

# **Cost-Effective Fabrication of High-Temperature Ceramic Capacitors for Power Inverters\***

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**Project ID# EDT-061**

**\*Work supported by the U.S. Department of Energy, Vehicle Technologies Program.**

# Overview

## Timeline

- Project start date: FY14
- Project end date: FY16
- Percent complete: 50%

## Budget

- Total project funding (FY14 - FY16)
  - DOE: \$3314K. Out of this total, subcontract to Delphi/Sigma Technologies = \$810K & Penn State University = \$450K
- Funding received in FY14: \$1149K
- Funding for FY15: \$305K

## Barriers addressed

- **A & C (Cost & Weight): Overall size and cost of inverters**  
*Capacitors are a significant fraction of the inverter volume ( $\approx 35\%$ ), weight ( $\approx 23\%$ ), and cost ( $\approx 23\%$ ).*
- **D (Performance & Lifetime): High-temperature operation**  
*The performance and lifetime of capacitors available today degrade rapidly with increasing temperature (ripple current capability decreases with temperature increase from  $85^{\circ}\text{C}$  to  $105^{\circ}\text{C}$ ).*

## Partners

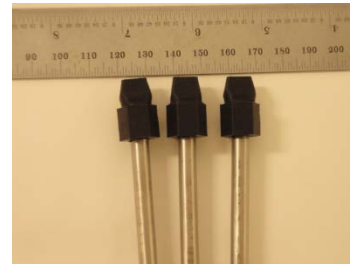
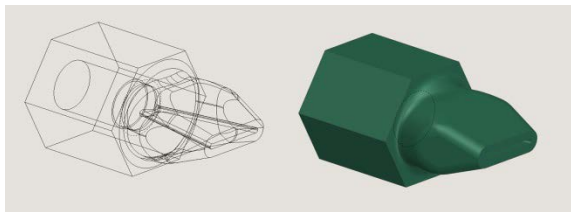
- Delphi Electronics & Safety Systems
- Sigma Technologies International
- Penn State University
- Project Lead: Argonne National Laboratory

# Project Objectives - Relevance

- **Overall objective:** Develop an efficient, cost-effective process for fabricating Pb-La-Zr-Ti-O (PLZT)-based DC-link capacitors for advanced power inverters in EDVs. PLZT films satisfy high-temperature & volumetric requirements for advanced capacitors (140°C/650 V) (EDT Goal).
- **Relevance:** This project addresses key barriers (*capacitor volume, high-temperature operation, as well as fail-safe operation and manufacturability*). Future availability of high-temperature inverters will advance the adoption of highly fuel-efficient EDVs in the marketplace.
- **Specific objective for March '14 – March '15:** Optimize high-rate aerosol deposition (AD) process and deposit PLZT films on metallized polymer substrates & characterize the capacitors; perform economic analysis to make advanced capacitors.
- **Uniqueness/impact:** Our approach will substantially reduce the size, weight, and cost of DC-link capacitors. PLZT films have high dielectric constant ( $k \approx 80$ ), high insulation resistance ( $>10^{13} \Omega\text{-cm}$ ), & high-temperature capability ( $>200^\circ\text{C}$ ) and will meet EDT requirements for capacitors operating at high temperature with high volumetric efficiency.

# Milestones

Month/Year	Milestone or Go/No-Go Decision	Progress Notes
Dec. 2014	Optimize process to make PLZT powder for use in AD process.	Process optimized; commercial supplier identified & powder used in AD process.
Mar. 2015	Define capacitor specifications for the selected inverter design.	A report on capacitor specifications and preliminary cost analysis done.
June 2015	Optimize AD process conditions to fabricate thicker PLZT films on thinly metallized polymer films.	PLZT film made by AD process has high k (~80 vs. ~3 for SOA polymer cap.) and low loss over the operational temperature, voltage, and frequency range.
Aug. 2015	Go/No-Go decision: Decision on film coverage area.	Nozzle design being optimized; 3D printing used to make nozzles.
Sep. 2015	Develop electrode architecture to obtain benign failure.	Developed electrode architecture; studied fault clearing event in PLZT films.
Dec. 2015	Begin integrating AD process into a roll-to-roll coating system.	On schedule.



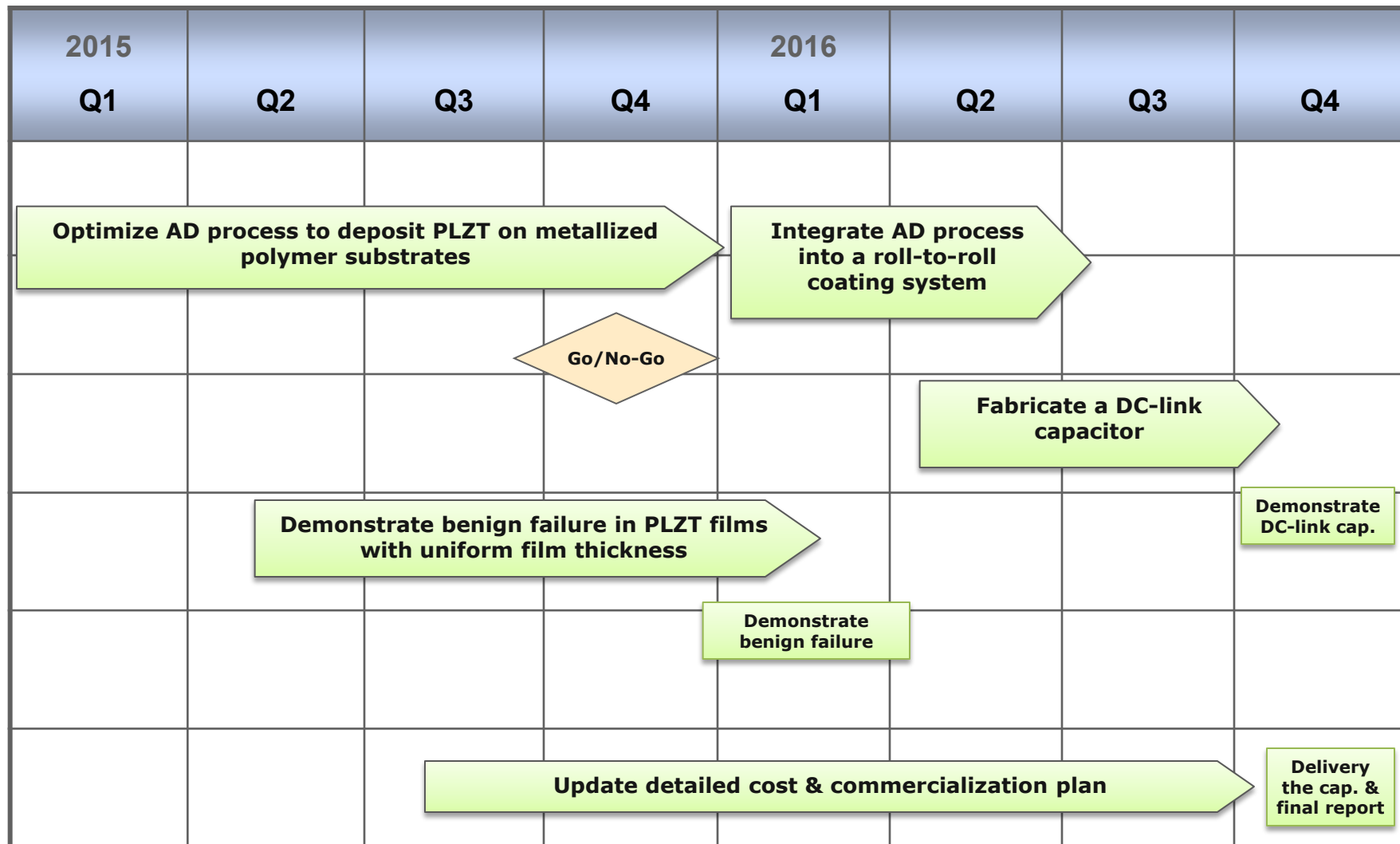
Design/3D printing by  
Chris Hohmann/ Sigma  
Technologies

