Processing parameters

After the studies conducted at **CNRS**, **the optimised composition** was selected by the consortium as selected composition for the HyCARE project, owing to its easy activation, good thermodynamics, kinetics, high reversible capacity at 55 °C and good cycling performances, as shown in **Deliverable 2.2**.
Then, a first 5 kg batch with the selected composition was produced by **GKN** at the industrial scale. The optimised and the standard GKN materials were produced and processed with different thermal treatments. These materials were distributed among partners of the consortium for characterization and for understanding the influence of composition (i.e. Mn amount) and different thermal treatments available at GKN on the hydrogenation properties.
This deliverable describes the GKN processing parameters for the synthesis and activation of the optimised and standard GKN alloys. Different experimental techniques were used among the partners for the characterization. Considering the goals of the project, an overview on the alloys properties have been presented together with a comparison of results across the partners. In details: structure, microstructure, particle size, density, thermal conductivity, composition, hydrogen storage properties (reversible capacity, thermodynamics, kinetics), poisoning and cycling properties have been reported.
The results reported in the deliverable will be integrated at the end of task 2.3, with further analysis and details, especially on cycling properties and resistance to poisoning of the selected material.

Testing, among HyCARE partners (CNRS, GKN, UNITO, HZG, IFE), of materials produced and processed by GKN evidenced no significant effect of thermal treatments on the microstructural and hydrogenation properties of the samples. Introduction of lower amount of Mn does not change significantly the hydrogenation properties, but it has a clear effect in shrinking the lattice parameter of the FeTi-type phase and shifting towards higher equilibrium pressure the PCI curves. The analysis performed confirm that the optimised samples are more suitable with respect to the standard ones.

The following general properties have been established:

- High content of Ti_4Fe_2O-type oxide phase: approx. 10 wt% from XRD in optimised sample, and approx. 20 wt% in standard samples
- Harsh activation: up to 100 °C and 20-50 bar needed. Milder activation observed (60-90 °C, 20 bar) by compacting the powders with addiction of a binder
- Limited reversible capacity: ~ 1.2 wt% between 2-25 bar at 55 °C
- Good cycling properties up to 25-40 cycles
- Thermodynamic were determined, with average values of ΔH~25 kJ/mol and ΔS~95 kJ/molK in absorption and ΔH~28 kJ/mol and ΔS~99 kJ/molK in desorption
- Good kinetics: t_{90} of ~ 5 minutes
- Poisoning under 50 ppm in volume of water together with oxygen (< 100 vol-ppm) and nitrogen (< 3000 vol-ppm) is in progress on selected sample

Since no significant improvement in reversible capacity or activation were observed as a function of thermal treatments, **HyCARE consortium selected the optimised composition produced by the established industrial process without thermal treatment**, as the best candidate to be scaled up to 5 tons for the demonstration tank. Furthermore, as the recorded usable storage capacity was lower than the initial target of the project (approx. 1.1-1.2 wt% compared to the initial target of 1.3 wt%), impact on powder production should be considered.
The full storage capacity of the LowMn samples of ~1.6 wt% is reduced when the process parameters of the system are considered. These process parameters are defined by the choice of the electrolyser, the fuel cell and the efficiency of the heating and cooling system installed. Indeed, the process parameters of the future system defined by the consortium consist in a maximum pressure of 25 bar, a minimum pressure of 2 bar at an averaged cycling temperature of 55 °C. According to the performed analysis presented here, it is confirmed by the different project partners, that the storage capacity that can be expected at 55 °C considering the limits of the system (2-25 bar) is equal to 1.1-1.2 wt%, below the set target of 1.3 wt%.