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Formation Reaction of Mg_2FeH_6: Effect of Hydrogen Absorption/desorption Kinetics

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In this paper we address some aspects of the kinetics of hydrogen absorption and desorption of Mg_2FeH_6 nanocomposite powder. The Mg_2FeH_6 material was prepared from a 2Mg-Fe composition by reactive milling under H_2 pressure at room temperature. After mechano-chemical processing, the material was mainly constituted of Mg_2FeH_6 having a hydrogen gravimetric capacity corresponding to 94% of the theoretical capacity. The kinetics of hydrogen absorption at constant temperature and the transformation of phases during this process were studied. The kinetics of hydrogen absorption presented a two-step behaviour. First, β-MgH_2 was formed with faster kinetics than Mg_2FeH_6 under constant temperature and H_2 pressure. Then, β-MgH_2 reacted with α-Fe forming Mg_2FeH_6 in a much slower reaction. Unexpectedly, reaction of hydrogen desorption showed an one-step kinetics, even if Mg_2FeH_6 and β-MgH_2 phases were previously present in the powder.

Keywords: *metal hydrides, high-energy ball milling, kinetics*

1. Introduction

Mg_2FeH_6 is a magnesium complex hydride belonging to the family of Mg_2THx (T=Fe, Co and Ni). It has a hydrogen gravimetric capacity of 5.4 wt.% which is higher than Mg_2CoH_5 (4.4 wt.%) and Mg_2NiH_4 (3.6 wt.%). The structure of Mg_2FeH_6 is cubic of K_2PtCl_6 containing octahedral $[FeH_6]^{-4}$ complexes anions surrounded by Mg in eight-fold cubic configuration[1]. This complex hydride has been reported to be a suitable material for thermo-chemical thermal energy storage around 500 °C due to its high enthalpy of hydride formation[2].

However, since its discovery in 1984[1], studies of Mg_2FeH_6 has been hampered by the difficulty to synthesize a pure material. This is due to immiscibility of Mg and Fe and thus the absence of the intermetallic compound, which could play the role of Mg_2FeH_6 precursor. Different techniques have been used to prepare Mg_2FeH_6 from 2Mg-Fe or $2MgH_2$-Fe mixtures: sintering[1], ball milling followed by sintering[3], ball milling of $2MgH_2$-Fe[4], reactive milling of 2Mg-Fe[5], cryomilling[6] and high pressure synthesis[7]. Regardless of the synthesis procedures some iron was always indentified in the processed material showing an incomplete reaction and resulting in a lower hydrogen capacity.

Another synthesis issue discussed in several papers is the mechanism of Mg_2FeH_6 formation. Some authors suggested that Mg_2FeH_6 is formed directly from the reaction of $2Mg+Fe+3H_2$[1,2,6,8], while others reported that the reaction involves two steps: the first one is the formation of MgH_2, and secondly, the reaction of magnesium hydride with Fe[5,9-11].

Polanski et al.[11] using in-situ synchrotron X-ray diffraction showed that MgH_2 is the Mg_2FeH_6 precursor. However, their measurements were performed by heating from room temperature to 500 °C under a initial hydrogen pressure of 85 bar. Under these conditions, the formation of MgH_2 could be kinetically favored at lower temperatures than Mg_2FeH_6. Thus, the reaction of Mg_2FeH_6 formation should be investigated at constant conditions of temperature and pressure.

Recently, we started a systematic investigation on the formation Mg_2FeH_6. From direct observation by TEM we found that the ternary hydride phase, Mg_2FeH_6, is formed with a columnar morphology, emerging from the MgH_2 particles and capped with Fe[12]. In this paper, we report the transformation of phases of a reactive milled 2Mg-Fe sample during hydrogen absorption process at constant temperature. Some aspects of the kinetics of hydrogen absorption and desorption are also discussed.

2. Experimental Procedure

As starting materials, magnesium (mesh –20+100, 99,80%) and iron (mesh –22, 99,998%), both provided by Alfa Aesar were used.