# Technical Approach/Strategy

- Higher dielectric constant ( $\approx 80$  vs.  $\approx 3$  for SOA polymer-based caps.) Pb-La-Zr-Ti-O (PLZT) film capacitor to achieve higher volumetric efficiency.
- High temperature PLZT film to meet/exceed  $140^{\circ}\text{C}$  operation temperature requirement.
- High-rate aerosol deposition (AD) process to economically manufacture the PLZT-based advanced capacitors.
- Smaller capacitor ( $\approx 0.3$  L), higher operation temperature ( $>200^{\circ}\text{C}$ ), benign failure mode and lower cost to impact industry.
- Team includes a Tier 1 automotive power electronics manufacturer (Delphi), a leading capacitor designer & capacitor OEM (Sigma), a leading university in the study of dielectrics (PSU), and a National laboratory (ANL) with strong expertise in energy technology R&D to commercialize the technology.

**Already demonstrated feasibility of PLZT material to meet EDT objectives.  
Focus of the current FOA project is to develop high-rate AD process.**

# FY15/16 Approach and Milestones



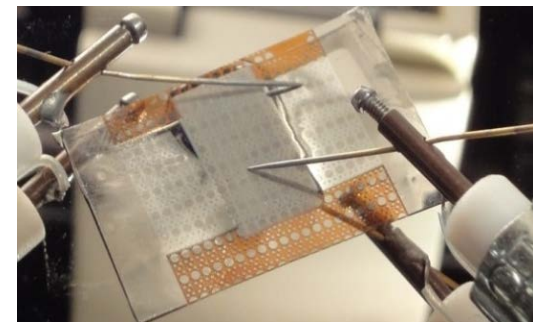
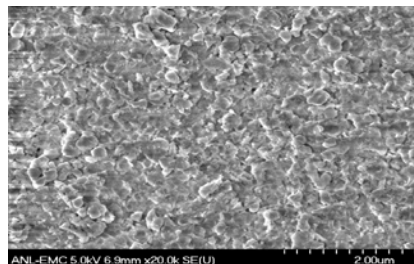
**Go/No-Go Decision:** Decision on film coverage area.

# Technical Accomplishments & Progress

**Objective:** *Develop an efficient, cost-effective process for fabricating PLZT-based DC-link capacitors*

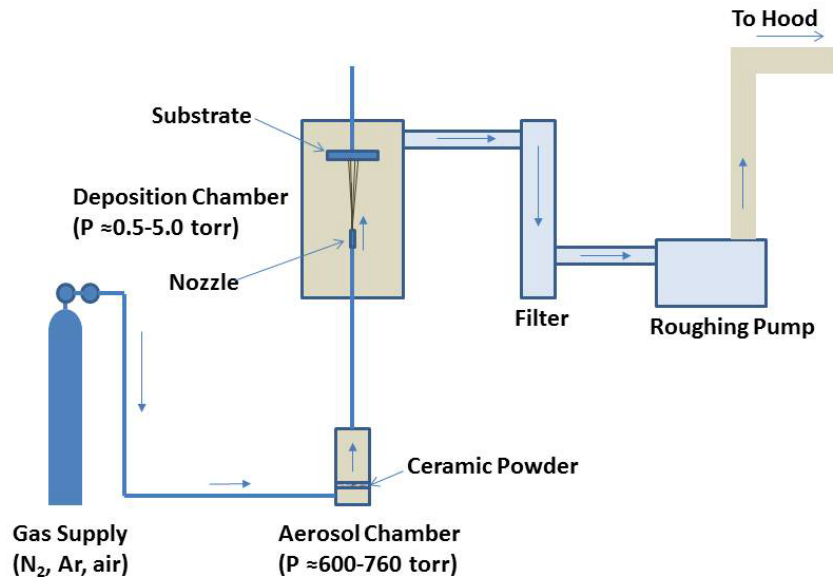
- Synthesized sub-micron PLZT powders for room-temperature, high-rate AD process and identified commercial supplier
- Fabricated  $\approx 8\text{-}\mu\text{m}$ -thick PLZT on aluminum metallized polyimide (PI) films in  $\approx 20$  min (*vs. 1 week by spin-coating*)
- Demonstrated that AD PLZT films on metallized PI films exhibit dielectric properties suitable for high-temperature applications
- Studied fault clearing event in AD films on PI
- Defined capacitor specification for the inverter & established materials cost targets

SEM of aerosol deposited film

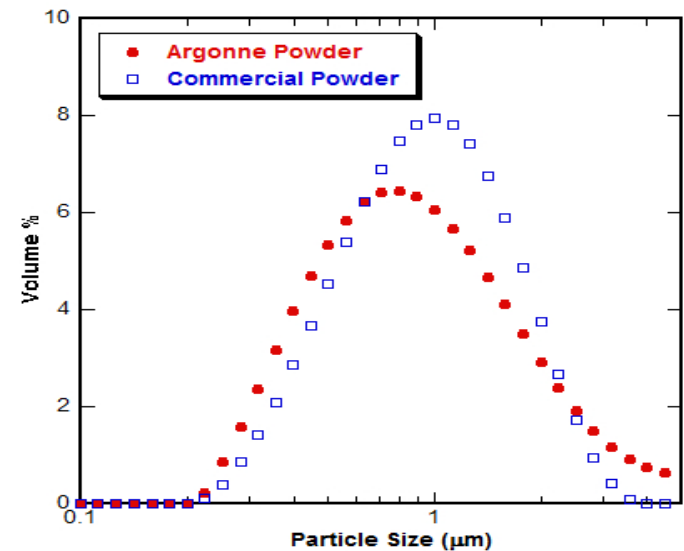


# Technical Accomplishments/Results (Cont.)

## Aerosol (high-rate) Deposition of PLZT Films (*present results*)

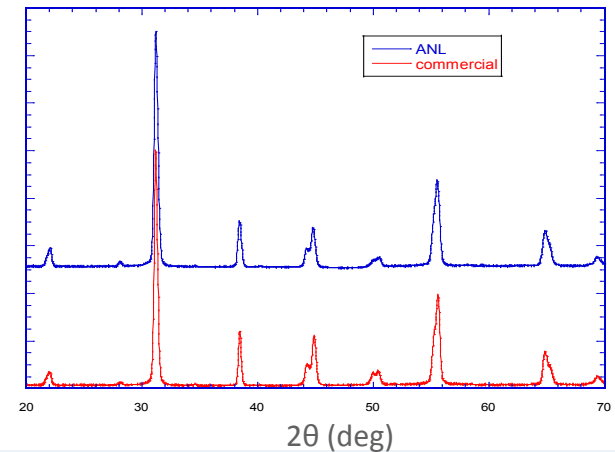


Particle Size Distribution



## Synthesis of sub-micron PLZT powder

- Process to make PLZT powder for high-rate deposition optimized.
- Commercial supplier identified & powder from commercial source used in AD process.

