Chapter 4
Surface Deformation Measurements of a Composite Cylinder Composed of Particulate Idoxuridine Crystals Embedded in Estane Under Compression

Pooyan B. Javadzadeh, Ehsan Mehrdad, Yao Ren, Nathan Peterson, Amy Clarke, Hongbing Lu

Abstract 5'iodo'2 idoxuridine has served as a reliable replacement for plasticly bonded explosives (PBX) in university environments to characterize mechanical behavior under quasi-static and dynamic testing conditions. Particulate idoxuridine crystals with particle sizes in the range of 75-150 μm are embedded in Estane polymer matrix to form a particulate composite cylinder specimen of 5 mm diameter. A metallic ink marker is applied to the surface of the composite cylinder, and volumetric images of the specimen are obtained using micro-computed tomography (μCT) at approximate 4 $\mu m/pixel$ resolution to determine the internal microstructure, including any voids. The metallic ink markers are distinguishable in each μCT volumetric image, and in the surface images of the same specimen, allowing for the correlation of internal microstructure to the surface speckles. The μCT pre-scanned specimen is compressed under quasi-static loading condition, where a stereo-microscope with a resolution of 4.0 μm/pixel is used to acquire the surface stereo images of the specimen undergoing deformation. The surface stereo images are analyzed using stereo (or 3D) digital image correlation to determine the surface axial, circumferential, and shear strains. These strain results are correlated to the internal microstructure for validation of meso-scale simulations of the specimen under compression. An 80-feet-long split Hopkinson pressure bar is used to apply compression while a high speed camera is used to acquire surface images at ambient temperature. Digital image correlation is used to determine the surface strains of the specimen. The markers again allow for correlation of the surface deformations to the microstructure. These experimental results provide detailed internal microstructure of the particulate composite specimen, which are correlated to the measured surface strains under both quasi-static and dynamic loading conditions. The results can be used for verification and validation of simulations of the mechanical response of particulate materials by direct numerical simulation (DNS), and to assist in the development of a computational multiscale micromorphic continuum mechanics framework.

Keywords Granular mechanics · Particulate composites · Micro-computed tomography · Digital image correlation · IDOX/Estane

Introduction

Numerous surrogate materials have been used for energetic particles in the plastic-bonded explosives (PBX. These include pentaerythritol, acetaminophen, and sucrose [1], [2], [3]. Each of such inert materials has its advantages and shortcomings. Yeager et al. [4] investigated multiple candidates with properties similar to HMX, these included IDOX, MDTTO, MET-I, N-BPFPO, PFBA, and PFBBA, they found that IDOX, N-BPFPO, and PFBA showed similar processability characteristics and thermal behavior. Later, Yeager et al. [5] investigated the thermal stability, density, crystal structure, and processing characteristics of IDOX, N-BPFPO, and PFBA, and concluded that the IDOX-based mock is less water-soluble than current

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Paul Allison et al. (eds.), *Dynamic Behavior of Materials, Vol. 1*, Conference Proceedings
of the Society for Experimental Mechanics Series, https://doi.org/10.13052/97887-438-0827-5_4

surrogate materials. Burch et al. [6] performed nanoindentation on IDOX crystals to investigate the mechanical properties of the powder and found that IDOX and HMX crystals have similar properties (e.g., a reduced modulus of 23.3 GPa for IDOX versus 25.2 GPa for HMX, and a hardness of 1.0 GPa for both), concluding that IDOX exhibits similar elastic and plastic mechanical behavior to HMX. Energetic materials typically contain approximately 95% explosive and 5% matrix or binder. The particle size distribution is often 50 wt.% fine ($<$75 μm) and 50 wt.% coarse ($>$150 μm). Historically, Estane 5703 polymer has served as the matrix in HMX and PBX composites. The mechanical properties of composite IDOX embedded in Estane have been investigated under quasi-static and high-strain rate conditions (e.g., using a split Hopkinson pressure bar) in [7], [8], [9]. In this study, we investigate the surface deformation of IDOX/Estane composite specimens—each 5 mm in both height and diameter (1:1 aspect ratio)—using a novel technique. First, the specimen is marked with conductive ink (with verification that the ink does not alter its mechanical properties, as confirmed by comparison with unmarked specimens). The marked specimen is then scanned, and stereo DIC is performed under quasi-static loading to study full-field surface transformation and deformation. The same method is applied under SHPB conditions using 2D DIC. This approach aids in aligning the DIC data with the pre-scanned specimen, thereby facilitating post-test segmentation for modeling and experimental verification.