High energy ball milling was carried out in a Fritsch Pulverisette 6 planetary mill using 25 balls and crucible made of hardened stainless steel. The 2Mg-Fe composition was weighed with a ball-to-powder-ratio of 40:1 and loaded together with the balls into a crucible of 160 cm^3. After three atmosphere cleaning cycles, consisting of evacuation and

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argon injection, the milling container was filled with 30 bar of H_2 (99.999%). The sample was milled at 600 rpm for 48 hours. The experiment was not interrupted for sampling and the H_2 pressure was not recharged either. All handlings were performed inside an argon filled glove box.

The kinetics of hydrogen absorption and desorption were measured using a home-made Sieverts-type apparatus. The first hydrogen desorption was done under 1 bar of H_2 by heating from room temperature up to 350 °C with a temperature ramp of 10 °C.min^{-1}. This procedure was performed to evaluate the hydride's decomposition behavior during heating and the amount of hydrogen absorbed after ball milling. After initial desorption, the kinetics of hydrogen absorption were measured under 20 and 30 bar of H_2 at 350 °C. The hydrogen desorption behaviour was also investigated at 350 °C under 1.6 bar of H_2.

X-ray powder diffraction measurements were carried out in air using a Bruker D8 Focus apparatus with a CuKα radiation. Percentage of phases was evaluated from the X-ray powder diffraction patterns by Rietveld method using Topas software13.

3. Results and Discussion

3.1. *Reactive milling of 2Mg-Fe*

Figure 1 shows the X-ray powder diffraction pattern of the 2Mg-Fe sample milled 48 hours under 30 bar of H_2. The powder was made of 83 wt.% of Mg_2FeH_6, 11 wt.% of α-Fe, and 6 wt.% of MgO as calculated from Rietveld refinement. Based on these values the sample composition was 1.76 Mg:Fe, close to the initial composition of 2Mg:Fe. The broad peaks indicate a small crystallite size and presence of microstrain. For the Mg_2FeH_6 phase, crystallite size was evaluated as 15.6 ± 0.3 nm and microstrain 1.09 ± 0.04%.

Mg and β-MgH_2 phases were not clearly identified in the X-ray powder diffraction pattern. It is worth mentioning that the small crystallite size, the lower electronic density, iron fluorescence and volume fraction of those phases in comparison with Mg_2FeH_6 and α-Fe phases can make their detection quite difficult. Another possibility is the oxidation of β-MgH_2 when the sample was exposed to air to have a X-ray powder diffraction pattern taken[14]. All those aspects could explain the differences on the initial sample composition (2Mg:Fe) and that one estimated by Rietveld refinement (1.76Mg:Fe).

Based on the Rietveld refinement and considering a theoretical capacity of 5.44 wt.% of hydrogen for Mg_2FeH_6, the ball milled powder has an estimated hydrogen capacity of 4.51 wt.%. It is important to point out that a pure Mg_2FeH_6 was not attained despite the fact that we chose a long milling time and a highly energetic milling condition. As mentioned and discussed in a previous study[9], the presence of iron was always reported after ball milling of 2Mg-Fe or $2MgH_2$-Fe compositions regardless of milling conditions.

3.2. *Hydrogen desorption under heating*

After ball milling under H_2 pressure, the 2Mg-Fe sample was mainly constituted of Mg_2FeH_6. In order to evaluate its hydrogen desorption behaviour and also the amount of hydrogen absorbed during the mechanochemical processing, a 2Mg-Fe sample was heated up to 350 °C under 1 bar of H_2 with a temperature ramp of 10 °C.min^{-1} using the Sieverts-type apparatus.

Figure 2 presents the curves of hydrogen desorption kinetics and temperature ramp of the 2Mg-Fe sample prepared by reactive milling. The hydrogen desorption began around 100 °C with a very slow kinetics. The hydrogen desorption kinetics became faster at around 290 °C and the reaction was completed in 7 minutes. The release of hydrogen around 100 °C from a 2Mg-Fe sample prepared by reactive milling was previously identified by Asselli et al.[9] using simultaneous thermal analysis of differential scanning calorimetry, thermogravimetry and quadrupole mass spectrometry.

