



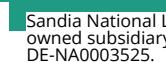
Sandia University Partnerships Network
GEORGIA TECH



Analysis of Thermal and Mechanical Cycling Effects on Energetic Materials with Digital Image Correlation



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Presented by

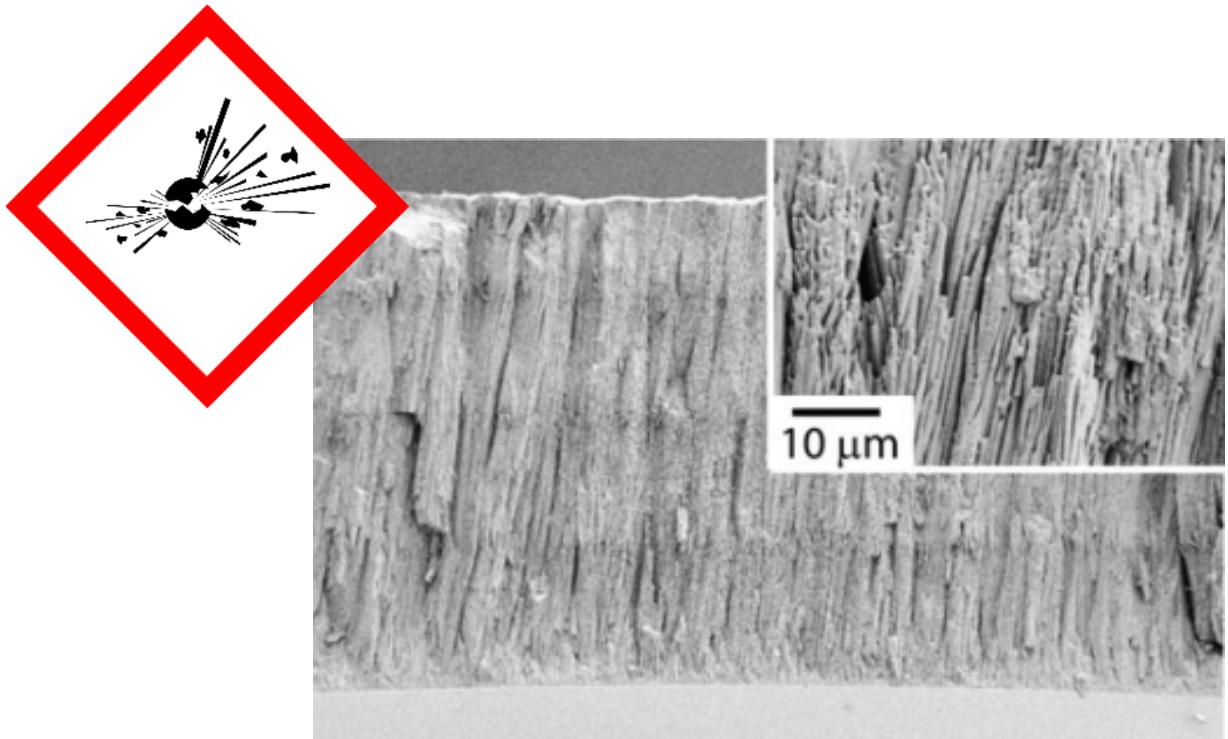
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Introduction and Motivation

- Understanding the stability and effect of aging is important for ensuring safety during manufacturing, transport, and storage
- Stockpiled energetic materials may need to be stored for long periods
- Analysis of how energetic materials perform after aging can give insight into the effectiveness and shelf life of existing equipment



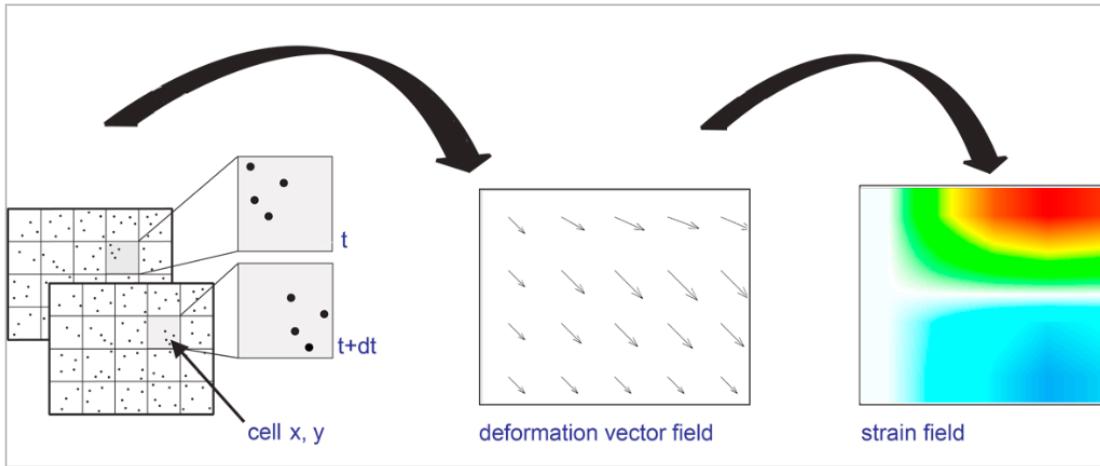
SEM image of a cross-section of a PETN thin-film sample [1]. PETN is an energetic material used in many applications.

