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In Situ Characterization of Deformation and Fracture Mechanisms in Bi-Component Agglomerates Using µCT-Based Digital Volume Correlation and Spatial Statistics

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Abstract. Understanding breakage mechanisms of agglomerates is essential for optimizing comminution and handling processes in particulate systems. In this work, the composition-dependent mechanical behavior of cylindrical agglomerates composed of glass and polystyrene was investigated using a combined ex situ and in situ approach. Ex situ compression tests were conducted to determine fracture force, stiffness, and primary fracture energy. The results reveal a non-monotonic, M-shaped dependence of fracture energy on composition, highlighting the complex interplay between particle properties and binder-mediated interactions. To resolve internal deformation mechanisms, in situ µCT experiments were combined with digital volume correlation (DVC), enabling full-field measurement of three-dimensional displacement and strain fields. Spatial strain localization was quantified using Local Indicators of Spatial Association (LISA) based on k-nearest-neighbor statistics. By combining volumetric and deviatoric strain clustering, distinct deformation regimes such as dilatational shear, pure shear, and compaction were identified. The presented methodology establishes a direct link between microscale deformation and macroscopic breakage behavior, providing new insights into agglomerate comminution processes .

1. Introduction

The breakage behavior of agglomerates plays a central role in comminution processes such as milling, granulation, and powder handling. Mechanical stability is governed by complex interactions between particle properties, binder bridges, and microstructural arrangement [1-4]. Conventional testing approaches provide macroscopic quantities such as strength or fracture energy but do not resolve the underlying deformation mechanisms. In contrast, X-ray computed tomography (µCT) combined with digital volume correlation (DVC) enables full-field measurement of internal deformation, offering direct access to strain localization and damage evolution [5].

However, the interpretation of heterogeneous strain fields remains challenging. Spatial statistical approaches such as Local Indicators of Spatial Association (LISA) provide a quantitative framework to identify and classify localized deformation patterns [9,10]. The objective of this work is to link macroscopic breakage behavior with microscale deformation mechanisms in bi-component agglomerates by combining mechanical testing, DVC, and spatial statistical analysis.

2. Materials and Methods

2.1. Agglomerate Preparation

Cylindrical agglomerates were produced from binary mixtures of glass (GL) and polystyrene (PS) particles with varying volumetric compositions (PS100, PS75GL25, PS50GL50, PS25GL75, GL100), enabling a systematic investigation. The primary particles had a characteristic size of approximately 200 µm, allowing particle-resolved imaging in subsequent in situ µCT experiments (cf. Fig.1). Agglomeration was performed using a liquid binder solution (7 w.% hydoxypropylemethylcellulose aqueous solution), with the absolute binder mass kept constant at approximately 15 mg after drying to ensure comparability between compositions and enable the analysis of composition-dependent breakage behavior.

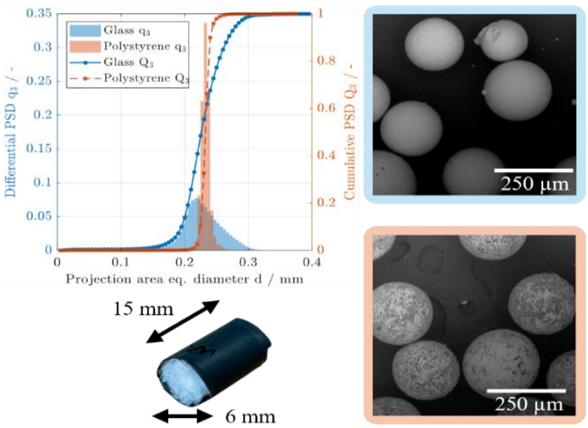

Figure 1. Particle size distribution (left) and SEM images (right) of the used primary particles with representation of an agglomerate in poly lactic acid (PLA) cover.

Particles were homogeneously mixed, followed by binder addition and compaction into cylindrical casings (d = 6 mm, L = 15 mm). After drying, solid binder bridges formed and the final binder mas was determined gravimetrically, similar to our previous work [1]. This procedure yields reproducible agglomerates with consistent binder content, enabling the isolated study of composition-dependent breakage behaivor.

