

Reactive milling is a process conducted with the use of usually high energy planetary ball mills. The aim of the process is not only grinding and fragmentation of the input material but also conducting **its reaction** with the **gas filling the pressure vial** in which the milling is taking place.

This technique, very common in materials engineering, sometimes called a “brutal chemistry method,” allows conducting chemical reactions **under conditions far from the equilibrium** (usually under much lower temperature and pressure). Very often, binary, ternary and complex hydrides - which are considered as **solid state hydrogen storage materials** are synthesized this way. As a model example, we can bring up magnesium hydride synthesis that normally requires $>450^{\circ}\text{C}$ and several tens of bar of hydrogen pressure while it can be successfully formed during reactive ball milling at room temperature and low hydrogen pressure. What more, as a result of the reaction, a polymorph of magnesium hydride is formed that requires gigapascals of hydrogen pressure if synthesized under “static” conditions. Reactive milling processes are unfortunately **long-lasting**, and therefore quite susceptible to contamination with the material of the cylinder and grinding balls. Many attempts were taken by scientists to synthesize binary and ternary hydrides but also borohydrides, alanates, amides, imides and other hydrogen-rich compounds. Oxides, nitrides and other chemical compounds syntheses are also possible and are within the interest of the researchers all over the world..

This process, known for many years is very simple in application and very effective at the same time. For that reason it very popular and successfully used in hundreds of laboratories all over the world. Usually, the process of reactive milling is carried out in such a way that **periods of intensive grinding are interrupted with a pause - to cool the cylinder (!)**. In cases of massive cylinders or efficient cooling, the process can be carried out without interruption.

The applicant states **that the process of mechanochemical synthesis should be the more effective, the higher the temperature** of such a cylinder and the batch itself is since this is a diffusion driven process and it depends to a large extent on the temperature. Also, in such a process, thermally activated processes (**recovery and recrystallization**) should occur, and therefore for some materials at a high temperature, **no strain hardening** of the material occurs. Also, increasing the temperature of the reaction may cause taking it to the processing window in which only existence of the selected products is possible (since other will decompose due to the higher temperature than the equilibrium one under selected pressure). In the case of grinding at room temperature, such reaction tuning is practically impossible. **This approach is revolutionary** and will allow, for the first time, the preparation of single-phase pure products of some reactions (e.g. the formation of a hydride complex Mg_2FeH_6).

The temperature at which synthesis of most (interesting for researchers) metal hydrides can be accelerated is, unfortunately, **higher than the maximum operating temperature of most popular structural plastics** (for example used for manufacturing seals and mounting elements) and electronic components - which can be used to control the process and transmit wirelessly the temperature and pressure data. Due to lack of such vials, there **have been no published research results** showing the effect of **temperature on the rate and efficiency of reactive mechanical** synthesis in the literature so far.

The applicant successfully **designed and built** a prototype of a milling vial, allowing mechanical synthesis at temperatures up to **450 °C and pressures up to 100 bar** (hydrogen), with continuous acquisition of both pressure and temperature. Therefore, at the current state of the art of this manufacturing technique, it is possible to carry out reactive milling processes at different temperatures and pressures.

The project aims to study the behavior of selected metallic hydride forming materials, as a function of temperature, but also pressure, during their mechanochemical synthesis. The obtained results will allow revolutionizing the production of some hydride materials - used for hydrogen storage and understanding the impact of external factors on the mechanisms and formation reaction rates.