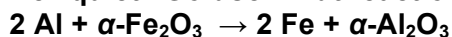


## The “quiet” Goldschmidt reaction



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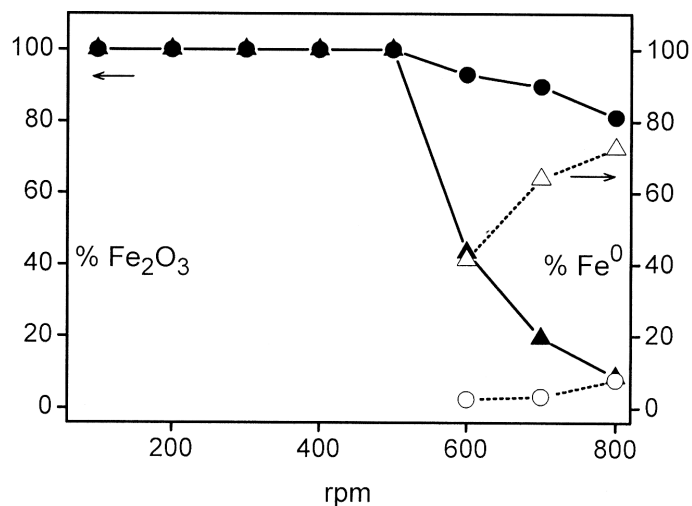
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The well-known *Goldschmidt* reaction, *i.e.* the reaction of (2 Al +  $\alpha\text{-Fe}_2\text{O}_3$ ), was investigated upon mechanochemical activation by employing Mössbauer and ESR spectroscopy in combination with thermal analysis (TA), magnetic susceptibility measurements and X-ray powder diffraction (XRD). Quite unexpectedly, both the mechanical treatment and the TA runs under air caused the reaction conditions of “quiet redox reactions” due to a retarding effect thus enabling a reaction study in a “slow motion mode”. This allowed the establishment of distinct partial steps of the integral reaction process depending on the intensity of the mechanical impact [1]. The mechanical impact was varied by different milling times, rotation frequencies (rpm), and beaker materials ( $\text{Si}_3\text{N}_4$  or agate).

The results obtained by the various analytical methods revealed a multitude of information about the reaction process, whereby the very early stages of the reaction could be characterized primarily by ESR and magnetic data, intermediates and final products, however, preferentially by XRD and Mössbauer data.

It could be shown that the fraction of  $\alpha\text{-Fe}_2\text{O}_3$  is much stronger decreased by milling in  $\text{Si}_3\text{N}_4$  and rotation frequencies of  $\geq 500$  rpm than by milling in agate beakers (Figure 1). Correspondingly, the part of  $\text{Fe}^0$  increased remarkably for rotation frequencies of  $\geq 500$  rpm. This increase is stronger by milling in  $\text{Si}_3\text{N}_4$  beakers than by milling in agate beakers.



**Figure 1** Evolution of the percentage of educt ( $\alpha\text{-Fe}_2\text{O}_3$ ) and product ( $\text{Fe}^0$ ) phases for the mechanochemically performed *Goldschmidt* reaction in  $\text{Si}_3\text{N}_4$  ( $\Delta$ ) or agate ( $\circ$ ) milling beakers as a function of the applied rotation frequency (rpm)

The results directly concern chemical systems with both general and fundamental interest as they belong to the field of self- or autoxidation for which the term flame-less combustion is accepted as well. They also can be regarded as model systems for high-temperature solid state syntheses proceeding as redox reactions. The combination of the employed analytical methods revealed to be helpful for investigating both the “quiet” and the vigorous *Goldschmidt* reaction. The obtained results are compatible and, partly, complementary and deliver an acceptable picture of the partial steps of the *Goldschmidt* reaction.

[1] R. Stößer, M. Feist, C. Willgeroth, F. Emmerling, M. Menzel, H. Reuther, *J. Solid State Chem.* **202** (2013) 173-90.