[1] Knepper, R., Tappan, A., Wixom, R., & Rodriguez, M. (2011). Controlling the microstructure of vapor-deposited pentaerythritol tetranitrate films. *Journal of Materials Research*, 26(13), 1605-1613. doi:10.1557/jmr.2011.177

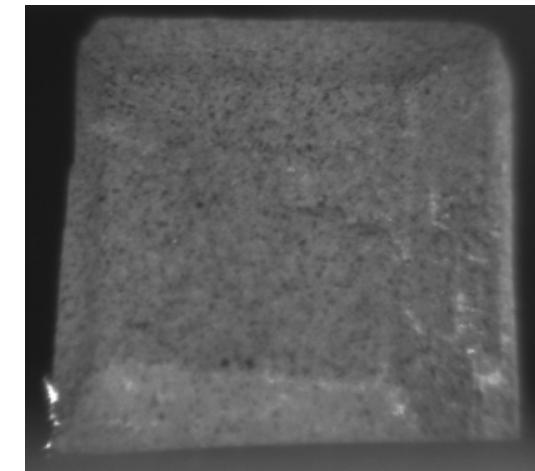


Introduction and Motivation – Digital Image Correlation

- Digital image correlation (DIC) is an optical techniques used to visualize deformation in samples
- DIC can be used to analyze the strain, fracture, and thermal expansion in samples of energetic materials and simulants as they are thermally cycled
- Requires random speckle patterns be applied to sample



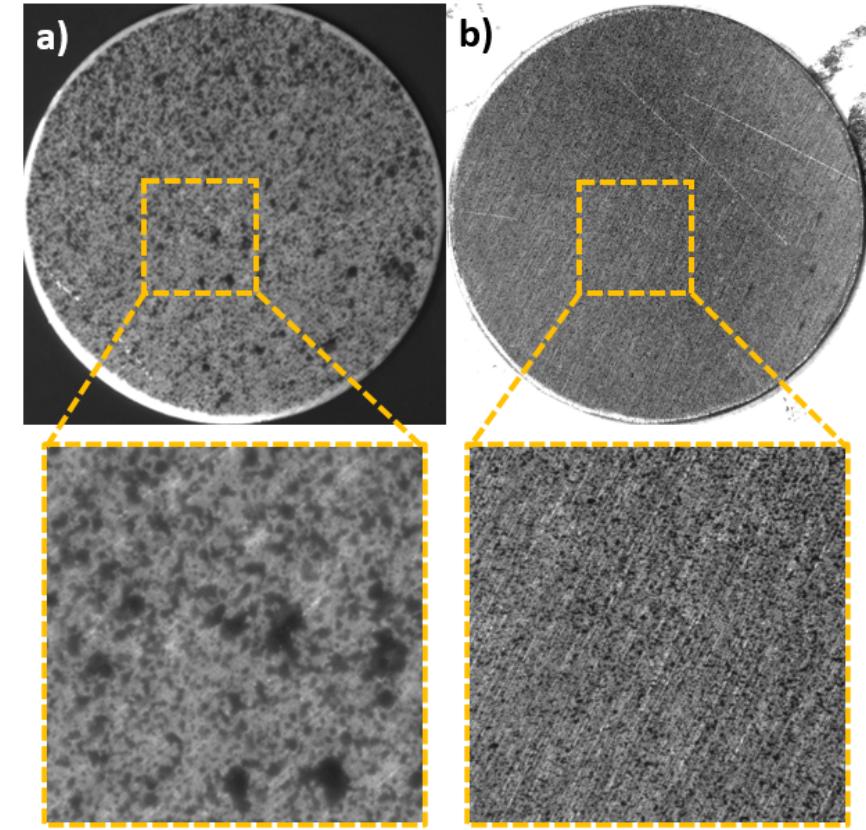
Obtaining strain field from DIC, LaVision: <https://www.lavision.de/en/techniques/dic-dvc/>



A patterned crystal of sucrose used in a DIC experiment. Sucrose is used a surrogate for PETN.

