Influence of microstructure on slag breakage behavior

Vo Thu Trang, Peuker Urs A.[1]

[1]Technische Universität Bergakademie Freiberg, Institute of Mechanical Process Engineering and Mineral Processing, Agricolastr. 1-09599 Freiberg

Abstract. This study investigates deformation, fracture propagation, and liberation mechanisms in lithium aluminate-bearing slags with varying cooling histories and resulting microstructures. In-situ compression experiments were conducted using X-ray computed tomography (XCT) combined with digital volume correlation (DVC) to quantify local deformation, fracture development and strain evolution during loading. Image-based analysis was used to quantify the relationship between fracture behaviour to microstructure, pore network and target phase features and liberation behavior and provides a framework to link the design engineered artificial minerals with improved comminution.

1. Introduction

Understanding the breakage and liberation behaviour of slags improves their recyclability and ensures that slag can serve as a secondary raw material for low concentrated valuable elements.
These elements are transferred to engineered artificial minerals (also called EnAM). To liberate the valuable mineral phases, comminution is needed. Besides generating smaller particles out of the parent particle and new surfaces, the valuable mineral phases (= target phases) become accessible for downstream separation processes. Since milling and crushing are cost-intense processes in mineral processing, optimization of breakage behavior and liberation is needed to increase process efficiency.
The relationship between microstructure, fracture behaviour, and liberation can be described through fracture mechanisms. Early liberation theory is introduced by Gaudin to distinguish between liberation by detachment and liberation by comminution [1]. Liberation by detachment describes fracture along grain boundaries or interfaces where attachment forces between adjacent phases are weaker and result into selective fracturing. In contrast, liberation by comminution describes fracture propagation through mineral phases independent of their interfaces, where liberation occurs mainly through particle size reduction and surface generation. In practice, fracture behaviour usually lies between these boundary cases and is influenced by the local microstructure.
Microstructural features, e.g. grain size, phase intergrowth, pores, defects and crystallographic interfaces, influence local stress distributions and therefore can determine preferential fracture pathways [2]. Consequently, fracture propagation in heterogeneous materials is not random by default. Preferential breakage occurs when fractures interact more frequently with specific phases, interfaces, or defects which lead to selective fragmentation of certain microstructural domains [3]. Such behaviour is relevant for engineered artificial minerals, where cooling and crystallization conditions determine target phase properties and therefore may affect comminution and liberation results [4].
This study investigates deformation and breakage mechanisms in lithium-bearing slag, a product of lithium extraction out of pyrometallurgical processes. In-situ X-ray computed tomography (XCT) compression and impact experiments are combined with digital volume correlation (DVC) to quantify internal strain and localize deformation during compression. The measured strain fields is then linked to the initial microstructure and mechanical properties of the slag sample.

2. Materials and methods

2.1. Materials

Synthetic lithium aluminate-bearing slags are produced at IME Process Metallurgy and Metal Recycling, RWTH Aachen. The slags vary in terms of cooling conditions and feed compositions which lead to different microstructures of lithium aluminate as a target phase.

Cylindrical samples were prepared to ensure reproducible sample geometry and stable positioning within the in-situ load cell during compression (Figure 1). The cylindrical geometry minimizes movement of the parent slag particle and provides more defined contact conditions between the sample and the compression pistons.

* Corresponding author: thu-trang.vo@mvtat.tu-freiberg.de

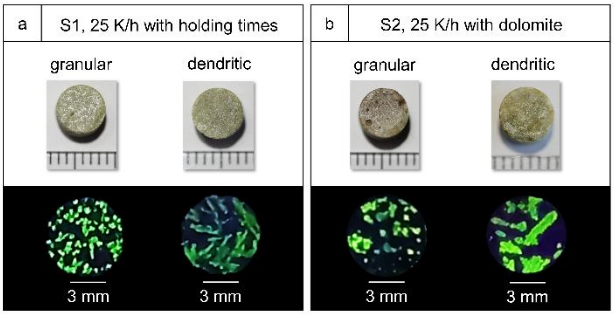

Figure 1. Cylindrical shaped slag samples, labelled as granular and dendritic under visible light and UV light conditions. The green fluorescent mineral phase is lithium aluminate (target phase). Slags come in different slag batches under different cooling strategies, e.g. a) 25 K/h with holding times, b) 25 K/h without holding times with dolomite.

2.2. Methods

2.2.1 Compression experiments

To compress slag samples, the CT5000 in-situ load cell (5 kN) from Deben UK Ltd. Was used and coupled with a Zeiss Xradia 510 Versa XCT system. Samples are positioned between two parallel silicon nitride ceramic pistons and compressed while force-displacement data and XCT projections were monitored simultaneously. The displacement rate is 0.1 mm/s and samples had a preload force of 5 N.