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2.2. In Situ µCT and Digital Volume Correlation

In situ compression experiments were performed using X-ray computed tomography (µCT) to capture the internal structure of the agglomerates at successive load steps (cf. Fig.2). A reference scan was acquired at an initial load level (5 N), followed by additional scans at increasing load increments (10 N). This enabled the tracking of internal deformation throughout the loading process (cf. Fig.3).

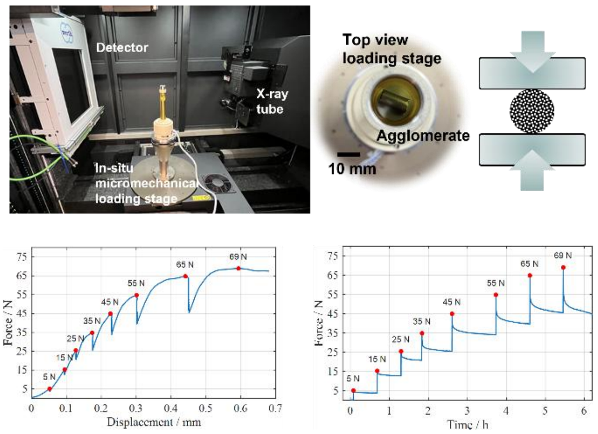

Figure 2. Experimental setup for the µCT in situ compression tests (top) and representative force displacement and force time curves of the stepwise compression procedure.

Three-dimensional displacement fields were computed using digital volume correlation (DVC), which is based on the correlation of grayscale patterns between the reference image I0(x) and the deformed image I1(x) [5-8].

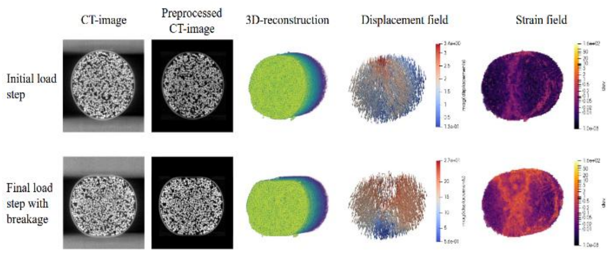

Figure 3. Representation of the workflow from grayscale image stack to the three dimensional displacement and strain fields.

An initial estimate of the displacement field u(x) was obtained by maximizing the cross-correlation function. The displacement field was subsequently refined using an iterative optimization approach by minimizing the gray-value residual.

$$C\Phi(u) = \sum[I_0(x) - I_1(x + u(x))]^2 \qquad (1)$$

Based on the resulting displacement field, the deformation gradient tensor F was computed as

$$\boldsymbol{F} = I + \nabla u \qquad (2)$$

from which the Green–Lagrange strain tensor E was derived

$$\boldsymbol{E} = \frac{1}{2}(\boldsymbol{F^T F} - I). \qquad (3)$$

From E, both volumetric ε_{vol} and deviatoric ε_{dev} strain measures were calculated, forming the basis for the subsequent analysis of deformation and breakage mechanisms.

2.3. Spatial Statistical Analysis

To quantify spatial strain localization, a Local Indicators of Spatial Association (LISA) approach was applied to the normalized strain fields. The analysis is based on Local Moran's I, which evaluates the degree of spatial autocorrelation of a variable relative to its neighborhood [10].

$$I_i = z_i \sum_j w_{ij} z_j \qquad (4)$$

Here, z_i denotes the standardized strain value at location i, and w_{ij}represents binary spatial weights defining the local neighborhood based on a k-nearest-neighbor (kNN) approach. In this study, the neighborhood includes the reference point and its ten nearest neighbors ($k = 10 + 1$). Positive values of I_i indicate spatial clustering of similar strain magnitudes (high–high or low–low), while negative values identify spatial outliers. Based on this classification, regions of strain localization can be distinguished from mechanically inactive zones.