Digital Image Correlation – Speckle Pattern

- Ideal speckle size between 3 to 5 pixels
- Smaller speckles lead to aliasing, while speckles that are too large reduce spatial resolution
- Spray painting and airbrushing tested for pattern application
- Spray paint speckle size distribution not very uniform, larger speckles overall
- Airbrush yielded more uniform speckle size distribution with more speckles within the optimal size range

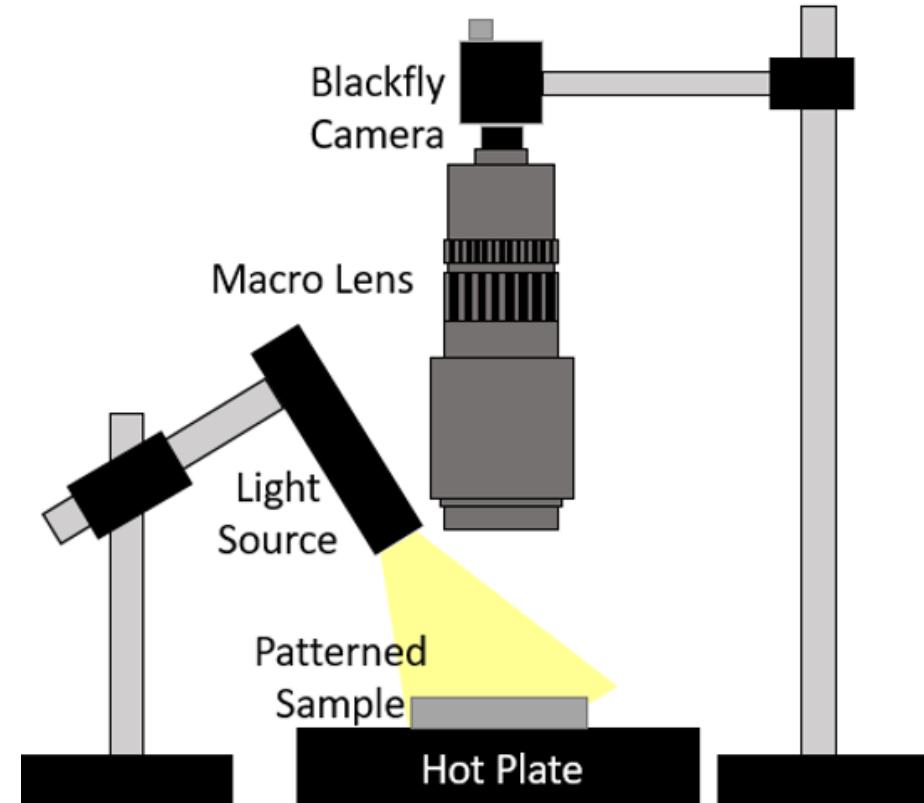


a) Sample with spray painted pattern and
b) sample with airbrush pattern are shown



