Chapter 6
Overcoming Deformation Measurement Limits Involving Material Fragmentation During In-situ Micro-Computed Tomography via Backward Digital Volume Correlation

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Abstract Quantifying internal damage evolution in particulate composites, such as plastic-bonded explosives (PBX), remains a significant challenge due to large localized deformations and complex crack networks. While in-situ X-ray micro-computed tomography (μCT) combined with Digital Volume Correlation (DVC) is a standard tool for 3D strain mapping, conventional DVC often loses correlation in regions of severe cracking or interfacial failure. This study presents a backward incremental DVC approach designed to maintain high-fidelity displacement tracking in the presence of significant structural discontinuity and fragmentation. In contrast to conventional methods, the backward DVC framework treats the final damaged geometry as the reference; this effectively preserves the integrity of displacement fields in regions of severe crack coalescence where forward tracking often fails. This technique was applied to a mock PBX composite consisting of IDOX crystals (75–150 μm) embedded in an Estane binder. Cylindrical specimens were subjected to unconfined compression with sequential μCT scans captured in-situ. Results demonstrate that the backward approach significantly improves the resolution of displacement fields near crack-affected zones, enabling the first-of-its-kind detailed observation of grain-binder delamination and crack coalescence in these materials. This methodology provides a robust framework for generating high-fidelity experimental datasets essential for validating grain-scale numerical simulations.

Keywords μCT · Backward DVC · Particle composite

Introduction

Plastic-bonded explosives (PBXs) are complex energetic composites consisting of a high volume fraction (80–97%) of energetic crystals embedded within a polymeric binder. This architecture is designed to maximize detonative power while

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ensuring the safe handling of the material by preventing unintended detonation from shocks or impacts. Recent literature has established that the mechanical performance of PBXs is highly sensitive to processing conditions such as packing density, binder type, and thermal history [1, 2]. Furthermore, enhancing both functional and mechanical properties often requires a trade-off; increasing crystal content improves detonation but typically compromises the structural integrity of the composite [3, 4, 5]. To mitigate the risks and costs associated with testing live explosives, inert "mock" surrogates like the IDOX/Estane composite are utilized. Conducted research [6, 2, 7, 8] has demonstrated that IDOX/Estane serves as a reliable surrogate for PBX 9501 across a wide range of strain rates and temperatures, making it an ideal candidate for studying the fundamental fracture mechanisms of particulate composites. While traditional surface-level characterization via Digital Image Correlation (DIC) has been effective for observing macroscopic failure patterns and strain localization [9, 7], it offers limited insight into the internal sub-surface deformation and grain-scale interactions that govern bulk response. Digital Volume Correlation (DVC) has emerged as a superior alternative, utilizing in-situ X-ray micro-computed tomography (μCT) to resolve three-dimensional deformation fields [10, 11]. However, as detailed in [12], standard "forward-marching" DVC algorithms often struggle with decorrelation in regions of severe cracking or interfacial delamination. To address this, the present study utilizes a backward incremental DVC technique, which performs correlations from the peak-deformed state back to the undeformed configuration. This approach facilitates more stable convergence in damaged regions and provides a clearer view of fragmentation and crack coalescence. By successfully capturing these complex internal mechanisms, this work provides high-fidelity experimental data necessary for validating multiscale numerical models that account for heterogeneous grain interactions.

Materials and Methods

Specimen Fabrication and Preparation

As an inert surrogate for HMX, an IDOX/Estane composite was synthesized. Recrystallized and ground IDOX crystals (75–150 μm) were blended with Estane resin at a 19:1 weight ratio (95% particulate loading). The mixture was hot-pressed at 50°C and 152.8 MPa for one minute, then machined into 5×5 mm cylindrical specimens optimized for unconfined compression.

In-Situ X-ray μCT Characterization

In-situ imaging was performed using a ZEISS Xradia 520 Versa integrated with a loading frame (schematic in Figure 1a). Specimens, lubricated with petroleum jelly to prevent barreling, underwent unconfined compression at 0.5 mm/min. Volumetric data were captured at six engineering strain levels: 0.0%, -4.0%, -8.0%, -12.0%, -13.2%, and -14.4%. Scans utilized 160 kV, 50.3 μA, and an 8-second exposure per projection over 360°, yielding a 12.93 μm isotropic voxel size. Reconstructed volumes were cropped to $601 \times 601 \times 501$ pixels (8-bit UINT) for computational efficiency.

