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Exploring Ceramic Component Design Non-Destructively with X-Ray Computed Microtomography

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ABSTRACT

- X-ray computed micro-tomography provides non-invasive three dimensional characterization and visualization of microstructural features within the interior of opaque solid objects. Although a very familiar examination tool in its medical manifestations, it is less known and utilized as a non-destructive technique in the materials community. It offers significant advantages for new product development, process control, materials performance prediction and failure analysis. Recent advances in technology used for generating x-ray computed tomography data has allowed for increased spatial resolution, improved contrast and 3D voxel representations of materials and components. This work will present the results from several studies that have incorporated x-ray microtomography in evaluating ceramic component reliability. As a powerful non-destructive inspection tool, the technique is being applied to characterize material degradation and high voltage breakdown of a ceramic ZnO based varistor component; image the internal macrostructure of a lead zirconium titanate (PZT) composition to qualitatively examine pore size and distribution; and to analyze the formation of internal defects while optimizing a forming process in the development of injection molded components. The interest in these and similar applications is their continued utilization in the production of specific ceramic components for use in various weapons systems. X-ray microtomography has provided the capability to look inside these components and initiate the necessary engineering for improved ceramic component design that leads to increased product reliability.

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Application of X-Ray MicroCT to Materials Science/Engineering Problems

➢ **Areas where the technique offers significant non-destructive evaluation advantages:**

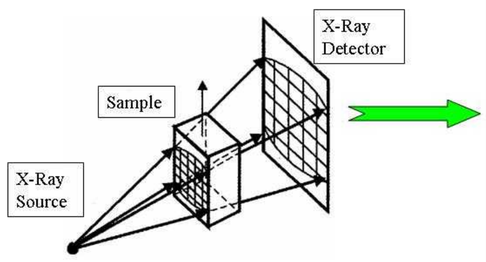
1. Process Control
2. Non-Invasive Metrology
3. Failure Analysis
4. Materials Performance Prediction

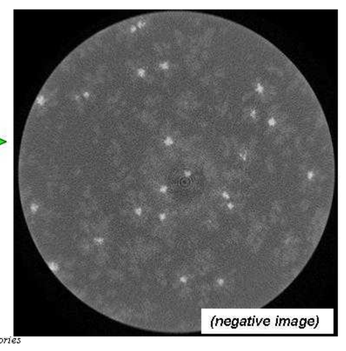
➢ **Case Studies – Active ceramic components for nuclear weapons subsystem applications**

1. **Niobium-Modified Lead Zirconate Titanate (95/5/2 PNZT)**
   - $Pb_{0.991}(Zr_{0.955}Ti_{0.045})_{0.9820}Nb_{0.018}O_3$
2. **ZnO Varistor**
   - High Voltage Breakdown
3. **Powder Injection Molding for Advanced Manufacturability**
   - Varistors

X-ray Microtomography (X-Ray Micro-CT) Used for Non-Destructive Evaluation

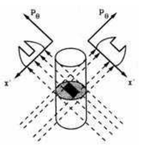
- **X-RAY COMPUTED MICROTOMOGRAPHY**
  - Non-destructive reconstruction of 3D object microstructure
  - Transmission shadow images from a microfocus x-ray source
    - Tungsten target with a beryllium window
  - X-Ray detector
  - Cone-beam reconstruction algorithm



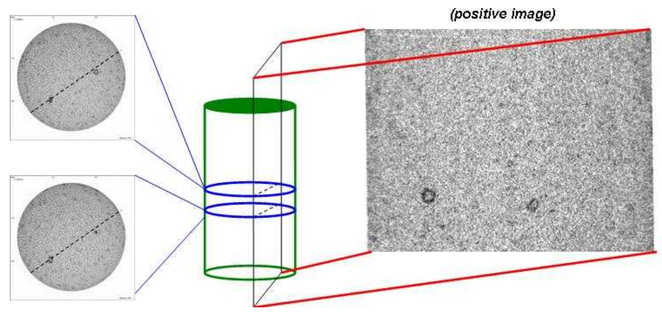


*X-Ray CT images – SkyScan – Microphotonics, FlashCT - Hytec, Inc. and Sandia National Laboratories

2D Pieces of Information from Multi-Perspectives = 3D Representations



- 2D pieces of information + multiple perspectives = 3D representation
  - More revealing than any single view alone
- X-Ray computed Microtomography – creates, digitizes, and stores x-ray shadows
- Mathematical "Reconstruction"
- Voxel-by-voxel pixel map of the material x-ray attenuation coefficients
  - Density, effective atomic number, average photon energy for the incident beam