Experimental Setup

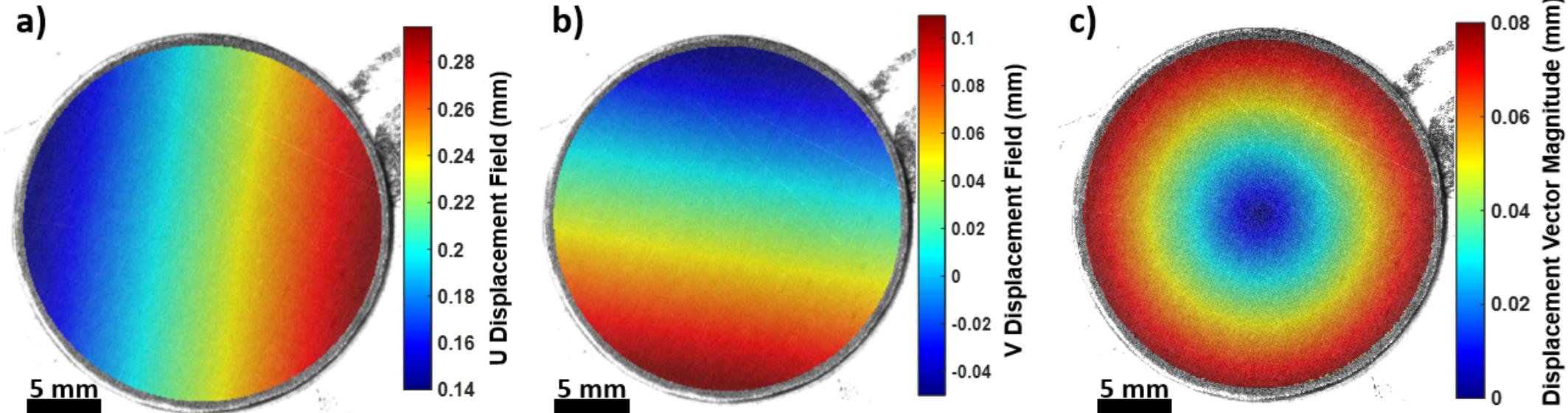
- A Blackfly CMOS camera (BFS-U3-32S4M, 3.45 μm pixels) and Navitar Zoom 7000 macro lens used for imaging
- Sample heated on hot plate with enclosure to ensure entire sample is at the same temperature
- A fan is used to reduce heatwave distortions when required





DIC Analysis

- DIC analysis spatial resolution depends on subset and speckle size
- Rigid body motion removed from displacement field to obtain thermal strain field

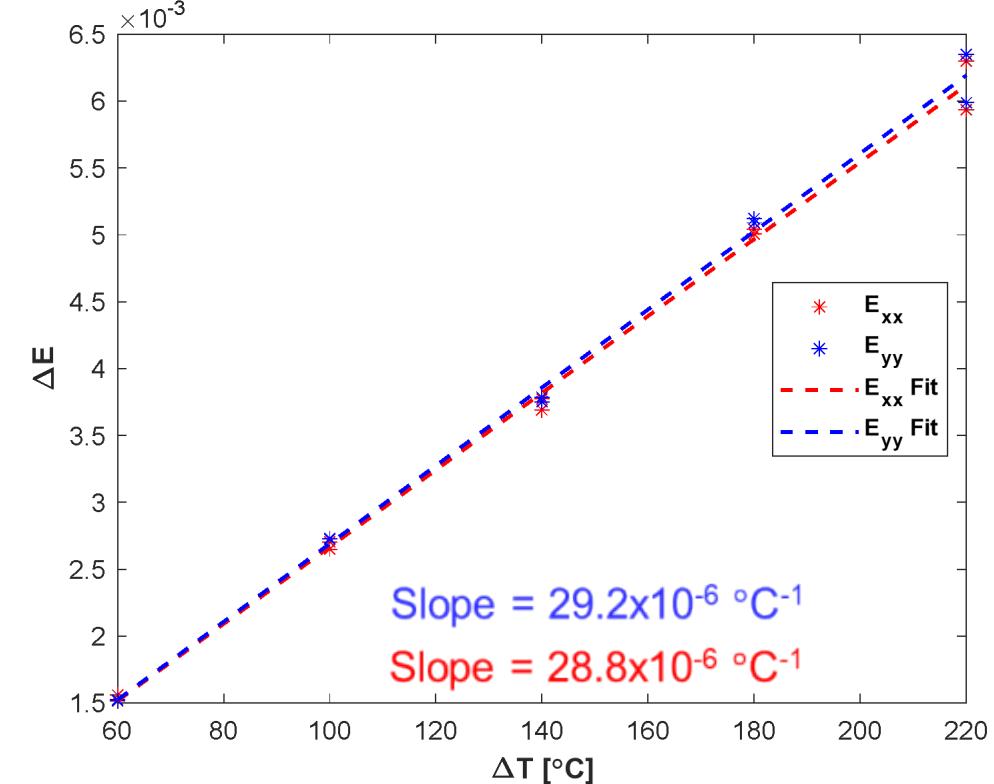


a) U-displacement, b) V-Displacement, and c) total displacement field for a sample of aluminum is shown after DIC analysis and removal of rigid body rotation and translation.