XCT settings are limited by the size of the load cell and attenuation coefficient of the ceramic, carbon-fiber window of the loadcell and the sample and lead to a voxel size of 8 µm.

2.2.2 Digital volume correlation

Dynamic and high-resolution scans from compression experiments on slag samples were acquired with CoreTOM in collaboration with UGCT, Ghent University – Centre for X-ray Tomography. Together with UGCT, a 4D image analysis workflow is developed and applied. The data is mainly processed using digital volume correlation (DVC) in Avizo. Image registration with respect to the initial scans prior to compression will be carried out to correct bulk motion and misalignment. Subsequently, dynamic scans will be analysed using DVC to derive displacement vectors and strain fields. The resulting displacement vectors will then be used to estimate the overall strain in the high-resolution scans.

3. Results and discussion

This study will provide quantitative insights into breakage mechanisms of lithium slag, including pore collapse, fragment rearrangement and fracture propagation. It introduces a reproducible workflow for large-scale 4D XCT analysis and generate HQ datasets for open access data management.

Recent findings are that lithium aluminate phase exhibit pre-damages prior to compression. In collaboration with Institute of Stochastics in Ulm University, three samples were virtually reassembled in three dimensions to investigate breakage mechanism quantitatively. It was shown that existing target phases can influence the pathway of fractures, i.e. target phases may act as defects that connect multiple fractures. With the increase of pore number and size, the pore network will dominate the direction of the fractures instead of the localization of target phases. In that case, fractures occur mainly between pores and lead to breakage of solid phase bridges.

During compression, the target phases act as defects, can influence the trajectory of fractures and tend to break into finer fragments than the matrix phase. Compact (granular) microstructures support preferential breakage, i.e. fracturing within a specific mineral phase such as lithium aluminate. Dendritic target phases tend to show more random fractures (Figure 2).

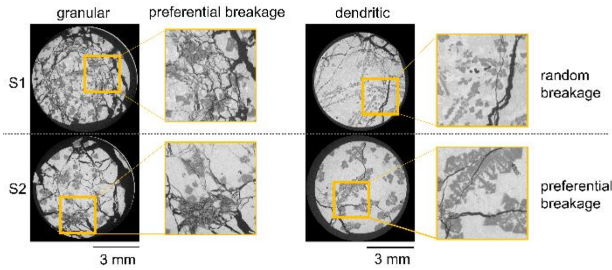

Figure 2. Qualitative image-based assumption of fracture mechanisms for different slag batches and microstructures.

Surface liberation increases with ongoing compression for all investigated slag types and microstructures. However, the increase is not proportional to the occurrence of fracture events. Small force drops usually lead to cracks on the periphery of the slag sample or fragments are generated without exposing the target phase substantially. Therefore, fracture initiation alone will not be sufficient enough to achieve effective liberation, although lithium aluminate may have shown features for more selective fracturing, e.g. containing pre-damages and producible in various microstructures.

Granular-labelled slag samples have shown to generate larger number of fine target phase domains with high surface liberation values. This indicates that fragmentation frequently intersected with lithium aluminate domains and lead to new accessible target phase surfaces. In contrast, target phase domains remain interconnected even after fracture beyond fracture events in dendritic samples and have shown lower overall surface liberation.

The image-based approach is able to approximate liberation behavior by means of geometric descriptors derived from segmented XCT data. Descriptors are able to quantify boundaries, fragmentation and connectivity and have enabled to put microstructure and fracture into relation to liberation. These image-based proxies are therefore suitable to estimate liberation trends between different microstructures.

However, image-based liberation descriptors are only an approximation of physical liberation. Surface liberation does not directly mean that there is a complete detachment because fine target phases can still be interlocked in the fragments despite appearing exposed in segmented data. This stems from segmentation uncertainty, voxel

resolution and partial volume effects that may influence the quantification of interfaces and small fragments.
Therefore, image-based descriptors should be seen as proxies for liberation potential than direct measurements of the process recovery.

4. Conclusion

This study presents the combination of in-situ X-ray computed tomography with digital volume correlation to locate deformation, fracture propagation, and breakage mechanisms in lithium-bearing slags. The workflow enables three-dimensional analysis of displacement and strain evolution during compression and links local deformation behavior and the slag microstructure.

Overall, the results show that breakage behavior in slags can not be explained only by mechanical loading conditions but must be interpreted in combination of microstructure, defect distribution, and pore architecture. The presented methodology provides a framework for connecting microstructural characterization with comminution behavior and can support the design of engineered artificial minerals with improved liberation and improved recyclability in downstream processing approaches.

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