High pressure milling vial
Gas-Temperature-Monitoring System

- Novel method for the synthesis of nanoscale functional materials
- Insight in physical and chemical processes during milling
- Controlled one-step mechano-chemical synthesis
- In-situ monitoring of hydride, nitride or carbide formation
- Quick check of catalyst efficiency

evicomagnetics

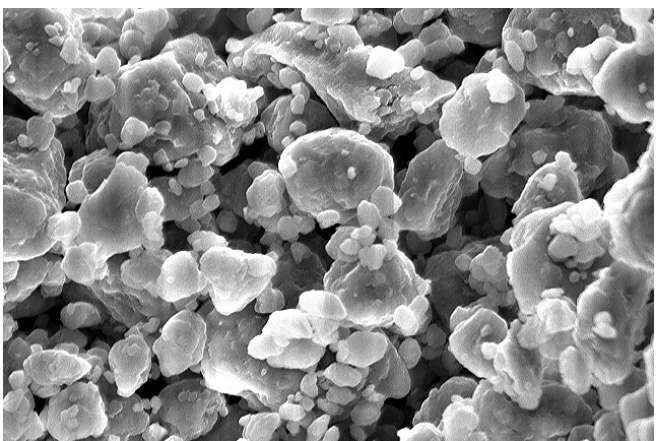
Field of Application

A new and powerful method for the mechano-chemical one-step synthesis of nanoscale functional materials has been developed.

It involves high-energy ball milling in an especially designed vial, allowing in-situ monitoring of temperature and of pressure (maximum operating pressure 150 bar (15.000 kPa) by incorporating a newly developed pressure-temperature measurement system as well as a radio emitter into the vial lid. An external receiver transmits the data to a data acquisition computer.

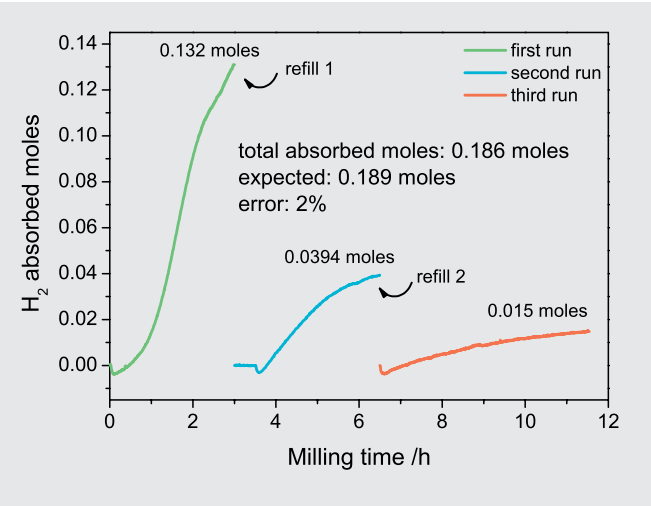
The system gives insight in physical and chemical processes during high-energy ball milling of functional materials such as hydrides, intermetallic compounds, ceramics, bulk metallic glasses etc. These processes can involve mixing, activating, (hydrogen-induced) amorphisation, (hydrogen-induced) phase changes and phase conversions.
It allows a quick check of catalytic efficiency during synthesis of functional materials. It permits accelerated hydride, nitride and carbide formation and nanocrystallinity, thus shorter processing times. Furthermore it allows milling processes to be monitored and optimised (rate of data acquisition can be adapted to the respective process, e.g. slow amorphisation or sudden phase changes).
The synthesis of magnetic and hydrogen storage materials and monitoring of hydrogenation reactions by reactive ball milling under high hydrogen atmosphere are the primary fields of application. Other material classes such as bulk metallic glasses and ceramics (TiN) are also of interest.

The system can be used on any planetary mill of adequate geometry where batch quantities of a material need to be milled in an enclosed container.