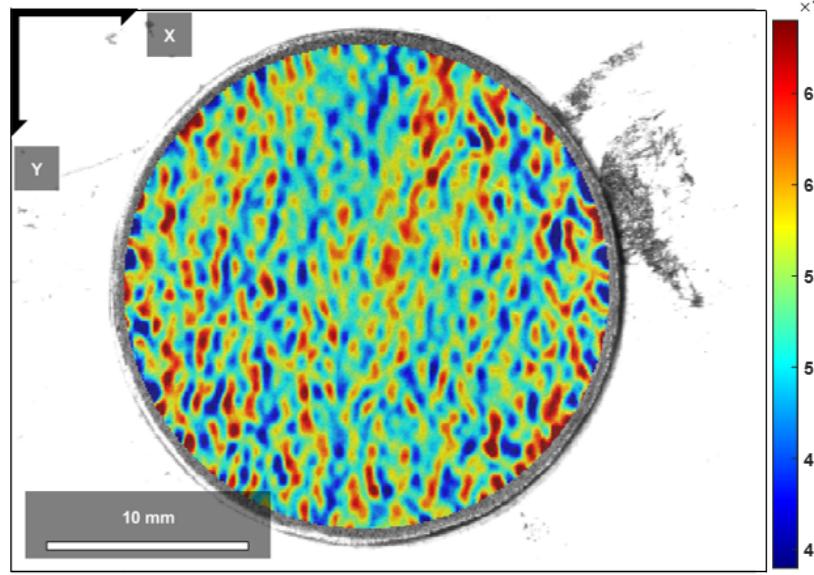
DIC Setup and Algorithm Validation

- DIC used to experimentally measure the CTEs of known materials (aluminum, zinc, and polycarbonate)
- Samples heated from 20°C to 240°C for metals and 160°C for polycarbonate
- Strain calculated for each temperature difference and fitted to obtain experimental CTE

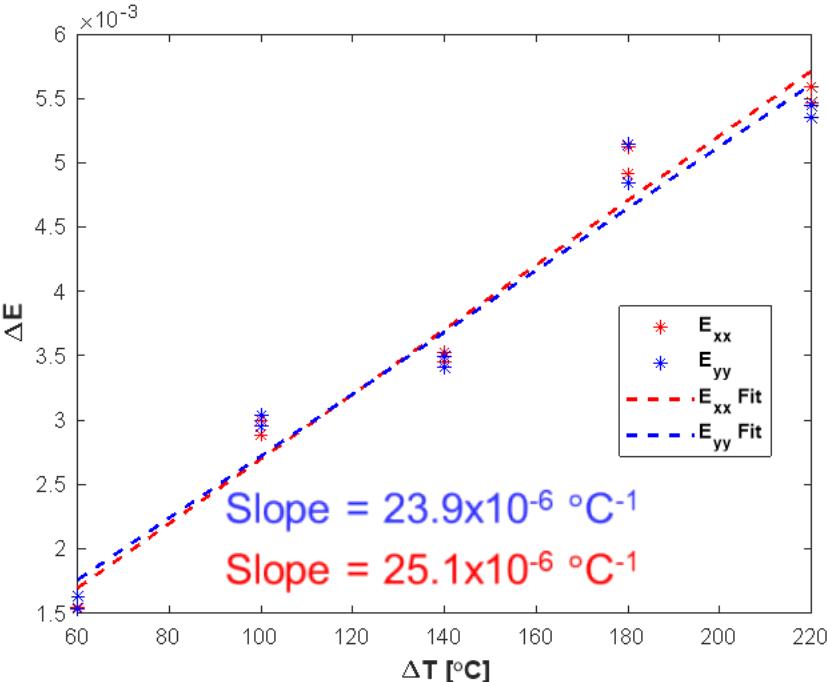


Thermal strain vs. temperature difference for a sample of zinc. The slope of the fitted line is the experimentally measured CTE. Literature values for the CTE of zinc range from 30 to $35 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$.