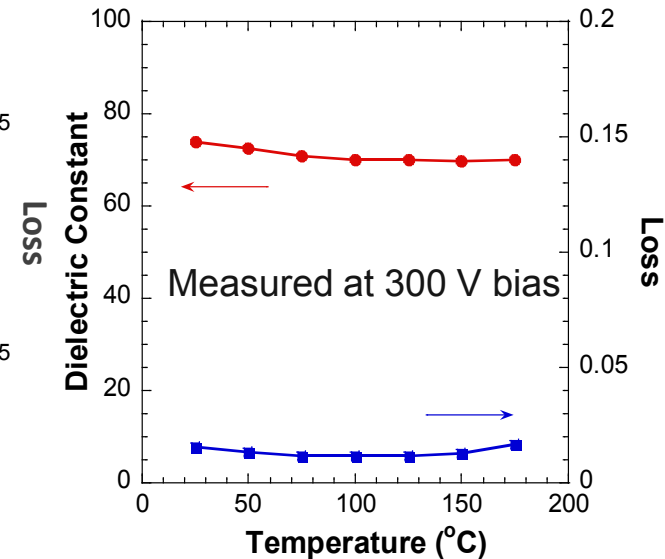
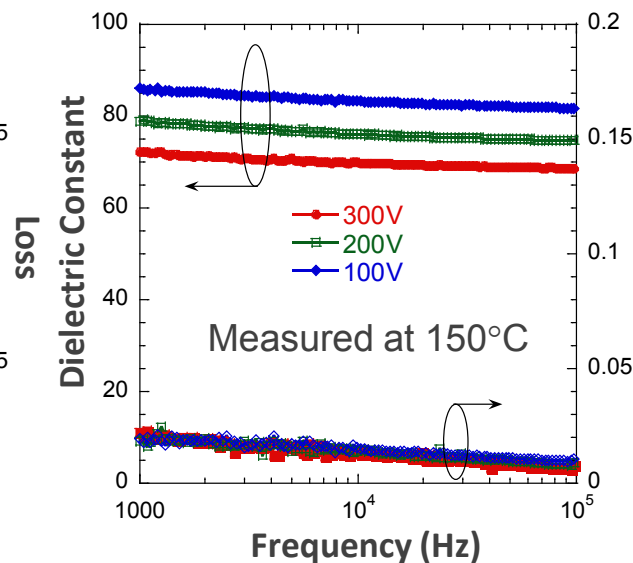
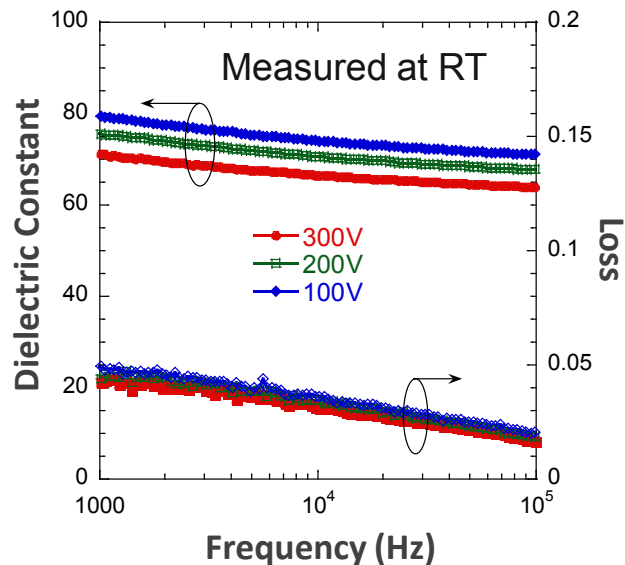
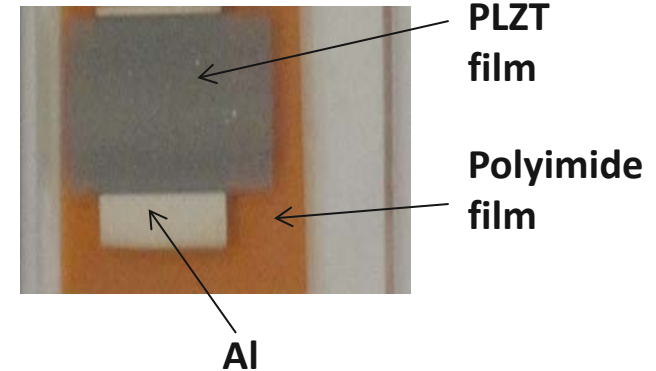
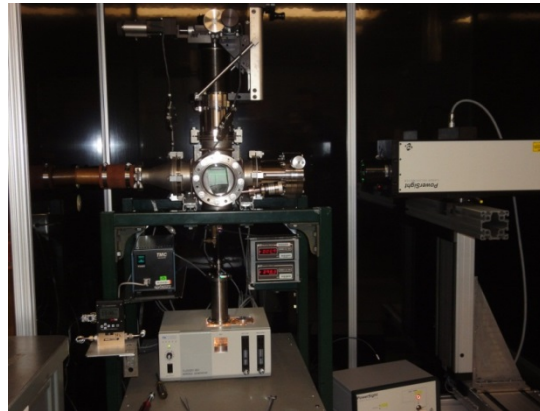
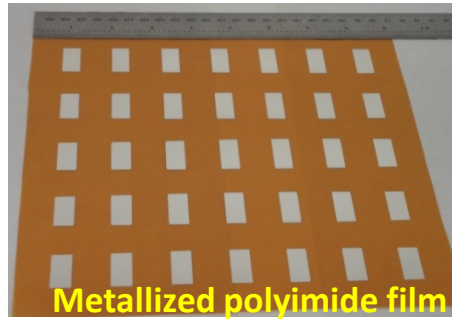


**Developed solution chemistry to make submicron PLZT powders for high-rate deposition process; identified commercial supplier.**