Materials

5'iodo'2 idoxuridine (IDOX) particles of (75-150 μm) were mixed with Estane, the IDOX crystals obtained from Los Alamos National Laboratory (LANL), were recrystallized. The mixture was stirred at 50 °C until fully dissolved and then transferred to a containment vessel for crystallization, The crystallized IDOX was filtered using ultrafine mesh coffee filter paper, and any remaining IDOX was washed off the filter using tetrahydrofuran (THF).

After drying, percussive grinding was used to break the larger crystals into smaller particles. These particles were mixed with Estane resin at a ratio of particulate to matrix of 19:1 by weight. The mixture was placed into a preheated die (90 °C) and compressed under 155 MPa for one minute.

Experiments

Quasi-static Compression and 3D DIC

Quasi-static compression tests were conducted in compliance with ASTM D695 standard using an Instron 5969 universal material testing machine equipped with a 500 N load cell. The experiment was performed under ambient temperature

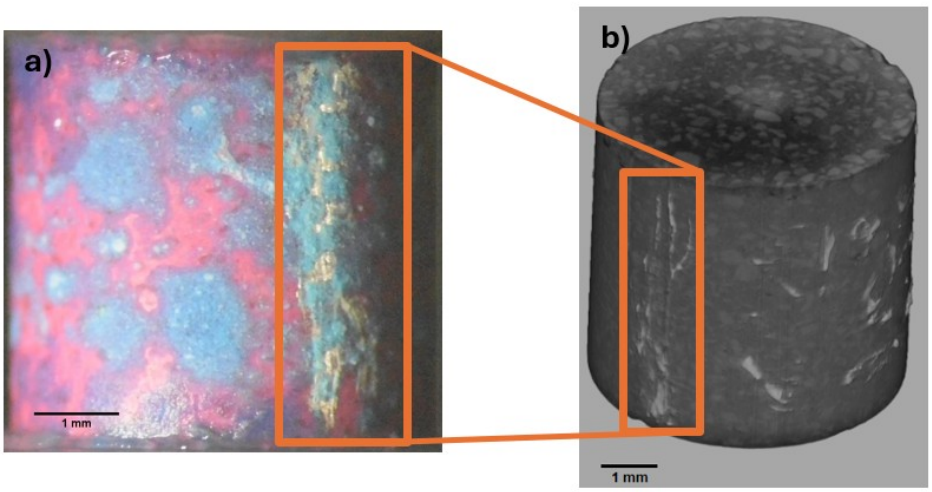

Fig. 1 (a) Representative of stereo DIC set up with a Zeiss microscope and Nikon 7200 cameras, (b) speckled 90 °C press IDOX/Estane with visible inks under X-ray computed tomography

conditions of 23 °C and 44% relative humidity. A Zeiss long working distance stereo microscope (Model 310187) was fitted with a pair of Nikon D7200 digital cameras (at 6000×4000 pixel resolution) each attached with a 105 mm lens, was used to acquire images of the specimen as the specimen deforms. First the specimen was painted with non-reflective water base paint to avoid glares and reflection of LED lights for better correlation results, Petroleum jelly (Vaseline) was applied to both ends of the cylindrical specimen to reduce surface friction as outlined here [10] then the specimen was carefully placed at the center of 2" platen compression and compressed at the rate of 0.5 mm/min. The specimen was placed in a pre-determined orientation and in order to make post processing data easier after 3D DIC analysis to ensure displacement fields match the acquired data from the machine and to match the grains in μCT. Fig. 1 (a) shows a 5 mm in height and diameter with applied speckles and conductive ink and Fig. 1 (b) shows corresponding marked strip visible under X-ray computed tomography (CT) with rendering 4 $\mu m/pixel$.

High strain rate compression 2D DIC

An 80-ft long SHPB was used to perform high strain rate compression on a pre-scanned 5 mm in height and diameter IDOX/Estane composite. A Photron high speed camera capable of achieving 120,000 fps was used with a 105 mm lens. Pulse shaping technique was adapted to achieve dynamic force equilibrium and constat strain rate during high rate of loading. For a striker of 457 mm, the duration of the experiment is 481 μs. In a typical SHPB experience transmitted wave is used to calculate stress, and reflected wave is used to calculate strain rate, as the wave travels in bars sufficient amount of time is required for the specimen to achieve equilibrium state; pulse shaping has proven to be reliable method in achieving equilibrium. In order to acquire material properties in dynamic testing, nearly uniform deformation is required. In a SHPB stress (σ_s) and strain rate ($\dot{\varepsilon_s}$) are calculated using equations (1) and (2), respectively.

$$\sigma_s = \frac{EA}{A_s}\varepsilon_t \tag{1}$$

$$\dot{\varepsilon}_s = \frac{-2c}{l_0}\varepsilon_r \tag{2}$$

where, E, A, and c are Modulus of elasticity, cross-sectional area, and elastic bar wave velocity, respectively and l_0 and A_s are the original length and cross-sectional area of the specimen respectively.