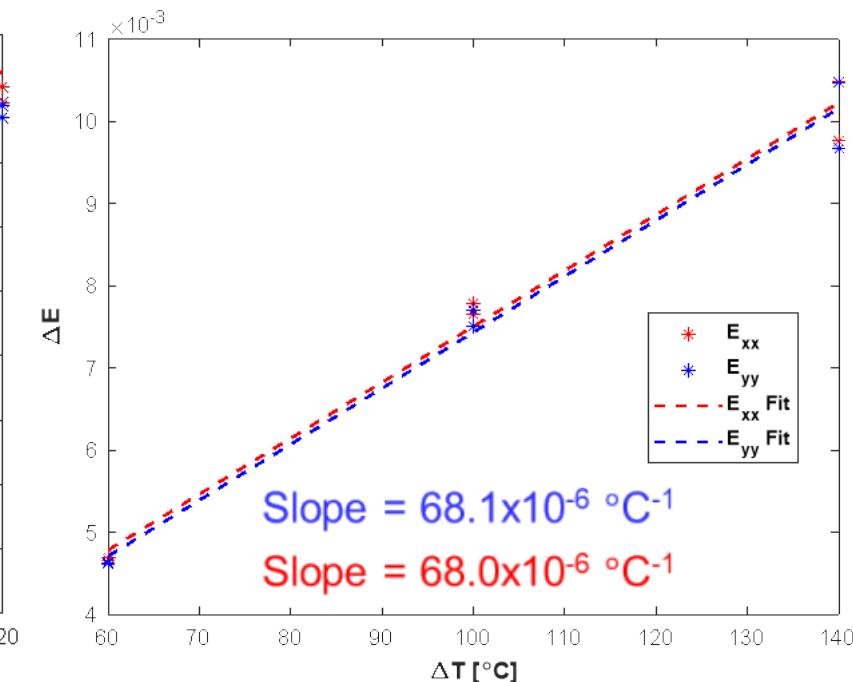
DIC Setup and Algorithm Validation Continued



E_{xx} strain result from an aluminum sample. The average strain is used for the fit and resulting CTE calculation.



CTE results for an aluminum disc sample. Literature values for the CTE of aluminum range from 23 to $30 \times 10^{-6} \text{ } {}^\circ \text{C}^{-1}$.



CTE results for a polycarbonate sample. Literature values for the CTE of polycarbonate are around $68 \times 10^{-6} \text{ } {}^\circ \text{C}^{-1}$.

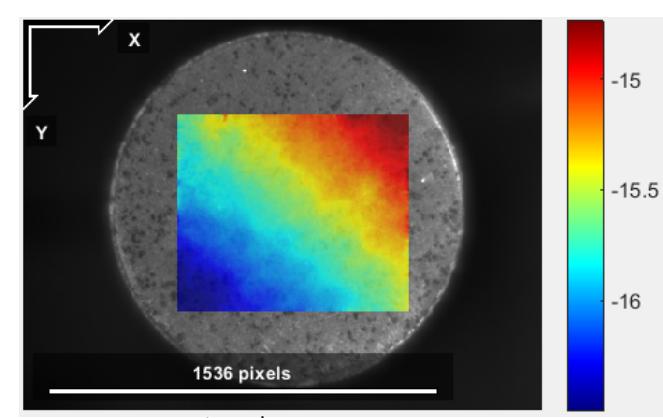
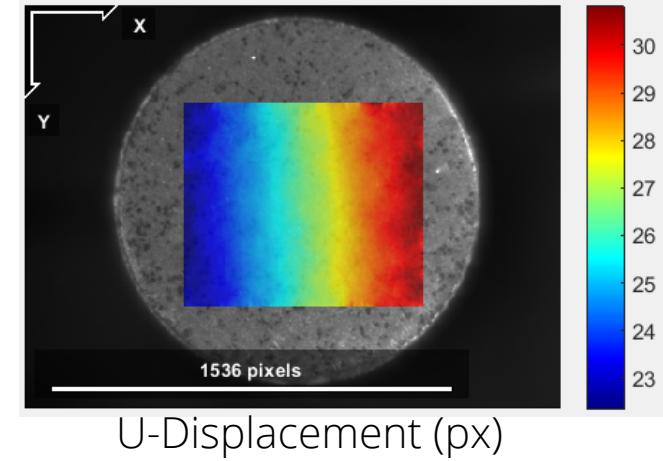


Thermal Cycling of Fe/KClO₄ Pellets

- Contrast between Fe and KClO₄ provide decent pattern
- Initial tests show consistent displacement and strain values
- Resulting strain/displacement values similar to TKP pellets

$T_0 = 20^\circ\text{C}$	$T_1 = 190^\circ\text{C}$	$T_2 = 24^\circ\text{C}$	$T_3 = 189^\circ\text{C}$	$T_4 = 24^\circ\text{C}$	$T_5 = 190^\circ\text{C}$	$T_6 = 35^\circ\text{C}$
Average strain E_{xx}	0.0091	0.0011	0.0090	0.0018	0.0090	0.0026
Average strain E_{yy}	0.0098	0.0011	0.0098	0.0018	0.0097	0.0026
$E_{xx}/\Delta T (10^{-6})$	53.63	-	53.34	-	52.84	-
$E_{yy}/\Delta T (10^{-6})$	57.37	-	58.07	-	57.28	-

DIC analysis results for an Fe/KClO₄ pellet with ~32% porosity.