# Technical Accomplishments/Results (Cont.)

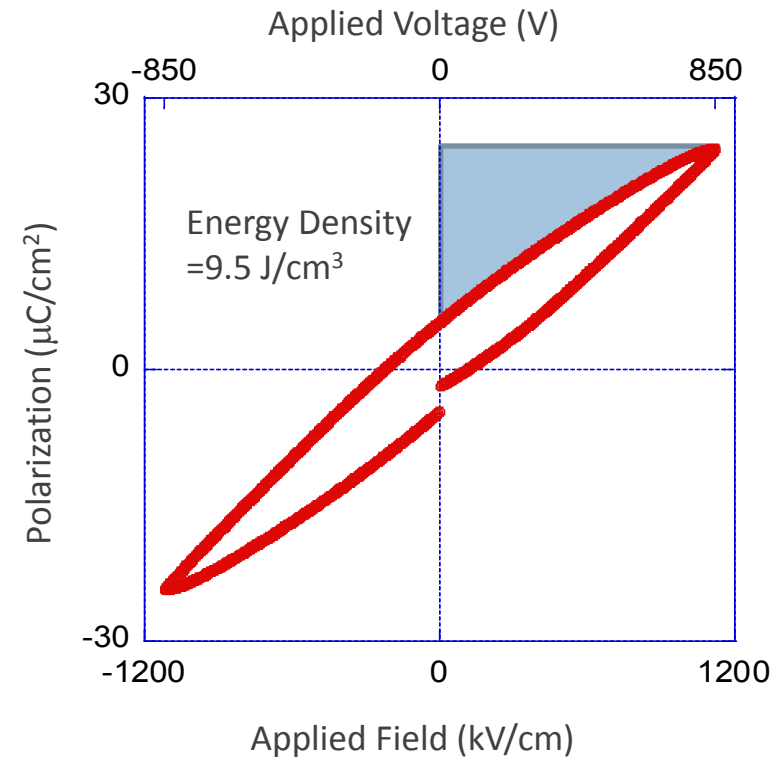
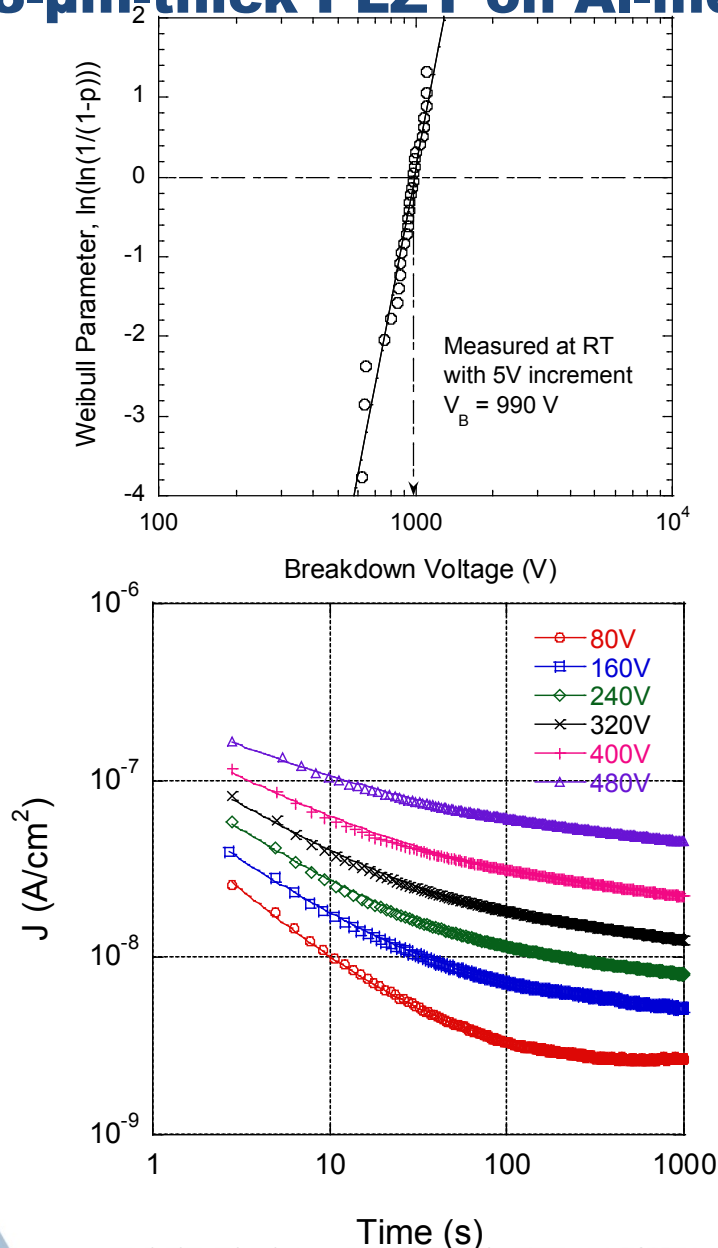
## Properties of $\approx 8\text{-}\mu\text{m}$ -thick PLZT on Al-metallized polyimide films by AD process



PLZT film made by high-rate AD process has high dielectric constant and low loss over the operational temperature, voltage, and frequency range.

# Technical Accomplishments/Results (Cont.)

## ≈8-μm-thick PLZT on Al-metallized polyimide films by AD



PLZT film made by high-rate AD process has breakdown voltage ≈130 V/μm, energy density ≈10 J/cm<sup>3</sup>, and low leakage current (<10<sup>-7</sup> A/cm<sup>2</sup>) at ≈500 V.

# Technical Accomplishments/Results (Cont.)

## Testing/Validation of Results by Delphi

### Dielectric Constant ( $\kappa$ )

4-2  
**98.4**

6-3  
**96.1**

4-3

2-3  
**95.8**

4-4  
**93.3**

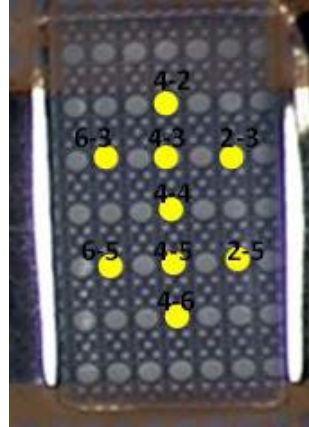
6-5  
**92.7**

4-5

2-5  
**94.8**

**Average 95.37**  
**SD = 1.96**

4-6  
**96.4**



### Voltage Breakdown (V)

4-2  
**828.3**

6-3  
**864.8**

4-3  
**1239.0**

2-3  
**878.5**

4-4  
**1102.3**

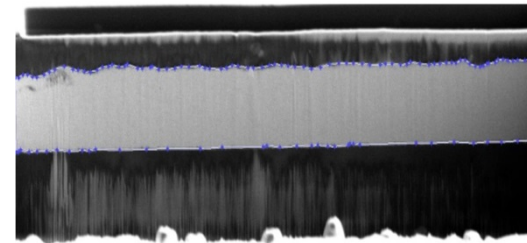
6-5  
**994.0**

4-5  
**1153.3**

2-5  
**1208.3**

4-6  
**1125.0**

**Average 1043.7**  
**SD = 156.3**



SEM HV: 3.0 kV WD: 8.78 mm LYRA3 TESCAN  
View field: 75.0  $\mu$ m Det: SE 20  $\mu$ m  
Stage Tilt: 55.0° Delphi E&S

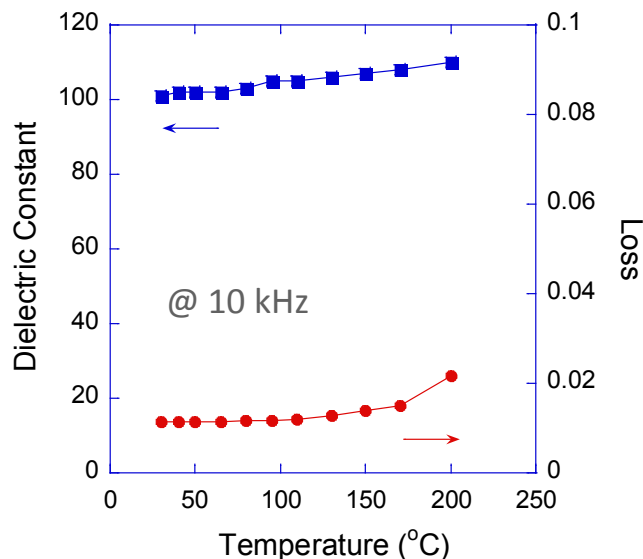
**Delphi measurement validates  
Argonne's result**

# Response to Previous Year's Reviewers' Comments

**Comment:** “that the thinnest Kapton® film on the market was only 7.5  $\mu\text{m}$  which costs \$2,000/kg. The commenter suggested that the team may consider using low-cost substrates, such as aluminum foil, thin PEN film, etc.”

