C12: Breakage mechanism in heterogenous structures – combining microstructure of EnAM and breakage / liberation behavior

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Overview

As part of the PP2315, the subproject C12 investigates the breakage and liberation of slags containing the EnAMs. To understand the behaviour of the comminution process of slags, a detailed microstructure analysis is necessary to differentiate between the amorphous phases and the crystallized and structured phases. Those phases can be subdivided even further into different solid components since the EnAM and the carrier matrix should have different chemical properties, durability and plasticity.

After the selection of single particles, a compression force will be exerted onto the sample. The deformation and crack formation will be monitored and observed by mounting the sample onto a load cell and conducting XRM measurements with each specific force step. Meso- and microstructural changes can be related to the load information, then we can assign to each initial structural property a resulting breakage mechanism. The latter describes a mechanism which causes mineral liberation and can be classified in liberation by comminution and by detachment by Gaudin. A fracture which propagates along grain boundaries is defined as an intergranular fracture and can be equally seen as the liberation by detachment. Fractures which are not depending on the texture of the minerals, grain boundaries and mineral properties are called transgranular (random) fractures. Since both are borderline cases, the real comminution process of chemical, mineralogical and structural heterogenous slag particles will show the superposition of both fracture types.

In-situ compression with a load cell and XRM measurements

The in-situ compression is divided in three parts (Figure 1): At the initial state, the single particle consisting of EnAM grains and its carrier matrix rests in a stable position between a moveable piston and the fixed punch. The sample is not exposed to any force yet and, therefore, does not show any deformation. This step provides the XRM data to analyse and describe the inner structure and phase distribution of different minerals in the unbroken particle.

By increasing the height of the piston, a force is exerted onto the single particle. The amount of force applied should be between 0 and the breakage force at which the particle is completely broken. The first compression scan is carried out with XRM where the first cracks and deformation effects can be observed in the particle. The second compression scan is made after the breaking event. In that state, the force must be higher than the breakage force and now the particle divided in fragments of different sizes and shapes.

The next step is the 3D-rebuilding process of the initial particle sample in cooperation with Z3. This process needs all three states of the compression experiment to relocate the fragments into their initial position before the breakage arose. With the help of the virtual assembled particle, the breakage plains and local deformation effects can be identified and correlated with the microstructure analysis from the first step.

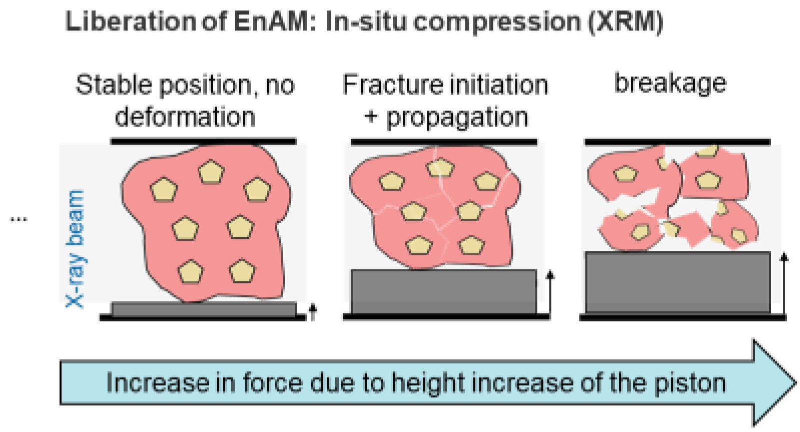

Figure 1: In-situ compression of a single particle with a load cell and XRM measurements

Microstructural requirements

For the better understanding of the fracture propagation, different batches of of slag generation (Z1) will used as the material to create single particles. Those particles differ in their structure which will be selected depending on their EnAM particle size and concentration, e.g. different concentration of EnAM with constant grain sizes and constant concentration of EnAM with three different grain sizes (Z1 with different cooling rates). Another requirement is a describable shape of the EnAM grains since the segmentation of those will be based on their structural and morphological properties.

Characterization of breakage plains by AFM measurements

The second compression state provides the debris particles. For the characterization of the surface morphology of breakage plains, samples need to be prepared to quantify local surface roughness and morphology and to detect the individual EnAM phases in the breakage plane. The measurements will be used for the repositioning of two planes to determine the breakage mechanism while investigating the microscopic geometry of the rupture. Both planes have to be aligned virtually by key-lock-principle. Then the distance between each plane can be determined so that is possible to detect deformations and whether the corresponding phases are the same (transgranular) or not (intergranular).

References

Gaudin, A. M. "Principles of mineral dressing" *The Journal of Physical Chemistry*. April 1940, 44, 4, 532–533.