Backward Incremental Digital Volume Correlation (DVC)

Deformation was quantified using the open-source IDVC framework. To overcome tracking failures caused by severe cracking, a backward incremental DVC approach was implemented as shown in Figure 1b. Correlation initiates at the peak deformed state ($\epsilon_z = -14.4\%$) and tracks back to the undeformed reference ($\epsilon_z = 0\%$), leveraging structural continuity to stably place control points around fractures as depicted in Figure 1b. The correlation of the backward DVC algorithm in minimizing the normalized sum of square differences (NSSD) is also shown.

The displacement vector field $\mathbf{u}$ is determined by minimizing the normalized sum of squared differences correlation objective function C:

$$C = \sum_{x \in \Omega} \left(\frac{g(x-u) - \bar{g}}{\sigma_g} - \frac{f(x) - \bar{f}}{\sigma_f} \right)^2 \tag{1}$$

Here, $f(x)$ and $g(x)$ represent reference and deformed intensity values; $\bar{f}$, $\bar{g}$, σ_f, and σ_g denote their respective means and standard deviations within the sub-volume domain Ω. Computations utilized 40-pixel cubic sub-volumes with 10,000 randomly distributed sampling points, tricubic interpolation for sub-voxel accuracy, and the Newton-Raphson method for convergence.