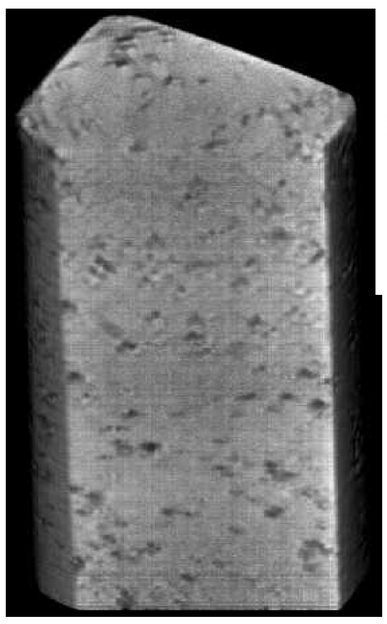


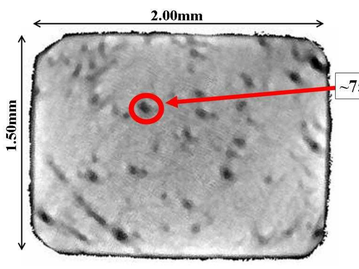
Evaluation of PZT Microstructure

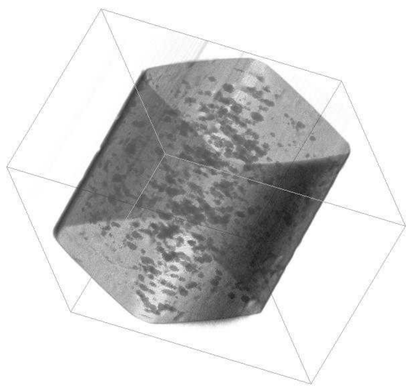
➢ **Niobium-Modified Lead Zirconate Titanate (95/5/2 PNZT)**

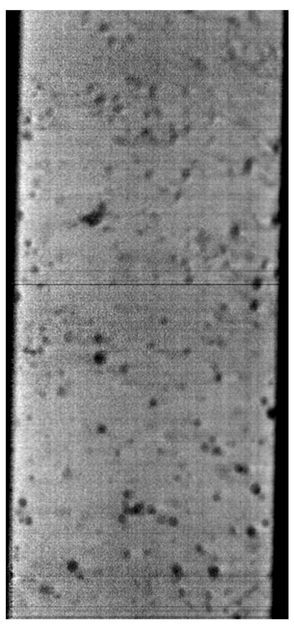
- $Pb_{0.991}(Zr_{0.955}Ti_{0.045})_{0.9820}Nb_{0.018}O_3$

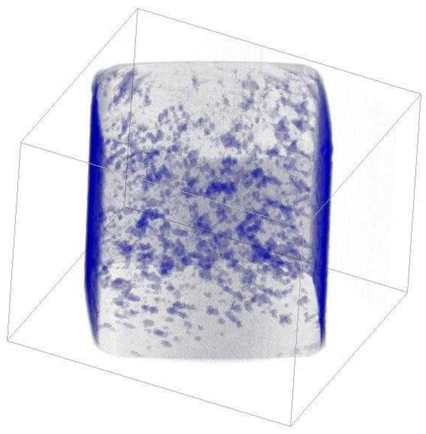
→Evaluate: degree of organic dispersion, microstructural homogeneity and forming defects.











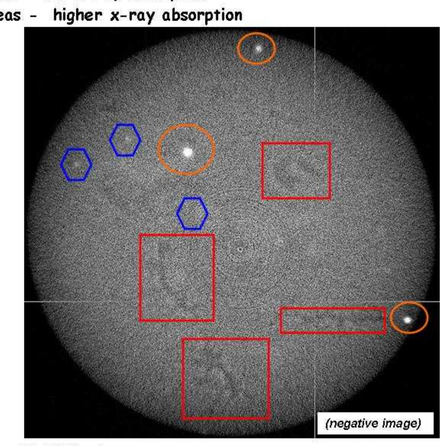
High Voltage Breakdown in ZnO Varistors

➢ Defective regions in the components are potential initiation sites for excessive current concentration that eventually leads to HVB.

X-Ray Micro-CT Reveals Defect Zones

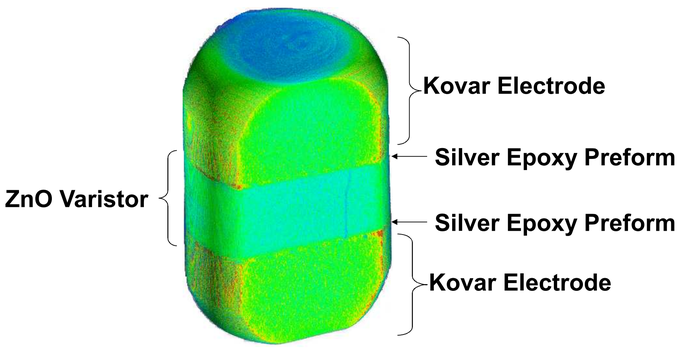
Negative MicroCT Image
- Darker areas - low x-ray absorption
- Lighter areas - higher x-ray absorption

Forming Defects
Delaminations
Cracks/Voids
Inclusions
Microstructural Inhomogeneity