The curve of hydrogen desorption kinetics shows that the sample desorbed 5.11 wt.% of hydrogen, which corresponds to 94% of its theoretical capacity. The maximum hydrogen capacity for the 2Mg-Fe composition is 5.4 wt. %. This hydrogen capacity is higher than the one evaluated by Rietveld refinement (4.51 wt.%). From these results, we can

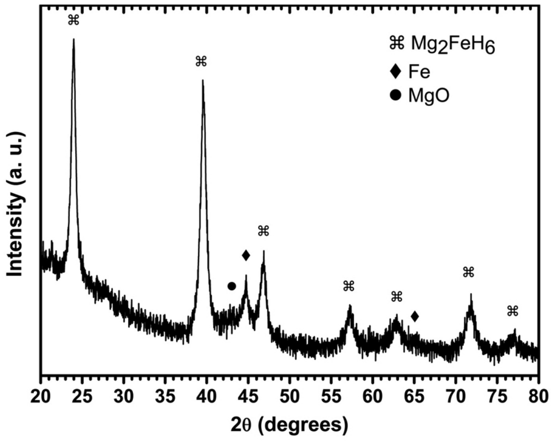

Figure 1. X-ray powder diffraction pattern of 2Mg-Fe reactive milled under 30 bar of H_2 for 48 hours.

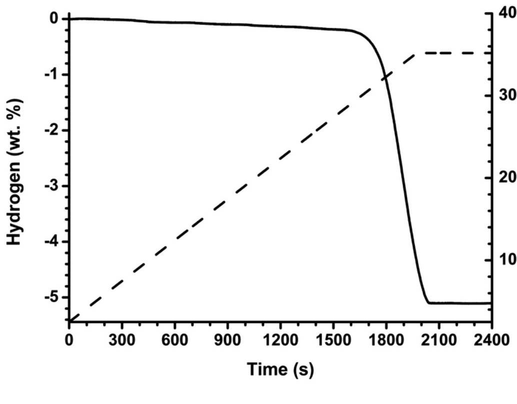

Figure 2. Simultaneous curves of temperature ramp (dash line) and hydrogen desorption kinetic (solid line) under 1 bar of H_2 of 2Mg-Fe reactive milled. Heating rate of 10 °C.min^{-1}.

hypothesize that the amount of α-Fe and MgO phases were overestimate by Rietveld refinement due to iron fluorescence and the difficulties to evaluate a nanocrystalline multiphase sample.

3.3. *Kinetics of hydrogen absorption*

After complete hydrogen desorption, the kinetics of hydrogen absorption was evaluated at 350 °C under two different H_2 pressures: 20 and 30 bar. Figure 3 presents the curves of hydrogen absorption kinetics of the 2Mg-Fe sample at 350 °C. The vertical axis of "Reacted fraction" is based on the theoretical hydrogen density of 5.44 wt.% for the 2Mg-Fe composition. Figure 3a draws attention to the absorption kinetic behavior in the first 20 minutes.

The hydrogen absorption kinetics under both pressures was very fast at the beginning and it seems that the reaction is terminated after only a few minutes. The use of a higher hydrogen pressure slightly accelerated the kinetics but mainly enhanced the hydrogen capacity. Nevertheless, the hydrogen capacity after 20 minutes was only 3.28 wt.% when the experiment was performed under 30 bar. This corresponds to 60 % of the theoretical capacity. As can be seen in Figure 3b the sample kept absorbing hydrogen after 20 minutes, although the kinetics was very slow. After 20 hours, the sample reached a hydrogen capacity of 3.56 and 4 wt.% under respectively 20 and 30 bar of H_2.

To investigate a possible change of phase composition upon hydrogenation, samples were quenched after 20 minutes and 20 hours under H_2 pressure. "Quenching" was done by closing the sample valve to the hydrogen gas source, retracting the heating unit from the sample holder area and cooling the sample holder down to room temperature in a few seconds using a cold water bath. As MgH_2 and Mg_2FeH_6 are both highly stable hydrides, the change of phase composition and hydrogen density are minimal upon quenching. Thus, the X-ray powder diffraction patterns of quenched samples truly reflect the crystal structure during the reaction of hydrogen absorption. Figure 4 shows the X-ray powder diffraction patterns of the 2Mg-Fe samples quenched after 20 minutes (Figure 4a) and 20 hours (Figure 4b) at 350 °C under 20 and 30 bar of H_2. By comparing Figures 4a, b, it is clear that even if the hydrogen capacity increased only slightly between 20 minutes and 20 hours the phase composition drastically changed. In Figure 4a, β-MgH_2 and α-Fe are the main phases in the sample hydrogenated for 20 minutes under 20 bar. Mg_2FeH_6 and Mg diffraction peaks are barely seen