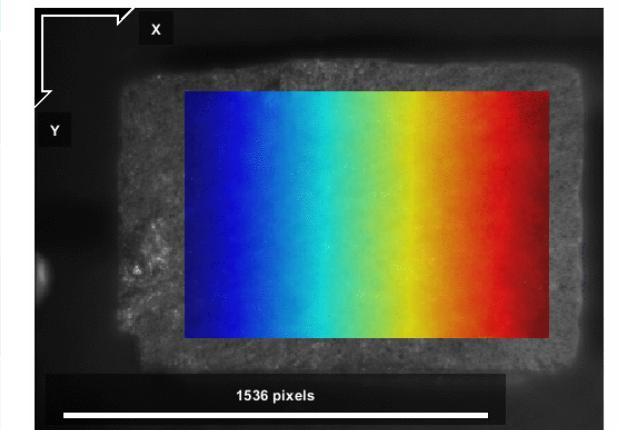
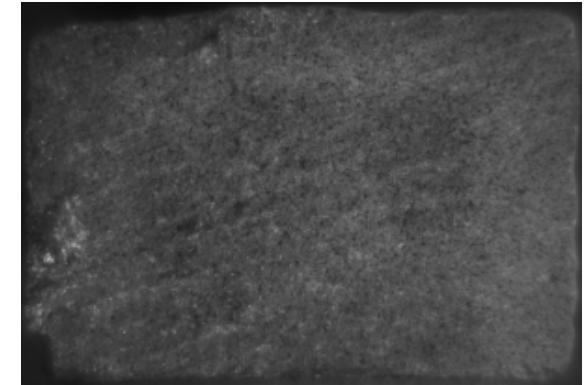


Thermal Cycling of Crystalline EM Surrogate

- Sucrose crystals used as surrogate for PETN
- Initial tests have some noise and drift over time

$T_0 = 22.1^\circ\text{C}$	$T_1 = 120^\circ\text{C}$	$T_2 = 31^\circ\text{C}$	$T_3 = 120^\circ\text{C}$	$T_4 = 23.7^\circ\text{C}$	$T_5 = 120^\circ\text{C}$	$T_6 = 22^\circ\text{C}$
Average strain E_{xx}	0.0077	0.0004	0.0081	0.0001	0.0078	-0.0001
Average strain E_{yy}	0.0073	0.0005	0.0077	0.0001	0.0073	-0.0002
$E_{xx}/\Delta T (10^{-6})$	78.48	-	82.35	-	79.61	-
$E_{yy}/\Delta T (10^{-6})$	74.77	-	79.15	-	74.60	-

DIC analysis results for an airbrush patterned sucrose crystal.