This statistical framework enables a quantitative and spatially resolved identification of deformation patterns and provides the basis for linking strain localization to underlying breakage mechanisms [7,10].

3. Results and Discussion

The in situ µCT–DVC analysis provides direct insight into the internal deformation processes governing breakage in agglomerates under compressive loading. The computed displacement and strain fields reveal highly heterogeneous deformation patterns that evolve significantly with increasing load (cf. Fig.3). Spatial statistical analysis based on Local Indicators of Spatial Association (LISA) enables a systematic quantification of strain localization within the agglomerates. The LISA plot matrices of the deviatoric (Fig.5) and volumetric (Fig.4) strain fields show pronounced composition-dependent differences in both the onset and spatial organization of high-strain clusters.

For mixed compositions (PS75GL25 and PS25GL75), high–high (HH) clusters emerge at comparatively low load levels and progressively localize into distinct regions that evolve into well-defined fracture planes. This behavior indicates a gradual accumulation of damage driven by localized deformation. In contrast, GL100 and PS50GL50 agglomerates exhibit a delayed formation of HH clusters, which remain more spatially diffuse until shortly before failure. This suggests a more abrupt breakage behavior with limited precursory strain localization (cf. Fig.4 and 5).

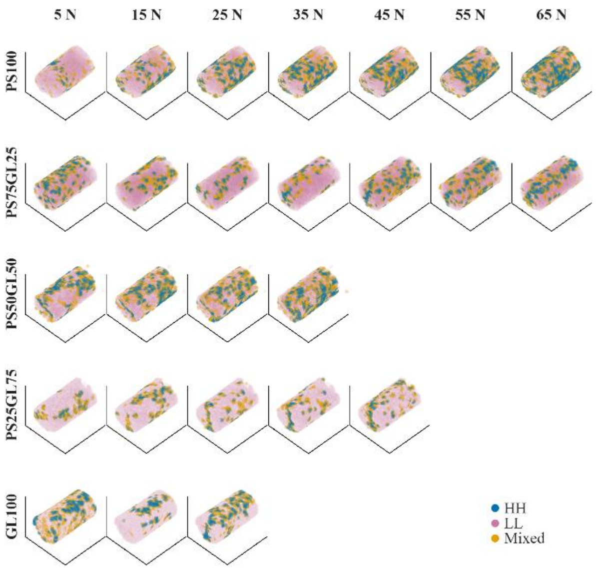

Figure 4. Local Indicators of Spatial Association (LISA) of the normalized volumetric response ε_{vol} for all compositions and load steps.

A fundamentally different deformation pattern is observed for PS100 agglomerates. Here, HH clusters appear early but remain spatially distributed throughout the entire volume, indicating a more homogeneous deformation process rather than localized fracture.

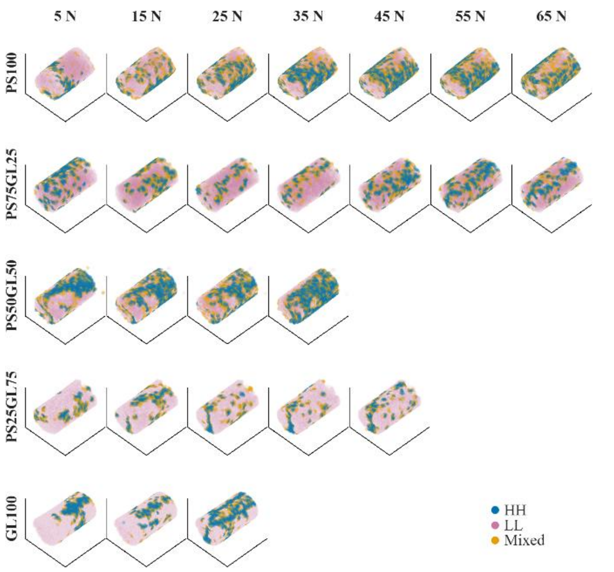

Figure 5. Local Indicators of Spatial Association (LISA) of the normalized deviatoric response ε_{dev} for all compositions and load steps .