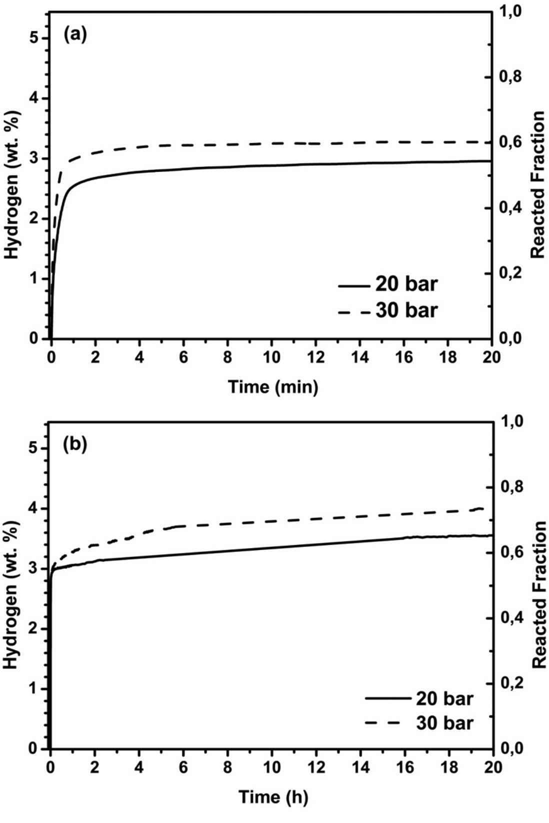

Figure 3. Curves of hydrogen absorption kinetics of 2Mg-Fe sample at 350 °C under 20 and 30 bar of H_2. The hydrogen absorption behaviour at the first 20 minutes and during 20 hours are shown in (a) and (b) respectively.

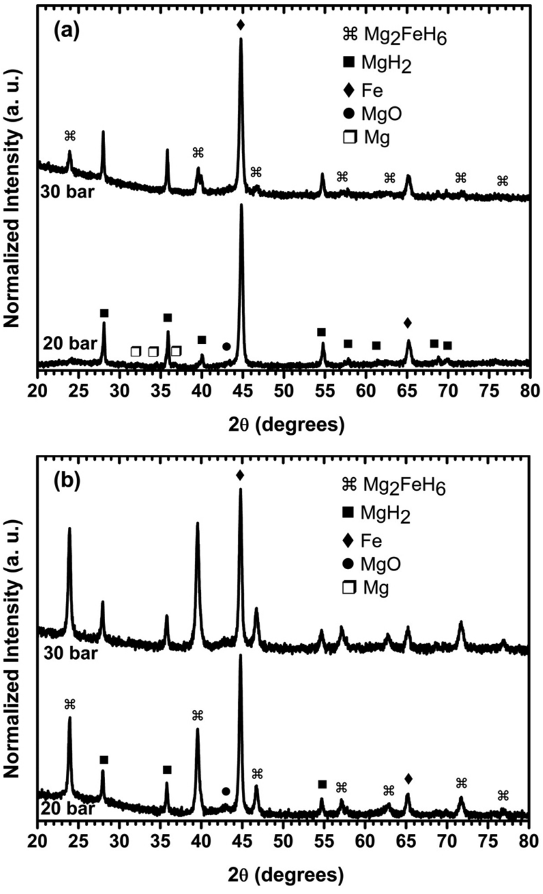

Figure 4. X-ray powder diffraction patterns of 2Mg-Fe sample after hydrogen absorption tests at 350 °C under 20 and 30 bar of H_2 for (a) 20 minutes and (b) 20 hours.

in the X-ray powder diffraction pattern. In the case of the sample hydrogenated under 30 bar for 20 minutes, some Mg_2FeH_6 has been formed but the main hydride phase was still β-MgH_2. No residual Mg was indentified. As shown in Figure 4b, after 20 hours, the hydrogenated samples were composed of Mg_2FeH_6, β-MgH_2, α-Fe and MgO, but it is clear that the percentage of Mg_2FeH_6 is much higher than after 20 minutes and that amount of β-MgH_2 is lower.

