# In situ diffraction: Elucidation of mechanochemical reactions of inorganic materials in a ball mill

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This contribution discusses the application of mechanochemistry to the synthesis and conversion of inorganic materials by combining mechanical energy with chemical reactions. Many synthesis reactions in ball mills do not require solvents, which makes them ecologically advantageous. In addition, mechanochemistry can lead to metastable products with interesting properties that would not be accessible by conventional synthesis methods.

The use of in situ methods, in particular X-ray diffraction (XRD), allows structural changes during the formation process to be analysed in real time. The synthesis of metal-organic frameworks was followed by the first implementation of a shaker mill on a diffractometer at ESRF.[1] However, while the synthesis of soft matter can be carried out in X-ray transparent polymer milling jars, the synthesis of many inorganic materials requires harsh milling conditions in steel jars.[2] The conversion of boehmite (AlOOH) to high surface area corundum ($Al_2O_3$) is an example of the sensitivity of mechanochemical reactions to the appropriate energy input to a particular reaction. It was only by gradually adapting the design of the beakers and modifying the mill that it became possible for the first time to observe the transformation in situ in a ball mill.

Analysis of the forming crystal structures and their microstructural properties provides insight into the formation mechanisms, reaction kinetics, and structure-property relationships of the resulting materials. Not only can synthesis be carried out in a ball mill, but catalytic reactions can also be performed without the need for external temperature or pressure. In addition, mechanochemistry can make it unnecessary to optimise the catalysts. Structural changes of catalysts can be monitored during a catalytic reaction by operando XRD experiments in combination with gas analysis.

This paper presents the technical development of ball mills for solid-gas catalytic reactions and examples of catalyst synthesis. In addition, the mechanocatalytic oxidation of CO over Au@$Fe_2O_3$ catalysts will be discussed [3], illustrating the versatility and potential of mechanochemistry in advancing catalysis and materials science.

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[3] Petersen, H.; de Bellis, J.; Leiting, S.; Das, S. M.; Schmidt, W.; Schüth, F.; Weidenthaler, *C. ChemCatChem.* (2023) **14(19)**: e202200703.

*The author would like to thank Dr. Hilke Petersen, Tobias Rathmann, Dr. Wolfgang Schmidt, and Jan Ternieden (all MPI Kohlenforschung for their contributions and support. DESY (Hamburg, Germany), a member of the Helmholtz Association HGF, is acknowledged for the provision of experimental facilities. Parts of this research were carried out at PETRA III and I would like to thank Dr. Martin Etter for assistance in using P02.1.*