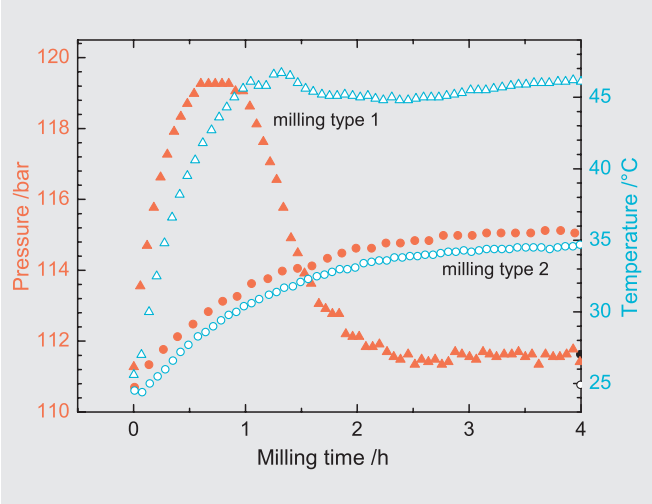
The influence of the milling parameters such as revolutionary speed, milling ball-to-powder ratio and milling time (to name only a few) can be investigated without a time-consuming series of interruptive experiments.
By measuring the milling vial temperature, an integral number for the process parameter temperature is enabled. It reflects the effects of friction, impact and conversion processes. Changes in the reaction are also recorded by the continual and highly sensitive measurement of the gas pressure within the milling vial. With this the interaction of the used gas with freshly created surfaces during milling (adsorption, absorption, desorption and decomposition) can be monitored.

Example 1: Hydrogenation and nanocrystallisation of pure Mg using reactive ball milling.

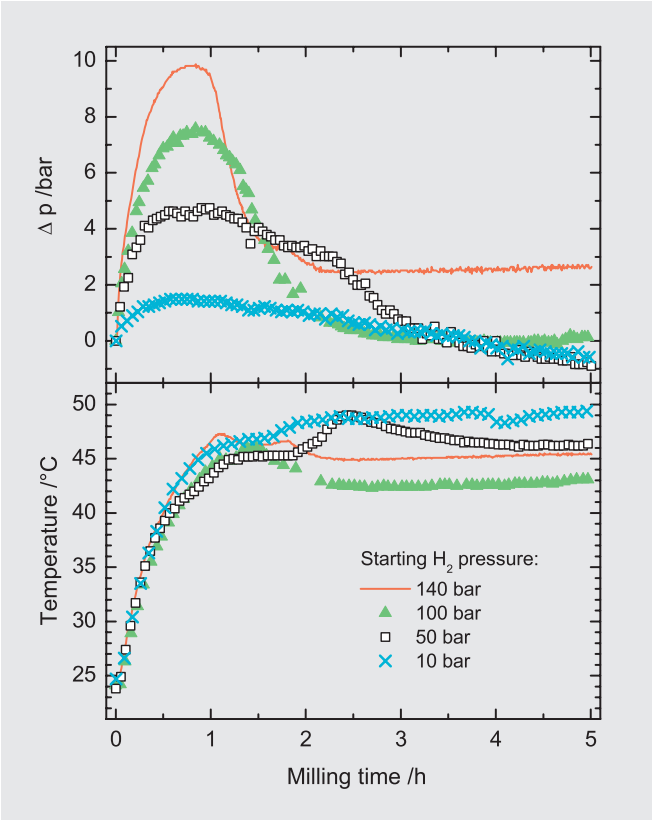


Absorbed moles of hydrogen vs milling time during reactive milling of Mg in 90 bar of hydrogen.

Example 2: One-step mechano-chemical synthesis of $NaAlH_4$, a prototypical high-density complex hydride, using reactive ball milling.