Figure 2 shows pre-scanned marked specimen with conductive inks which are visible to X-ray without changing the geometry of the specimen. During high strain rate experiment, the randomly applied markers were used as speckles on the surface of the specimen to measure surface deformation during SHPB experiment. Overall, two specimens were tested under the same condition at 550 s^{-1} strain rate, with overlapping results.

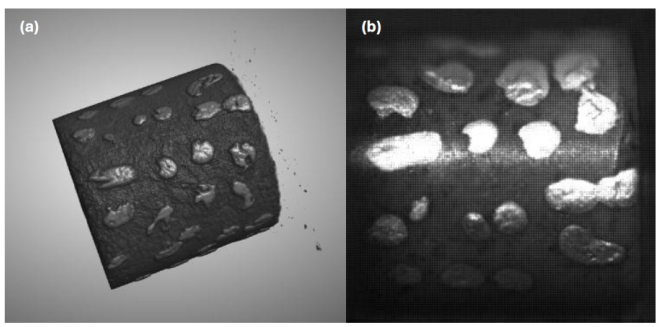

Fig. 2 Corresponding conductive ink markers (a) micro-CT of 5 mm IDOX/Estane specimen (b) same specimen placed in SHPB before impact

Results and Discussion

In this study we conducted quasi-static and high strain rate experiments on 5 mm in height and diameter IDOX/Estane specimens manufactured at 90 °C and used 3D and 2D DIC to acquire surface deformation during quasi-static and high strain rate testing, respectively.

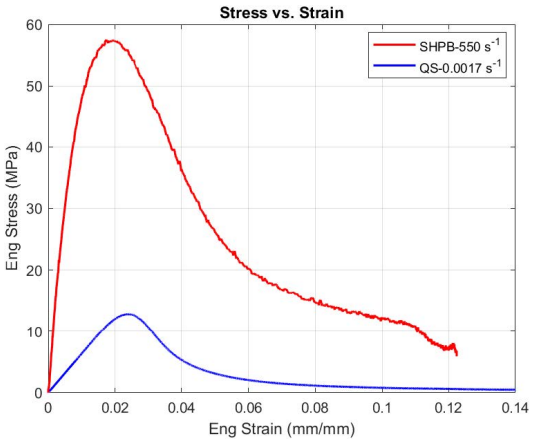

Fig. 3 Representative of stress-strain relationship of 5 mm IDOX/Estane during quasi-static at 0.0017 s-1 strain rate (blue) and stress-strain relationship under uniaxial SHPB experiment at 550 s-1 strain rate (red).

We tested 5 specimens of 5 mm in height and diameter of composite IDOX/Estane to ensure repeatability of the data. Fig. 3 shows stress-strain relationships of both quasi-static and high strain rate experiments of 5 mm specimen at room temperature. Since the margin of error for all the tests during quasi-static and medium strain rate was withing 5%, only one plot of each experiment is presented. As shown in Fig. 3 both specimens reach max stress during low and medium strain rate at almost the same strain, following a linear region and after which reflects the plastic region. It can be seen that the stress-strain curve indicates the behavior of soft materials, there is pre-peak region corresponding to elastic section following immediately by plastic region.

Fig. 4 illustrates 2D DIC acquired during SHPB, since crystal structure of IDOX is triclinic with space group of P1, and since triclinic lattice structure lacks symmetry elements, this makes the composite IDOX/Estane highly anisotropic. The

