina substrate

Drying - 120°C for 15 minutes

Sintering - 1100°C for 1 hour in a sealed bismuth atmosphere