The mass percentage of phases were evaluated by Rietveld refinement and are shown in Table 1. During first 20 minutes of the absorption reaction under 20 bar of H_2 the main hydride phase formed was β-MgH_2 (35.6 wt.%). After 20 hours, the β-MgH_2 phase was reduced to 16.8 wt.% while at the same time the α-Fe content was reduced from 53.8 to 34.8 wt.%. The total weight reduction of β-MgH_2 and α-Fe was 37.8 wt.%. If we add this number to the original percentage of Mg_2FeH_6 (2.9 wt.%) we get 40.7 wt.%, which is very close to its amount deduced from the X-ray powder diffraction pattern after 20 hours (43.1 wt.%). This is consistent with the hypothesis that after a quick formation of β-MgH_2, this hydride reacted with α-Fe to produce Mg_2FeH_6. Here, it is worth pointing out that the molar mass of iron (55.8 $g.mol^{-1}$) is slightly higher than the double of β-MgH_2 (26.3 $g.mol^{-1}$) one. Thus, 18.8 wt.% of β-MgH_2 reacting with 19 wt.% of α-Fe represents approximately 2 moles of β-MgH_2 reacting with 1 mol of α-Fe to form 1 mol of Mg_2FeH_6.

One can observe the same kinetic behaviour using 30 bar of hydrogen pressure. In this case, after 20 minutes the formation of Mg_2FeH_6 was well underway but there was still a high proportion of β-MgH_2 and α-Fe. The percentage of the complex hydride Mg_2FeH_6 was slightly higher and consequently the amount of α-Fe phase was slightly lower when compared to the results after 20 minutes under 20 bar of H_2. After 20 hours, the total mass loss of β-MgH_2 and α-Fe was 34.0 wt.% while the mass fraction of Mg_2FeH_6 increased 34.8 wt.%. Thus, again it confirms that β-MgH_2 reacted with α-Fe to produce Mg_2FeH_6. However, even using a higher hydrogen pressure of 30 bar, a considerable amount of α-Fe is still unreacted after 20 hours at 350 °C.

The content of hydrogen calculated from the hydride phase percentages are presented in Table 1. The calculation was based on the hydrogen theoretical capacity of Mg_2FeH_6 (5.44 wt.%) and β-MgH_2 (7.6 wt.%) and on the percentage of phases estimated by Rietveld refinement. Adding up the amount of hydrogen in mass % calculated for the two hydride phases we get 2.87 wt.% (20 min) and 3.62 wt.% (20 h) for the hydrogenation under 20 bar, and 3.15 wt.% (20 min) and 4.05 wt.% (20 h) for the hydrogenation under 30 bar. These results agree very well with the values obtained by the measurements of hydrogen absorption kinetics presented in Figure 3.

Comparing the curves of hydrogen absorption kinetics showed in Figure 3 with their respective X-ray powder diffraction patterns in Figure 4, we can clearly state that the steep slope at the first minutes of absorption correspond to the reaction of Mg with hydrogen and the second slower step is the formation of Mg_2FeH_6 from the reaction of β-MgH_2 and α-Fe. In comparison to the experiment performed under 20 bar, the higher amount of hydrogen absorbed and consequently a higher amount of Mg_2FeH_6 phase after 20 hours of hydrogen absorption under 30 bar, is attributed to the higher driving force applied. Driving force is defined as the ratio of applied pressure over the equilibrium pressure. As our experiments were performed at constant temperature, we could assume that the equilibrium pressure of Mg_2FeH_6 and β-MgH_2 phases were constant.

It is important to emphasize that the complex hydride Mg_2FeH_6 has a lower equilibrium pressure than β-MgH_2[2,4,8]. This means that Mg_2FeH_6 is a more stable hydride than β-MgH_2. Besides, this also means that in a H_2 pressure-temperature condition where the formation of both hydride phases are thermodynamically possible, the driving force of Mg_2FeH_6 formation will always be higher than β-MgH_2 one. Nevertheless, our results showed that β-MgH_2 was formed before Mg_2FeH_6 with a very fast kinetics. Then, in a much slower reaction, β-MgH_2 reacted with α-Fe to form Mg_2FeH_6. This reaction has been directly observed by Danaie et al.[12]. Formation of MgH_2 before Mg_2FeH_6 is thus due to the much faster kinetic of the former reaction.