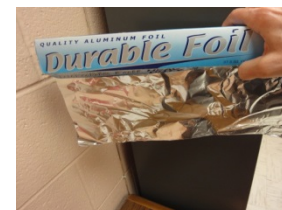
**Response:** yes, the reviewer is right. We are aware of the high cost of very thin Kapton film and the team is looking for cheaper alternates. The AD process is amenable for deposition on variety of substrates including both polymers and metals. In fact we have already demonstrated the capability of AD depositing PLZT on very thin (8- $\mu\text{m}$ -thick) Al foil. We are now depositing PLZT on thin (12- $\mu\text{m}$ -thick) PEN films.

***~8- $\mu\text{m}$ -thick PLZT on 12- $\mu\text{m}$ -thick PEN by AD process***



**Comment:** “The reviewer was concerned that two of the collaborators were on one of the competing capacitor development projects, and asked if the project team could support both with the appropriate resources when needed.”

**Response:** “Judging by the collaboration and interactions we are having with them, we feel that they are dedicated to this project and will provide enough resources to ensure that this project succeeds.”



***PLZT on ~8- $\mu\text{m}$ -thick Al foil by AD***



# Collaboration and Coordination with Other Institutions



DELPHI

Inverter design engineering (direct customer for the technology), defining overall capacitor requirements, supplying critical knowledge of automotive power electronics, testing/validating results, demonstrating DC-link capacitor with an automobile power inverter.



Sigma Technologies

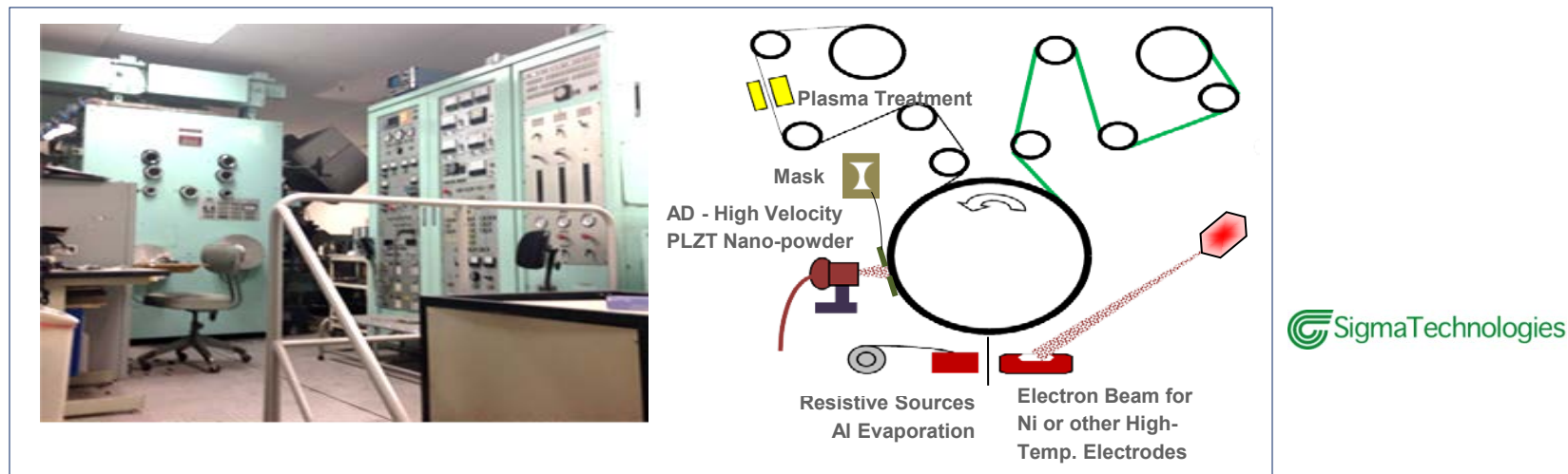
Roll-to-roll deposition systems, multilayer coating technologies, coating capacitor films to improve breakdown and self-healing properties, background to convert lab-scale process into an industrial-scale process.



Dielectric characterization, reliability testing, electrode design & deposition, testing/validating results.

# Remaining Challenges and Barriers

- **Can PLZT-based films be produced by a roll-to-roll process?**
  - Sigma has in place several roll-to-roll and batch metallizing and coating systems that will be retrofitted to perform AD process on continuously moving substrates.



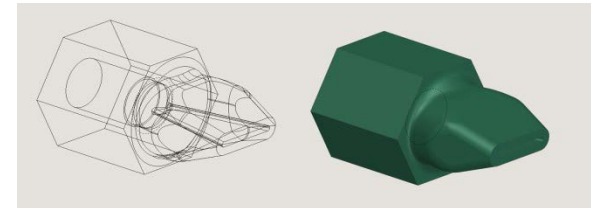
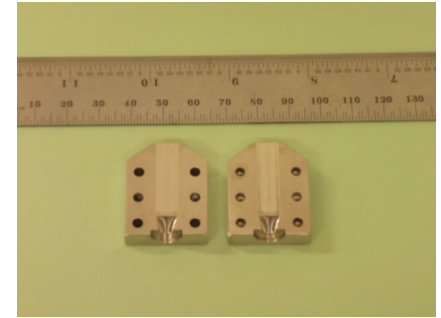
- **Can the thin ceramic dielectrics be wound similar to polymer film capacitors?**
  - The risk involved in winding the rolls of metallized PLZT films is the formation of micro-cracks. The stress-strain properties dictate the minimum bend radius.