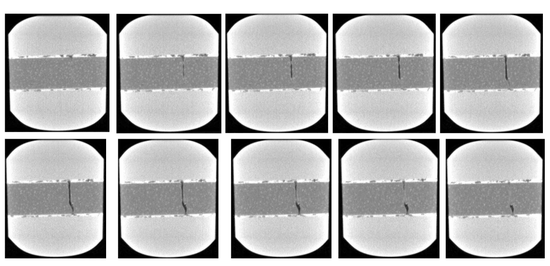


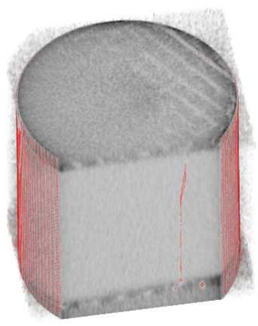
*X-Ray CT images – SkyScan - Microphotonics and FlashCT - Hytec, Inc.

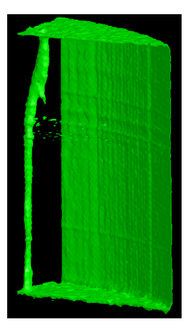
➢ Result = Breakdown Track – 20-30um hollow filament where the current concentration produced localized heating and eventual melting of the material.

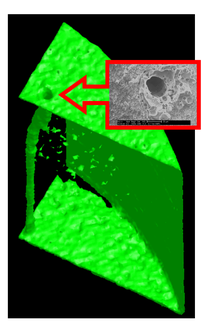


➢ 2D layers are used to construct 3D images of breakdown.









Optimizing Injection Molding Parameters

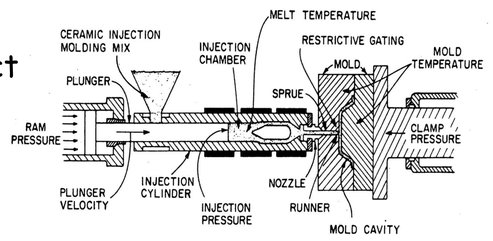
➢ Hydrostatic forming technique for shaping powders using organic binders and fillers.

➢ Thermoplastic based organic system is mixed with ceramic powder composition.

o Organics Ratio:
- 5wt% Stearic Acid - Surfactant
- 45wt% Proflow 3000 – Polypropylene and Polyethylene Random Copolymer (Plasticizer for wax)
- 50wt% Paraffin Wax – Straight-Chain Hydrocarbon

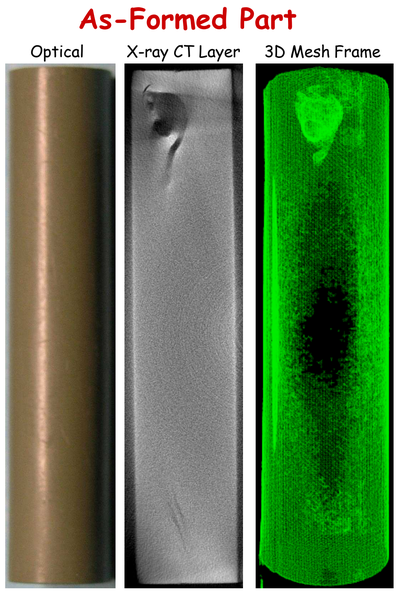
➢ Numerous molding parameters can be adjusted that affect component quality.

➢ X-Ray microCT is used to evaluate optimizing these parameters.

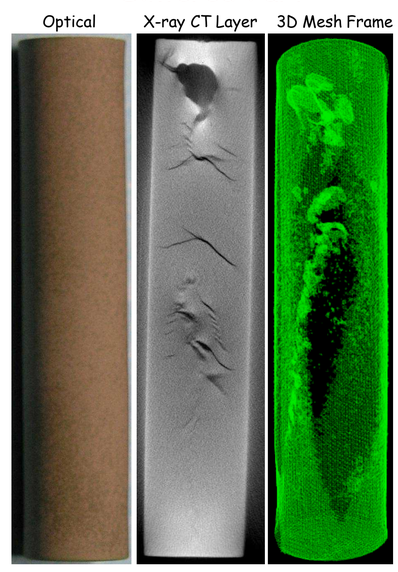


➢ Defects created during forming result in low quality sintered components.

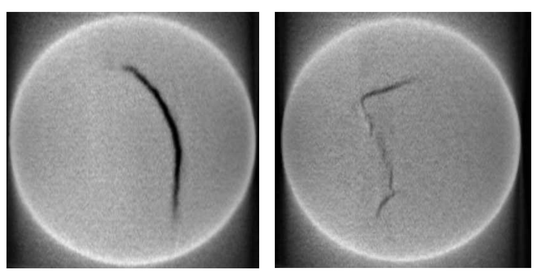
As-Formed Part



Sintered Part



➢ Smile face and S-shaped defects due to selected molding parameters.



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