FISR project TEPSI:
Nanostuctured Mg for fast and reliable hydrogen delivery

ABSTRACT: The tailoring of hydrogen sorption kinetics of Mg hydride represents one of the most challenging opportunities for a safe storage and reliable use of hydrogen in on-board applications. The main technological limitations required for most applications, represented by the slow desorption kinetics at the too high desorption temperatures, can overcome by nanostructured hydrides and catalysts. By means of the support of detailed characterization of both functional properties and microstructure [1,2], the aim of the TEPSI project is to study the effect of nanostructures on the performances of the Mg-based hydrides and to perform new complementary synthesis methods. In particular, Mg-based nanostructured materials (thin films or nanopowders) are prepared by (1) Sputtering techniques, a suitable method for the synthesis of model materials where the properties can be easily correlated to the microstructure; (2) inert gas condensation, with the aim to study the fine details of hydrogen uptake and release in 3D nano-sized model systems and (3) high energy ball milling techniques which represents the main road for industrial scale up.

**HIGH ENERGY BALL MILLING:**
MgH₂ with catalysing Fe particles

**METALLOGRAPHIC ANALYSIS**

![DTA curves of MgH₂ in different conditions](image)

**FIRST PRINCIPLE CALCULATION**

<table>
<thead>
<tr>
<th>Sample</th>
<th>$t_1/2$ (sec)</th>
<th>$k_u$ (kJ mol⁻¹)</th>
<th>Rate Limiting Step</th>
</tr>
</thead>
<tbody>
<tr>
<td>pure MgH₂</td>
<td>3250±35</td>
<td>141±5</td>
<td>Nucleation</td>
</tr>
<tr>
<td>Mg + 0.06 at.% Nb</td>
<td>2390±55</td>
<td>140±5</td>
<td>Diffusion + Nucleation</td>
</tr>
<tr>
<td>Mg = 1 at.% Nb</td>
<td>290±10</td>
<td>78±5</td>
<td>Diffusion + Nucleation</td>
</tr>
<tr>
<td>Mg + 2-5 at.% Nb</td>
<td>110±10</td>
<td>51±5</td>
<td>Diffusion</td>
</tr>
</tbody>
</table>

**TEM images before and after H₂ sorption:**
In this study, cycling of Nb-doped Mg, the material passes from a micro- to a nano-structure.

**EXAFS measurements on Nb-doped Mg confirmed the presence of Nb atomic level clusters (~20nm) in as deposited sample, the presentation of NbH in hydrogenated samples and their size reduction after the desorption reaction.**

**Mg NANOPARTICLES by INERT GAS CONDENSATION:**

Typical TEM images of the Mg nanoparticles prepared by IGC apparatus at different He pressure associated to DSC curves of the respective samples. By decreasing the He pressure during the synthesis process, the average nanoparticle size decreases and the desorption performance improve.

**NANOSTRUCTURE MATERIALS by INERT GAS CONDENSATION:**

Without catalyst low energy SE is able to reveal microstructures images BSE confirm the results charging artefacts in high voltage SE images

We observe a coarse microstructure and a better microstructure at Mg/MgH₂ interface.