This behavior reflects the more compliant nature of the polymer-based system and suggests a transition from fracture-dominated to deformation-dominated response. While the LISA plot matrices provide spatial insight into strain localization, the stacked bar and line plots enable a quantitative assessment of cluster evolution. Across compositions, the fraction of HH clusters increases with load, reflecting the progressive development of localized deformation. However, the rate and extent of this increase differ significantly, highlighting the influence of composition on damage evolution (cf. Fig.4 and 5).

The combined evaluation of volumetric and deviatoric strain fields allows the identification of distinct deformation mechanisms. Regions characterized by high deviatoric strain and positive volumetric strain are associated with dilatational shear, indicating particle separation and crack initiation (cf. Fig.6). In contrast, negative volumetric strain indicates compaction-driven deformation, corresponding to local densification and load redistribution.

The breakage mechanism plot matrix reveals that mixed agglomerates exhibit a strong contribution of dilatational shear localized along emerging fracture planes, whereas glass-rich systems show a higher contribution of compaction prior to failure. Polystyrene-dominated systems are characterized by distributed deformation with less pronounced localization.

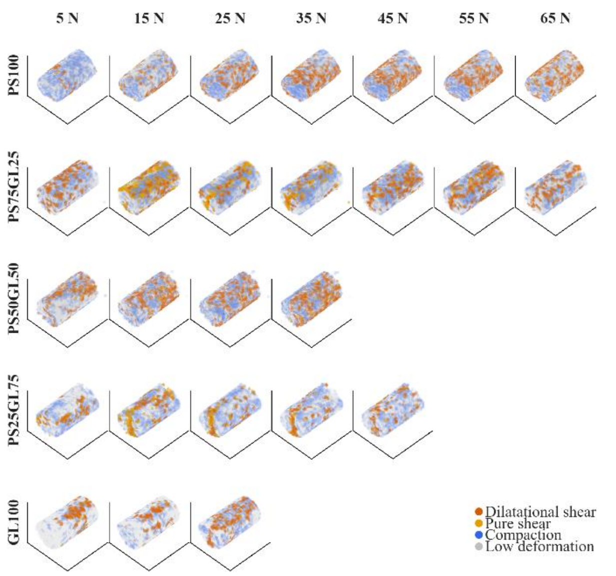

Figure 6. Spatial plot matrix of strain-based breakage mechanisms across all agglomerate compositions and load steps.

Overall, the results demonstrate that breakage in agglomerates is governed by the spatial organization and interaction of different deformation mechanisms rather than by a single dominant failure mode. The combination of in situ μCT, DVC, and spatial statistical analysis provides a powerful framework to resolve these mechanisms and to establish a direct link between micro-scale deformation processes and macroscopic breakage behavior in particulate systems.

4. Conclusions

This study demonstrates that the combination of in situ μCT, digital volume correlation, and spatial statistical analysis provides significant potential to enhance the information content obtained from tomographic experiments and to resolve breakage processes at the microscale. By enabling the spatially resolved quantification of internal deformation, the approach allows detailed insights into the mechanisms governing fracture initiation and propagation within agglomerates.

The application of LISA to volumetric and deviatoric strain fields enables a quantitative identification of strain localization and its progression under load. By combining both strain measures, distinct deformation regimes such as dilatational shear, compaction, and distributed deformation can be identified and linked directly to breakage processes.

The results reveal strong composition-dependent differences in deformation behavior. Mixed agglomerates exhibit early strain localization and the formation of well-defined fracture planes, whereas glass-rich systems show delayed localization and more brittle behavior. Polystyrene-dominated systems, in contrast, are characterized by more homogeneous deformation.

Overall, the presented approach provides a mechanistic understanding of agglomerate breakage by linking microscale deformation patterns to macroscopic failure behavior. This framework offers significant potential for improving predictive models and optimizing comminution processes in particulate systems.

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