Despite having clear results showing that β-MgH_2 played the role of Mg_2FeH_6 precursor, the direct formation of Mg_2FeH_6 from Mg and α-Fe is still present. Of course, if the applied pressure is lower than the equilibrium pressure of MgH_2 but higher than equilibrium pressure of Mg_2FeH_6 then the only reaction possible is the direct formation from Mg and α-Fe.

3.4. *Kinetics of hydrogen desorption*

The dehydrogenation behaviour of the 2Mg-Fe sample after the hydrogen absorption tests was also studied. The kinetics of hydrogen desorption was measured at 350 °C under 1.6 bar of hydrogen. Figure 5 shows the curves of hydrogen desorption after being exposed to 20 and 30 bar of H_2 for 20 hours.

Table 1. Mass percentage of phase estimated by Rietveld refinement from the X-ray powder diffraction patterns of the 2Mg-Fe samples after the hydrogen absorption test (error ± 1%). The amount of hydrogen in mass % calculated to the hydride phases are shown in parentheses.

	20 bar		**30 bar**	
	20 min	**20 h**	**20 min**	**20 h**
Mg_2FeH_6	3 (0.16)	43.1 (2.34)	15 (0.82)	50 (2.72)
β-MgH_2	36 (2.71)	16.8 (1.28)	31 (2.33)	18 (1.33)
α-Fe	54	35	48	27
Mg	2	trace	trace	0
MgO	6	6	6	5

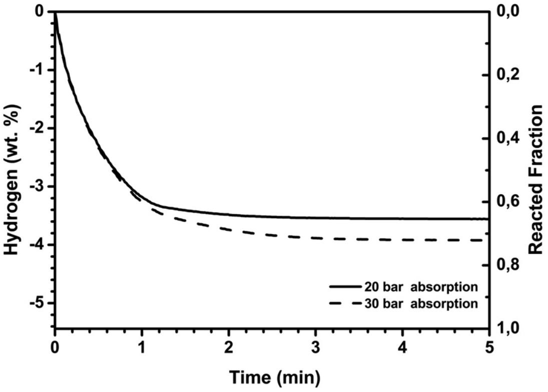

Figure 5. Curves of hydrogen desorption kinetics of 2Mg-Fe sample at 350 °C under 1.6 bar of H_2. The hydrogen desorption behaviour was measured after the hydrogen absorption tests at 350 °C under 20 and 30 bar of H_2 during 20 hours.

The reaction of hydrogen desorption is very fast, more than 80 % of the amount of hydrogen previously stored is released in one minute and the reaction of hydrogen desorption is fully achieved in less than 5 minutes. The intrinsic kinetic behaviour is similar in both curves and the difference in the amount of hydrogen released is related to the content of hydrogen previously absorbed (Figure 3b). Differently from the curves of hydrogen absorption kinetics, the desorption behaviour apparently does not present a two step reaction.

We have to emphasize that the interpretation of the one-step reaction of desorption process could be related to the measuring condition and its very fast kinetics. In order to confirm this behaviour a further in-situ desorption analysis is required.

4. Conclusions

We studied the hydrogen storage properties of a 2Mg-Fe sample prepared by reactive milling under H_2 pressure. After reactive milling, the sample was mainly constituted of Mg_2FeH_6 with a hydrogen capacity of 5.11 wt.% which is 94 % of the theoretical capacity.

After complete hydrogen desorption of the ball milled sample, the hydrogen absorption kinetics were measured at constant temperature and H_2 pressure. The hydrogen absorption was found to be a kinetically driven two-step reaction.

Under our measuring conditions, despite Mg_2FeH_6 being thermodynamically more stable than magnesium hydride, β-MgH_2 was formed before Mg_2FeH_6 with very fast kinetics. Then, α-Fe reacted with β-MgH_2 to form Mg_2FeH_6 in a much slower reaction. Therefore, we could conclude that, under the temperature and pressure conditions of this study, the reaction path is mainly determined by the kinetics.

A complete reaction of Mg_2FeH_6 formation could not be reached even after 20 hours at 350 °C under 30 bar of H_2. It shows the importance of new approaches to improve the kinetics of Mg_2FeH_6 formation, such as, the use of catalyst and new processing procedures.

Hydrogen desorption kinetics was also investigated. Under our measuring conditions, the dehydrogenation process presented a very fast one-step kinetics.

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