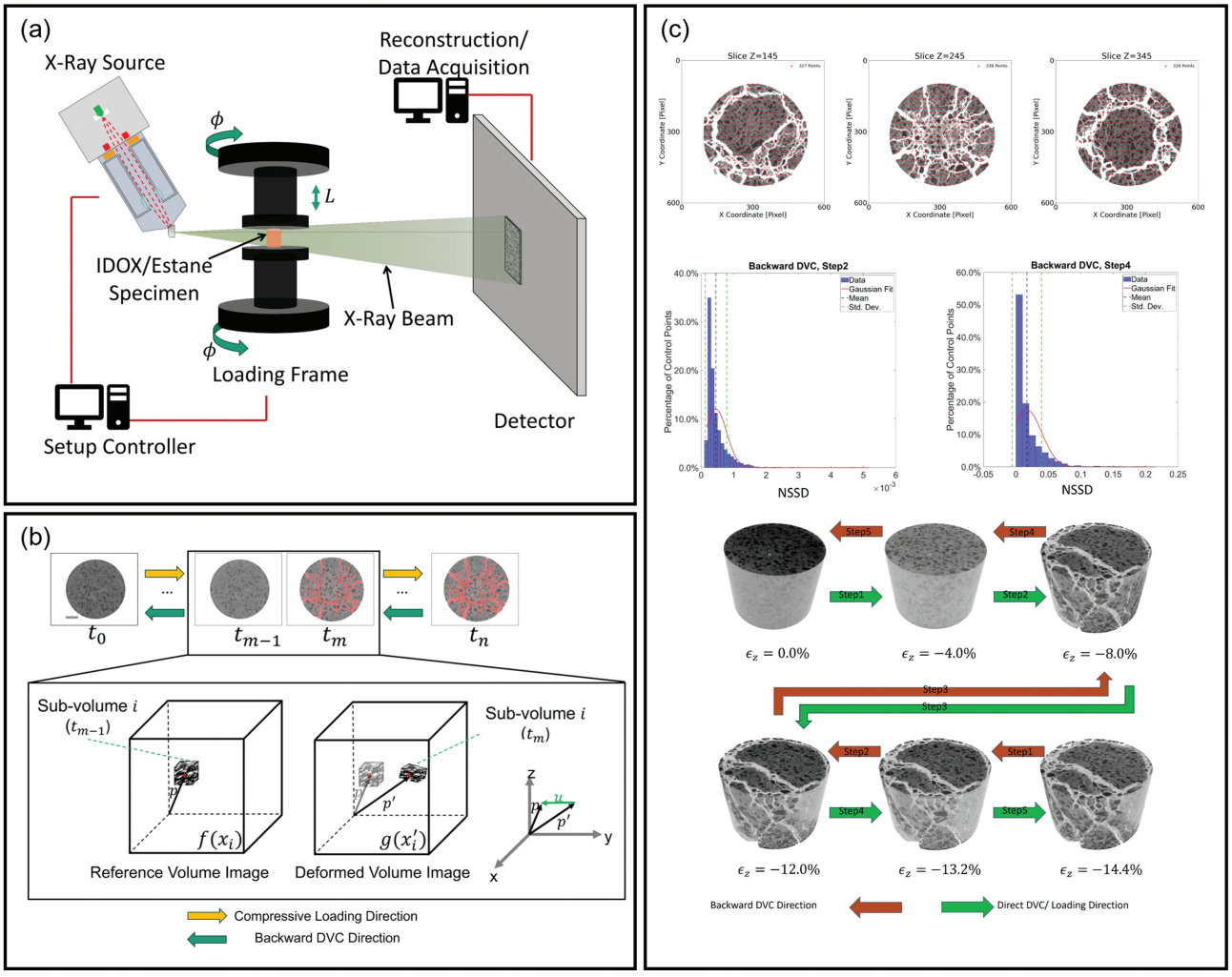

Fig. 1 (a) The schematic of X-ray µCT and loading frame setup to conduct an in-situ load/scan experiment (b) The schematic of sub-volume tracking in backward incremental DVC analysis (c) The distribution of control points located at material points to track the displacement field in the backward incremental DVC method, convergence behavior of the backward incremental DVC minimization algorithm under extreme and minimal damage restoration scenarios, forward and backward incremental DVC direction demonstration

Results and Discussion

In-situ X-ray μCT analysis provided a detailed volumetric visualization of internal damage evolution in the IDOX/Estane composite. During the initial loading phase ($\epsilon_z = 0.0\%$ to -4.0%), the specimen exhibited minor rigid body rotation and uniform compression without macroscopic cracking. However, at $\epsilon_z = -8.0\%$, significant damage initiated, characterized by binder cracking, interfacial debonding, and crystal grain fracture. As strain increased to -14.4%, deformation transitioned from crack initiation to the expansion and coalescence of existing networks. Cross-sectional views revealed a non-uniform, X-shaped fracture pattern aligned at $\sim 45^\circ$ to the loading axis, indicative of shear-dominated failure. This partitioned the specimen into a continuous central core and fragmented peripheral patches, demonstrating a highly heterogeneous mechanical response.

Volumetric rendering demonstrated that isolated 2D slice analysis is insufficient to resolve full mechanical behavior due to complex, non-uniform crack paths. However, applying 3D forward-marching DVC proved computationally challenging, as large crack openings at the second loading step caused widespread correlation failure. To maintain tracking fidelity, a backward incremental DVC approach was implemented, using the peak deformed state ($\epsilon_z = -14.4\%$) as the reference to

"virtually unload" the specimen. A thresholding method restricted control points to the material domain, placing them stably up to the crack edges. The algorithm exhibited robust convergence; even during the most challenging phase of extreme damage restoration (virtual crack closure step), only 10 control points (0.23%) failed to correlate. Gaussian fits of the normalized sum of squared differences (NSSD) confirmed high correlation quality, with per-step runtimes between 1000 and 2485 seconds on a dual Intel® Xeon® workstation.

Systematic inversion of the backward displacement fields reconstructed a direct DVC solution. The resulting fields (Figure 2) successfully resolved complex internal motions and boundary-driven gradients. A significant increase in displacement magnitude drove the initial crack fragmentation, after which displacement patterns varied sharply across the isolated patches—the right-side zone exhibited substantial motion while the bottom region remained relatively stationary. Furthermore, the top region of the specimen exhibited displacement magnitudes nearly an order of magnitude higher than the fixed base, reflecting the influence of the moving platen. Ultimately, the backward DVC methodology successfully quantified the complex, inhomogeneous internal deformation of the composite, providing high-fidelity data for evolutional investigation.