***We will address these challenges in our future work***



# Proposed Future Work

- **Optimize AD process parameters to deposit PLZT films on metallized thin polymer films and characterize the capacitors**
  - Optimize nozzle design to deposit larger area films
  - Demonstrate thickness uniformity over larger area
  - Deposit films on thin, low-cost carrier substrates
- **Demonstrate benign failure feature in PLZT films with uniform film thickness**
  - Evaluate different designs with heavy edge electrodes to maximize self-healing process
- **Transfer the AD process to Sigma's roll-to-roll deposition system**
  - Handling of thinner substrate will be easier with Sigma's system
- **Demonstrate the DC-link capacitor with an inverter**
- **Update detailed cost & commercialization plan**
- **Deliver the DC-link capacitor to DOE & submit final report**



# Summary

**We are developing an efficient, cost-effective process for fabricating high-temperature DC-link capacitors for advanced power inverters.**

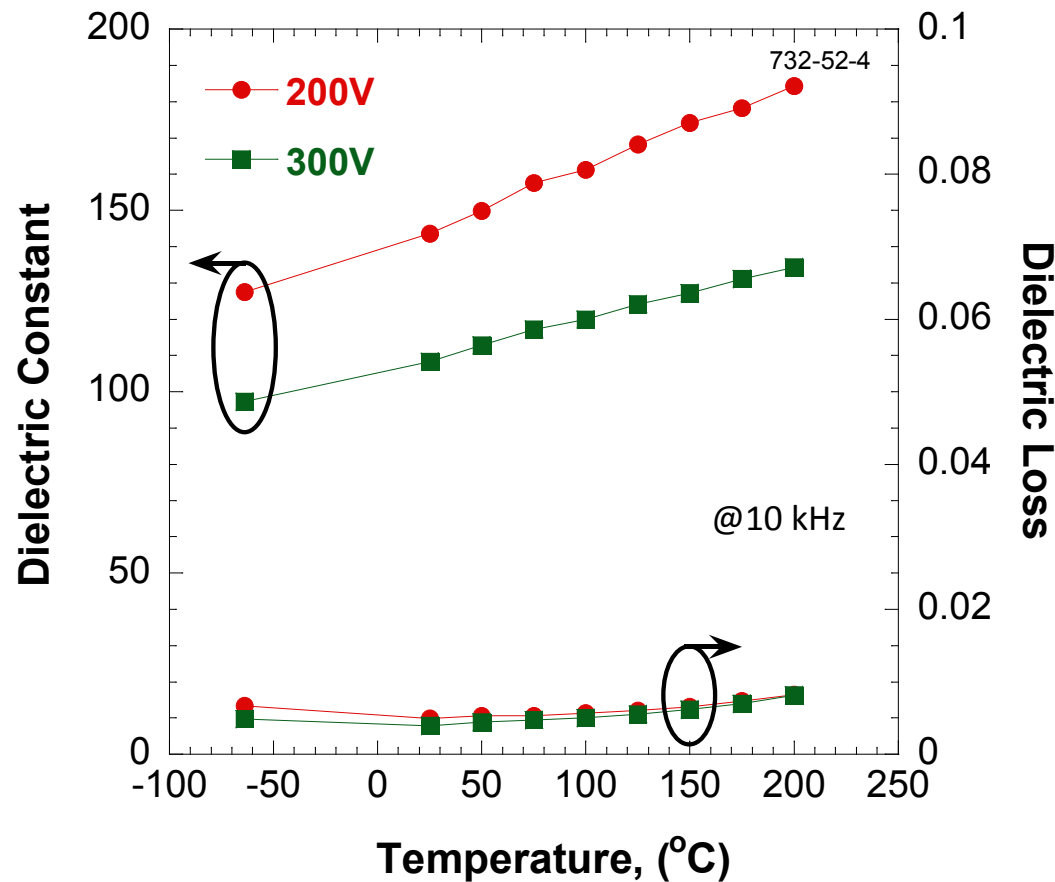
- The project addresses key barriers (*size, high-temperature capability, as well as manufacturability*) to achieve advanced DC-link capacitors.
- Aerosol deposition (AD), a high-rate, room-temperature film deposition process, is being developed to reduce capacitor cost.
- Demonstrated that AD PLZT films on metallized PI films exhibit dielectric properties suitable for high-temperature applications.
- The team includes a Tier 1 automotive power electronics manufacturer (Delphi), a leading capacitor designer & capacitor OEM (Sigma), a leading university in the study of dielectrics (Penn State), along with ANL, a National laboratory with strong expertise in energy technology R&D to commercialize the technology.
- Capacitors made in this work can be rolled into Delphi's inverter products.
- Patents: Five issued; Publications/Presentations: >50 made.



# Technical Back-up Slides



# Accomplishments: Temperature Dependent Dielectric Properties of PLZT Films at 200V & 300V (made by spin-coating)



Measured  $k \approx 110$  & loss  $\approx 0.004$  (i.e., 0.4%) @ 10 kHz & 300 V bias on a 3- $\mu\text{m}$ -thick PLZT on Ni-foil

$\text{ESR} = \text{DF}/2\pi f c$  (DF = loss factor;  $f$  = frequency;  $c$  = capacitance).

Calculated ESR for 1000  $\mu\text{F}$  cap. based on measured material properties

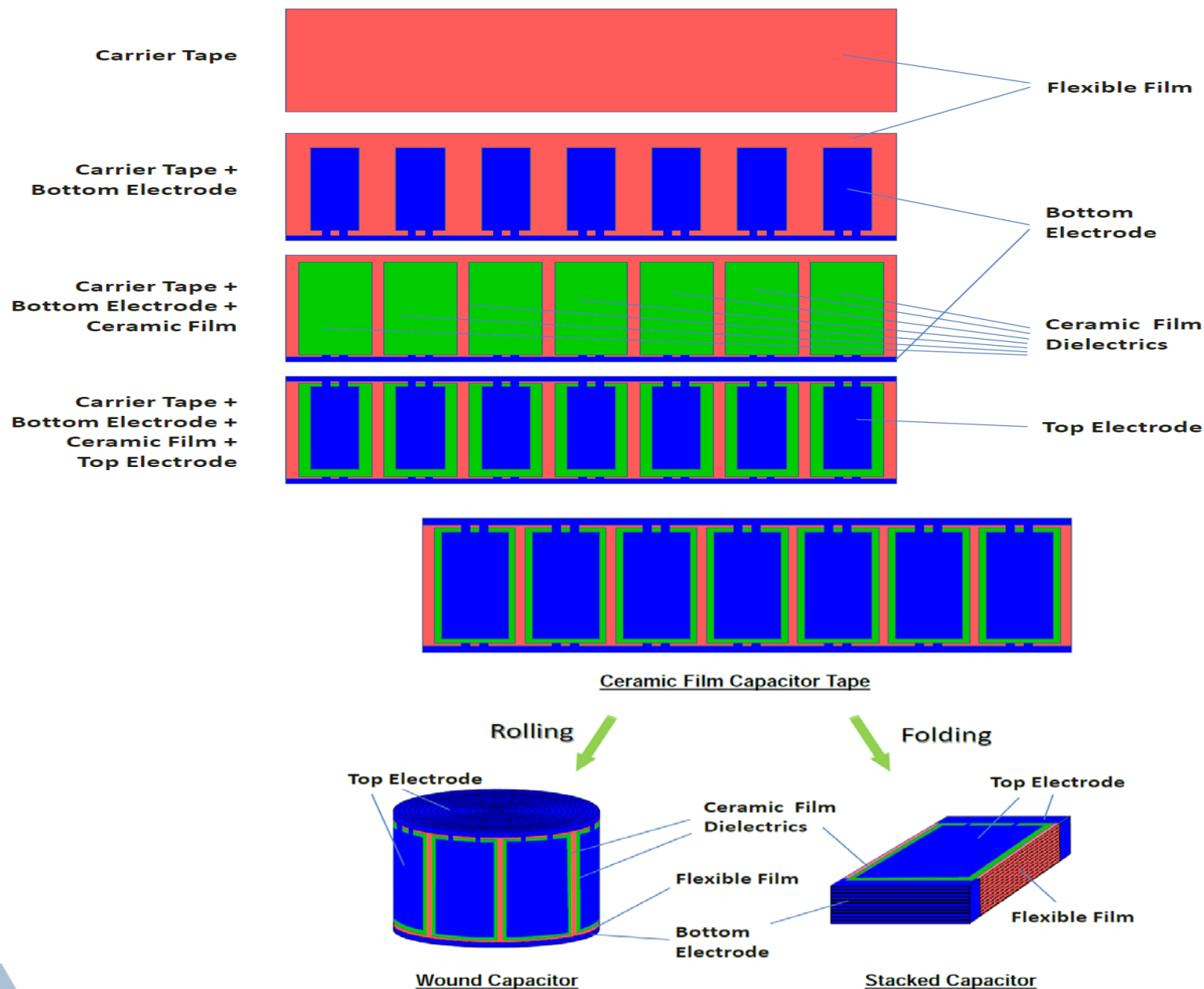
Bias Voltage (V)	ESR @ -64°C (m $\Omega$ )	ESR @ RT (m $\Omega$ )	ESR @ 150°C (m $\Omega$ )
200	0.11	0.08	0.10
300	0.08	0.06	0.10