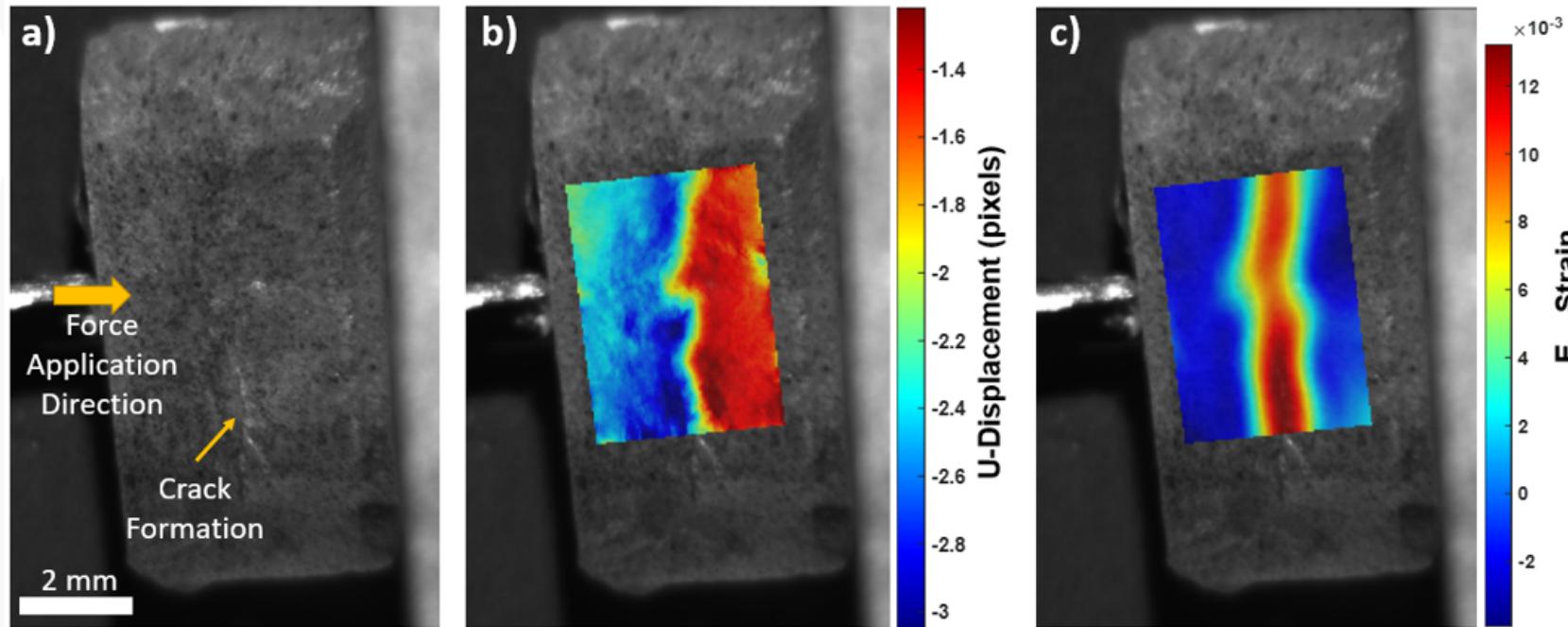


U-Displacement field (px) showing increased displacement on high temperatures of cycles



Mechanical Fracture of Crystalline EM Surrogate

- Sucrose crystal fractured with force applied at edge of crystal
- Fracture can be visualized in the displacement and strain field

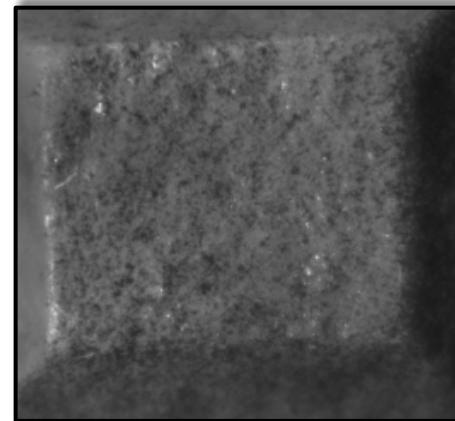
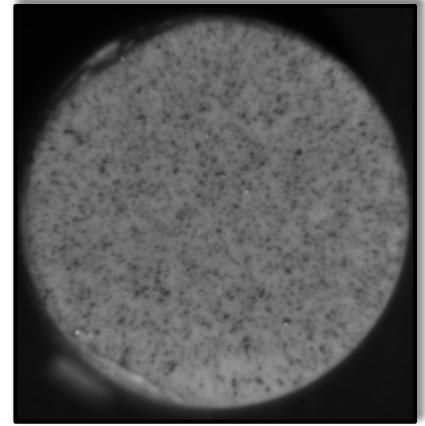
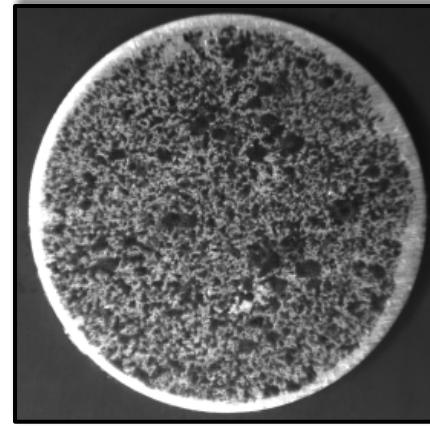


a) An image of the patterned sucrose crystal is shown as force is applied to the crystal and a crack forms.
The b) u displacement field and c) E_{xx} strain field resulting from DIC analysis is shown



Next Steps and Future Work

- Challenges
 - Magnitude of thermal strain is typically small, sensitive to noise
 - Take several images and average to reduce noise impact
 - Fan can be used to reduce heatwave distortion
 - Fracture propagation in crystalline material is very fast
 - Need high-speed cameras to capture fractures
- Next Steps
 - Increase number of cycles using thermoelectric unit
 - Vary ramp rate and soak time
 - Try experiments with PETN and other energetic materials





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Questions?