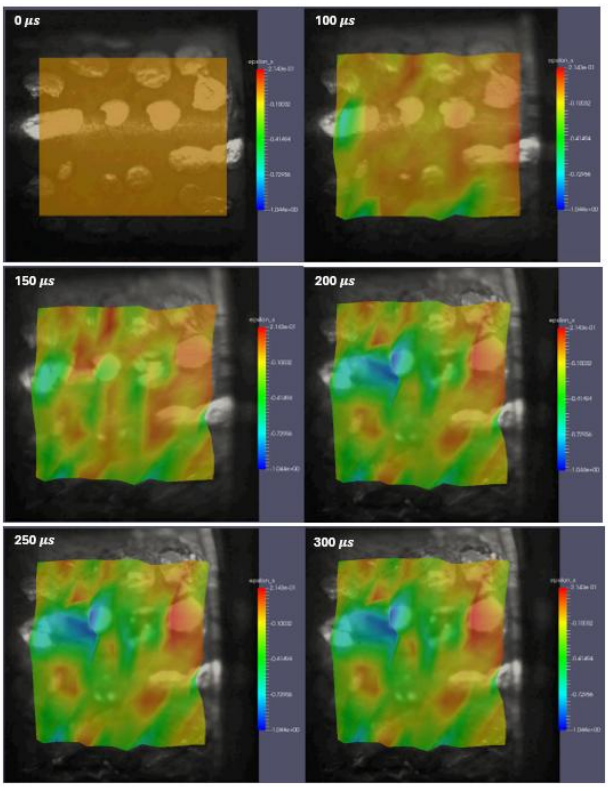

Fig. 4 Illustration of 2D DIC during high strain rate testing on 5 mm specimen

strain patterns are not radially symmetric, concluding the material does not respond identically in all direction, this suggests the composite specimen has different mechanical properties along different orientations. In general, composite materials exhibit anisotropic mechanical properties due to crystal structure. As the specimen deformed uniformly, the distribution of strain in the SHPB test suggests different stiffness and compliance in different regions. A total of two specimens were tested under the same conditions and approximately 550 s^{-1} strain rate, both materials showed similar behavior. This method helps in aligning data obtained during DIC to match the pre-scanned sample, to further assist simulation of mock energetic materials using high performance computers (HPC). The results can be utilized to verify and validate simulations of the mechanical behavior of particulate materials through direct numerical simulation (DNS) and to support the development of a computational multiscale micromorphic continuum mechanics framework.

Acknowledgments We acknowledge the support of the Department of Energy, National Nuclear Security Administration, Predictive Science Academic Alliance Program (PSAAP) under Award Number DE-NA0003962. We also appreciate support from the NSF under Award Numbers 2219347 and 1726435. H. Lu acknowledges the Louis A. Beecherl Jr. Chair for additional support. We are grateful to Dr. Alexandra Burch at Los Alamos National Laboratory for providing the crystal IDOX used in this work.

References

1. D. Eilerman and R. Rudman, "Refinement of pentaerythritol," Acta Crystallogr B Struct Sci, vol. 35, no. 10, pp. 2458–2460, Oct. 1979, doi: 10.1107/S056774087900964X.
2. J. M. Welton and G. J. McCarthy, "X-Ray Powder Data for Acetaminophen (C8 H9 NO2)," Powder Diffr., vol. 3, no. 2, pp. 102–103, Jun. 1988, doi: 10.1017/S0885715600013348.
3. G. M. Brown and H. A. Levy, "Sucrose: Precise Determination of Crystal and Molecular Structure by Neutron Diffraction," Science, vol. 141, no. 3584, pp. 921–923, Sep. 1963, doi: 10.1126/science.141.3584.921.
4. J. D. Yeager, A. L. Higginbotham Duque, M. Shorty, P. R. Bowden, and J. A. Stull, "Development of inert density mock materials for HMX," Journal of Energetic Materials, vol. 36, no. 3, pp. 253–265, Jul. 2018, doi: 10.1080/07370652.2017.1375049.
5. J. D. Yeager et al., "Development of a New Density and Mechanical Mock for HMX".
6. A. Burch, J. Yeager, and D. Bahr, "Nanoindentation of HMX and Idoxuridine to Determine Mechanical Similarity," Crystals, vol. 7, no. 11, p. 335, Nov. 2017, doi: 10.3390/cryst7110335.
7. A. C. Burch, V. K. Euser, B. M. Morrow, C. M. Cady, and J. D. Yeager, "Compressive strength of the plastic-bonded explosive surrogate idoxuridine under quasistatic and dynamic compression," Journal of Composite Materials, vol. 56, no. 26, pp. 3949–3956, Nov. 2022, doi: 10.1177/00219983221125904.
8. C. Liu, C. S. Woznick, S. J. Scott, A. L. Duque, M. J. Herman, and J. D. Yeager, "Brazilian disk compression testing of plastic-bonded idoxuridine mock explosive," Journal of Energetic Materials, vol. 41, no. 3, pp. 319–350, Jul. 2023, doi: 10.1080/07370652.2021.1968073.
9. M. J. Herman, C. S. Woznick, S. J. Scott, J. T. Tisdale, J. D. Yeager, and A. L. Duque, "Composite binder, processing, and particle size effects on mechanical properties of non-hazardous high explosive surrogates," Powder Technology, vol. 391, pp. 442–449, Oct. 2021, doi: 10.1016/j.powtec.2021.06.009.
10. W. Chen and B. Song, Split Hopkinson (Kolsky) Bar: Design, Testing and Applications. in Mechanical Engineering Series. Boston, MA: Springer US, 2011. doi: 10.1007/978-1-4419-7982-7.