ANL's PLZT film has high-temperature capability; high dielectric constant (high volumetric efficiency); and very low ESR

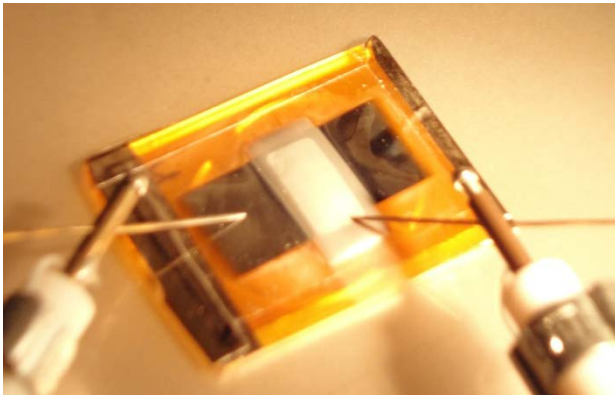
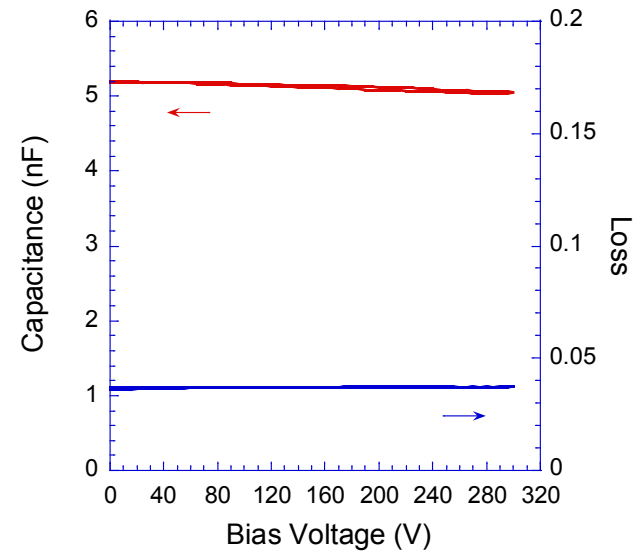
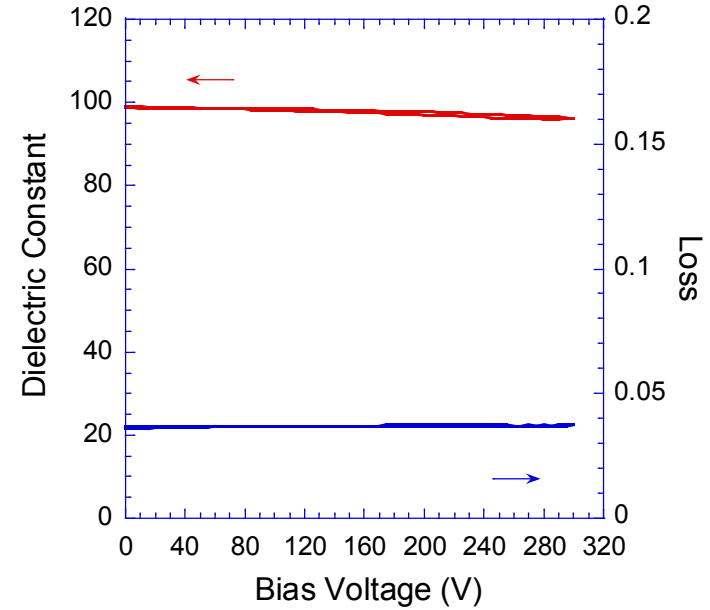
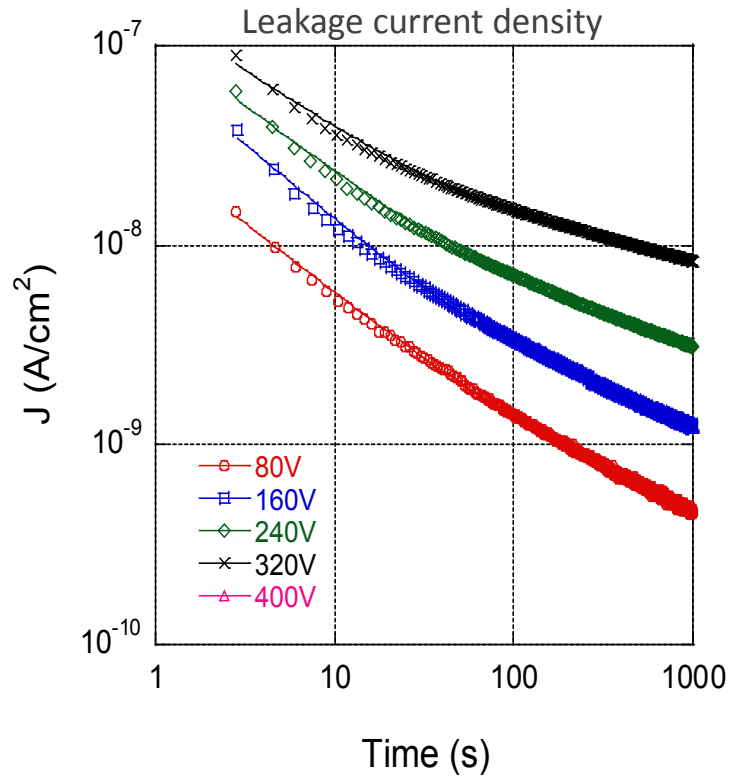
# Specifications for DC-link capacitor & technology that enables PLZT-based capacitor to meet specifications