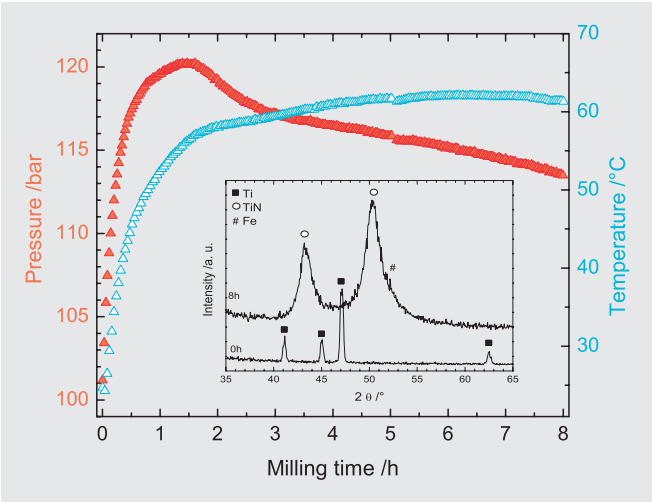


Evolution of the hydrogen pressure (red symbols) and the temperature (blue symbols) inside the vial during the milling of NaH + Al + 4 mol% $TiCl_3$ using a high (milling type 1) and low (milling type 2) energy input. Varied is the number of milling balls and the revolutionary speed. Using the high energy input the synthesis is completed after only two hours.



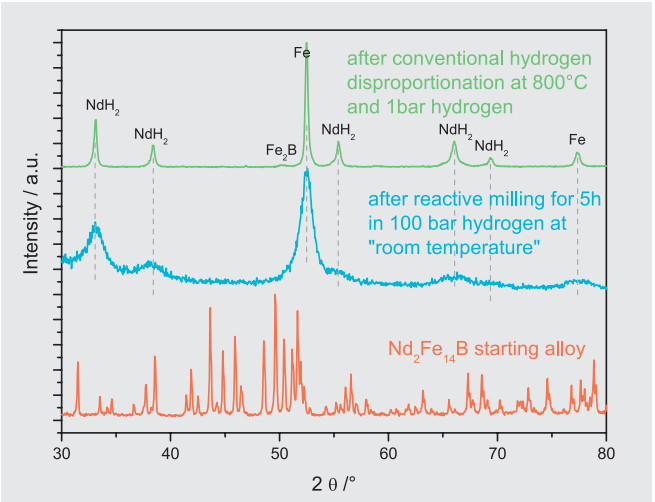
Evolution of (top) the hydrogen pressure variation (Δp) and (bottom) the temperature during milling of NaH + Al + 4 mol% $TiCl_3$ using different starting hydrogen pressures. Using low hydrogen pressure, the progress of the reactions is sluggish.

Example 3: Formation of titanium nitride using reactive milling.



Evolution of the nitrogen pressure (red symbols) and the temperature (blue symbols) inside the vial during the milling of Ti using a nitrogen starting pressure of 100 bar. The inset shows the XRD pattern before and after 8 h milling indicating the full transformation into TiN.

Example 4: Hydrogen-induced disproportionation of $Nd_2Fe_{14}B$ using reactive milling.



XRD patterns of $Nd_2Fe_{14}B$ starting alloy (bottom), after 5 h milling in 100 bar hydrogen (middle) and after conventional hydrogen disproportionation (top): for both reactions, the alloy is fully transformed into NdH_2, Fe and Fe_2B; the former, however, yielding a much refined disproportionated mixture.

Features

- Continuous **in-situ** monitoring of pressure and temperature during ball milling
- p_{max}= 150 bar of hydrogen (smaller pressure ranges with higher resolution available: up to 1-2-5-10-20-50-100 bar)
- Operation time up to 40 h
- Resolution of data acquisition 40 points/s
- Radio range up to 40 m
- Powerful dedicated software
- USB interface
- Volume of milling vial: 220 ml
- Vial material: hardened steel
- Swagelok ball valve

Options

- Two channel system for simultaneous use of two milling vials

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