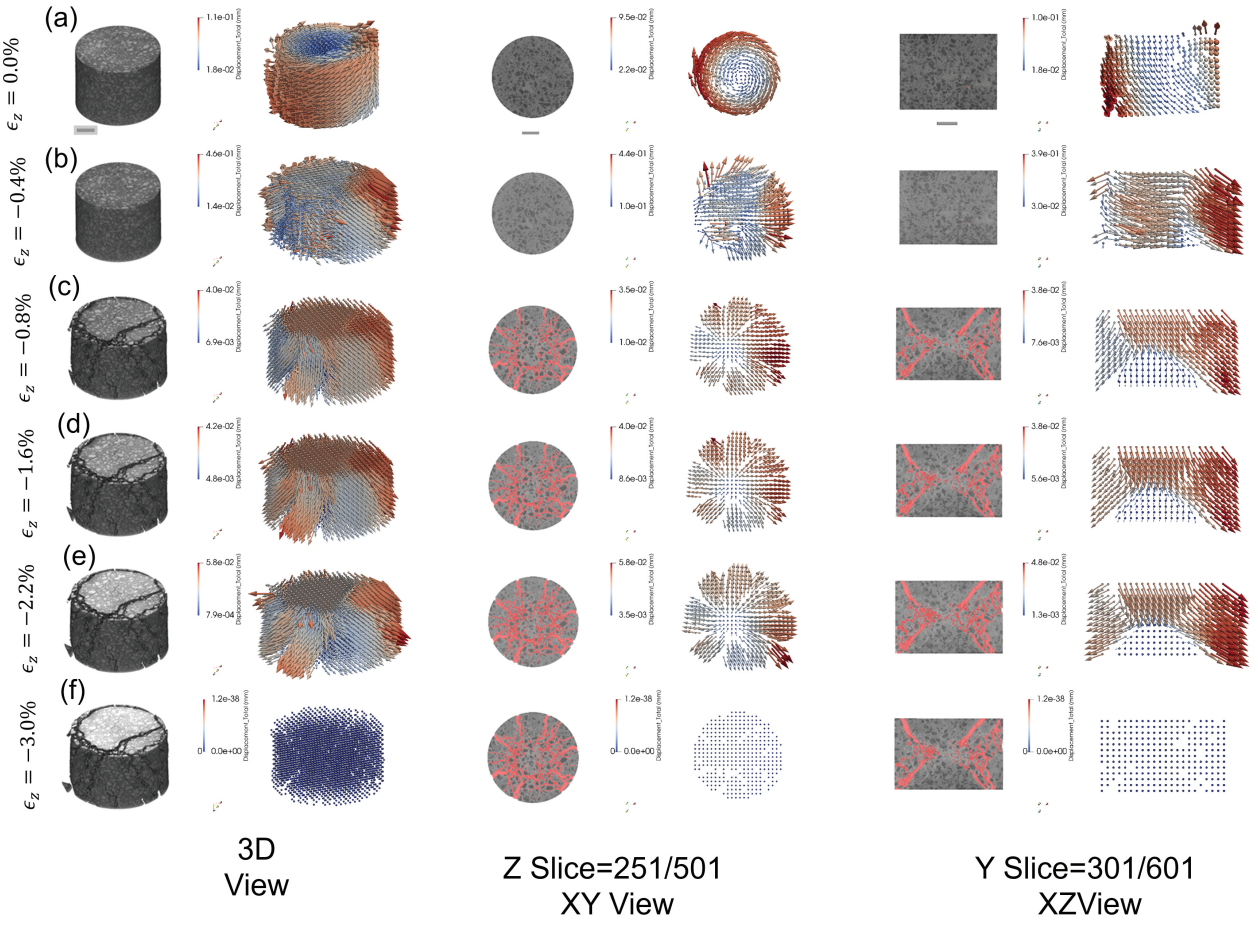

Fig. 2 The progress of the displacement fields at each loading phase, shown in 3D, isolated surface in XY plane (Z slice 251/501), and isolated surface in XZ plane (Y slice: 301/601): (a)$\epsilon_z = 0.0\%$, (b)$\epsilon_z = -4.0\%$, (c)$\epsilon_z = -8.0\%$, (d)$\epsilon_z = -12.0\%$, (e)$\epsilon_z = -13.2\%$, (f)$\epsilon_z = -14.4\%$.

Conclusion and Future Work

This study validates the effectiveness of the backward incremental DVC technique for characterizing the internal mechanics of highly filled particulate composites. By utilizing IDOX/Estane as a high-fidelity inert surrogate for PBX-9501, we identified two primary fracture modes: patchy fractures driven by local grain interactions and macroscopic shear bands forming an X-shaped morphology. The results quantify the transition from elastic-viscous flow to brittle failure and provide high-fidelity

experimental data necessary for validating multiscale numerical models. Ultimately, this work establishes a reliable framework for investigating the internal structural evolution of energetic-like materials, bridging the gap between microscopic damage mechanisms and bulk mechanical reliability.

Future work will focus on utilizing these high-fidelity 3D kinematic datasets to develop and validate micro-mechanics-informed damage criteria for particulate composites, as well as applying the backward DVC framework to track failure mechanisms under varying loading conditions in KelF/F50 polymer sand composite.

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