Feature	Specification	Proposed technology to meet specifications
Volume	$\leq 0.6$ L	High dielectric constant; $\leq 0.3$ L
Voltage	nominal: 325 V; peak: 600 V	High breakdown strength ( $> 200$ V/ $\mu\text{m}$ )
Capacitance	$700 \mu\text{F} \pm 10\%$	High dielectric constant ( $D_k \approx 100@ 450\text{V}$ )
Temperature	$-40^\circ\text{C}$ to $140^\circ\text{C}$	High temperature dielectric material with ferroelectric Curie temperature, $T_c \approx 200^\circ\text{C}$
Ripple Current	165 Arms continuous, 295 A peak	Low ESR ( $\leq 1$ m $\Omega$ ); PLZT performs better as it becomes hotter
Frequency	$\geq 50$ kHz	Ferroelectric based capacitors are used for high frequency applications
Dissipation Factor (DF)	$\leq 0.4\%$ at 1 kHz	DF of PLZT is $\approx 0.4\%$ at 300 V; DF decreases with increase in bias voltage
ESL	$\leq 5$ nH	ESL $\leq 1$ nH have been reported for ferroelectric-based multilayer capacitors
Failure Mode	Benign (open)	Benign failure in single-layer PLZT has been demonstrated. Proposed electrode architecture to obtain self-clearing in multilayer and wound capacitor
Life @ operating conditions	$> 13,000$ h	Highly accelerated life testing (HALT) is proposed to predict capacitor life-time.
Cost	$\leq \$30$	Inexpensive PLZT films and base-metal electrodes will be continuously deposited as is conventionally carried out in the polymer film industry.

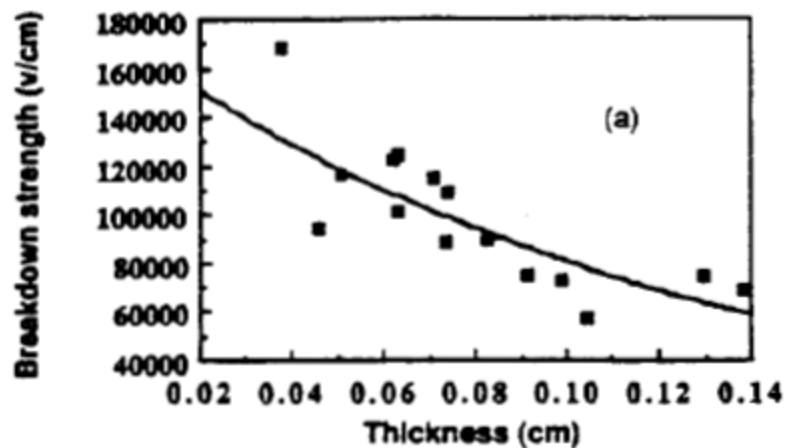
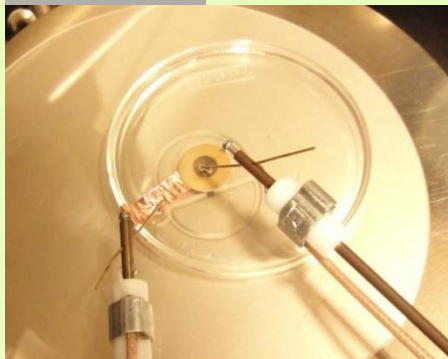
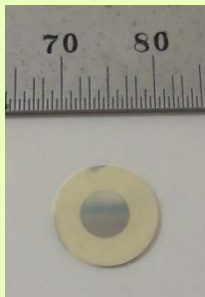
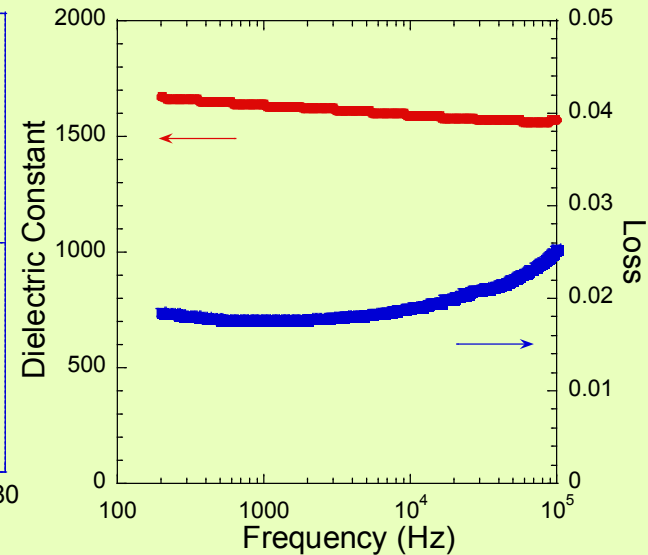
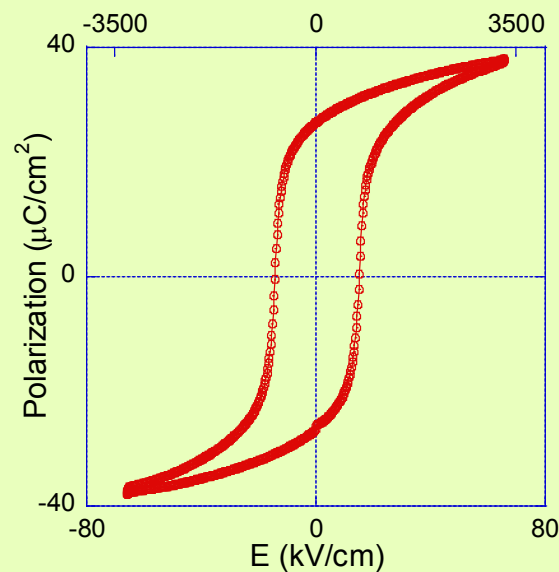
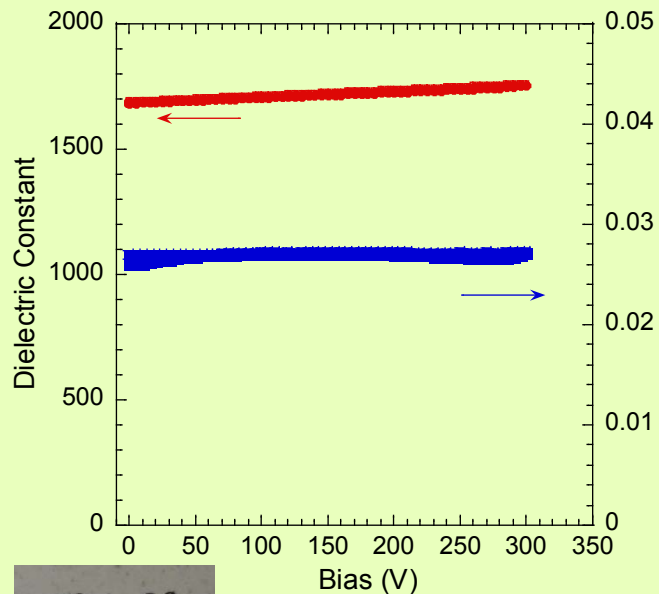
# Benign failure in PLZT film capacitor



# $\approx 8\text{-}\mu\text{m}$ thick AD PLZT/Al/polyimide with large area electrode



# Properties of a $\approx 0.5$ -mm-thick PLZT disk



Breakdown strength as a function of thickness for a hot-pressed PLZT 9.5/65/35 ceramic. Ref: E. Furman & L. E. Cross, ISAF, 577, 1994.