

## RESEARCH ARTICLE

## Features of Hydride Synthesis

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**Abstract:** Lithium, sodium, and titanium hydrides have been synthesized. It has been found that lithium hydride is formed at a temperature of 650-700°C and a pressure of 12 atm. Lithium hydride contains 11 mass.% of hydrogen. Hydrogen desorption from lithium hydride occurs at a temperature of 700°C. Lithium hydride decomposes in water to form lithium hydroxide and hydrogen. Sodium hydride is formed at a temperature of 300-450°C with a hydrogen absorption level of up to 4 mass%. Hydrogen desorption from sodium hydride occurs at a temperature of 350-400°C. Titanium hydride is formed at a temperature of 600-650°C with a hydrogen absorption level of up to 4 mass%. Hydrogen desorption from sodium hydride occurs at a temperature of 450-650°C. Lithium, sodium, and titanium hydrides can be used in hydrogen storage and transportation processes.

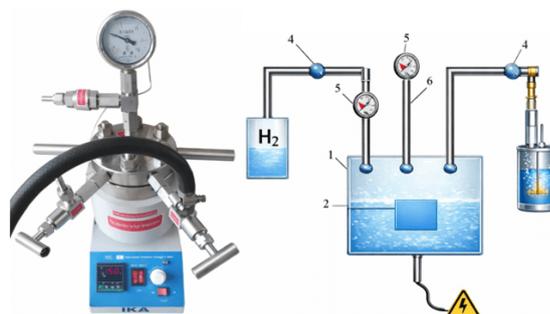
**Keywords:** metal hydrides, lithium hydride, sodium hydride, titanium hydride, hydrogen flow, aspect ratio

## 1 Introduction

By definition, metal hydrides are metal compounds with hydrogen. These compounds exhibit a high sorption capacity for hydrogen and can absorb large amounts of hydrogen and release it under certain conditions [1–11]. As a result, hydrides find applications in various fields, from environmental science to space technology. In this work, we investigate the synthesis processes of certain metal hydrides, including lithium, titanium, and sodium hydrides.

## 2 Methods

The hydrogenation process was carried out in a stainless steel reactor equipped with a high-temperature nozzle (Figure 1). The samples were pre-dried at 200°C, weighed, and then placed in the reactor.



**Note:** 1 - steel-smelting furnace-reactor; 2 - absorber; 3 - hydrogen cylinder for supplying gas to the reactor; 4 - shut-off and control valves; 5 - instruments for measuring pressure and temperature; 6 - thermocouple; 7 - gas tank.

**Figure 1** Photo (a) and diagram of a high-temperature reactor (b)

Before heating, hydrogen was introduced into the reactor to purge the chamber of air. After purging, the valve was closed and hydrogen was supplied at a pressure of 15 atm. Then, heating was initiated according to the specified temperature program. The experiments were conducted at temperatures ranging from 150 to 700°C. Cooling was performed either slowly in a turned-off furnace or rapidly, depending on the required conditions. After cooling, the mass (m) of the

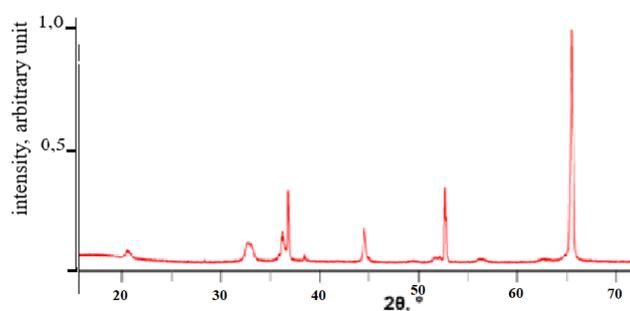
sample was measured. The degree of metal hydrogenation was estimated using the size ratio, which is defined as the ratio of the mass difference between the hydrogenated sample and the initial sample:

$$\alpha = \frac{M - M_0}{M_0} \times 100 \quad (1)$$

### 3 Results

Titanium powders absorbed hydrogen at a level of 3.8 wt.% at 12 atm and 700°C, which is slightly lower than the theoretical value of 4.04 wt.% for TiH<sub>2</sub>. Lithium samples showed high hydrogen absorption rates. Traditionally, these compounds are obtained by direct hydrogenation of molten metal. However, modern research focuses on reducing energy consumption and improving product purity. The process of obtaining a metal hydride is based on the interaction of a metal melt (lithium Li at ~700°C, titanium Ti at 650°C, and sodium Na at ~300-400°C) with pure hydrogen gas under pressure.

At a temperature of 700°C and a pressure of 12 atm, the hydrogen content in lithium metal reached 12.5 wt.%. At 350°C at a pressure of 12 atm, sodium absorbed hydrogen in an amount of 4 wt.%. Titanium showed activity for hydrogen sorption at the level of 4 wt.% at a temperature of 650°C and a pressure of 12 atm with the formation of TiH<sub>2</sub>. Figure 2 shows an X-ray of the initial lithium metal sample before exposure to hydrogen. (see Figure 2)



**Figure 2** X-ray diffraction pattern of the initial sample of metallic lithium before exposure to hydrogen

The X-ray diffraction pattern is characterized by a set of intense and narrow diffraction maxima corresponding to metallic lithium with a body-centered cubic lattice. The diffraction maxima and calculated physical values are listed in Table 1.

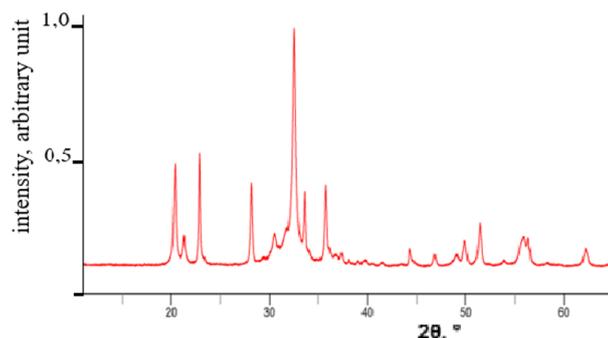
**Table 1** Diffraction characteristics and interplanar distances of Li

No.	$2\theta$	$\theta$	Interplanar distance (Å)	Miller indices (hkl)	Note
1	36.2	18.1	2.479	(110)	The main peak of lithium
2	52.3	26.15	1.748	(200)	The second reflex
3	65.1	32.55	1.432	(211)	The maximum intensity
4	77.0	38.5	1.237	(220)	The additional reflex

The main reflections were recorded at angles of  $2\theta$ , corresponding to the (110), (200), (211), and (220) planes, which is typical for FCC structures. The absence of additional peaks associated with lithium hydrides or oxides (LiH, Li<sub>2</sub>O, LiOH) indicates the high phase purity of the sample and minimal surface oxidation. The interplane distances dhkl were calculated using the Bragg law. For the most intense (211) reflection at  $2\theta = 65.1^\circ$ , the value  $d_{211} \approx 1.432$  Å was obtained. The cubic unit cell parameter was 3.507 Å for the (211) reflection, which is consistent with the data for metallic lithium at room temperature ( $a = 3.51$  Å).

The X-ray diffraction pattern (Figure 3) of lithium metal after treatment in a hydrogen atmosphere at a pressure of 12 atm and a temperature of 700°C allows us to assess the phase composition, degree of hydrogenation, and structural changes in the initial material. The diffraction pattern demonstrates a significant weakening of the peaks of the initial lithium metal, as well as the appearance of new diffraction maxima characteristic of LiH with a NaCl-type cubic lattice and the Fm3m space group, demonstrating partial or complete transformation of the metal into a hydride under these conditions.

Additional weak peaks in the range of  $20^\circ$ - $35^\circ$  correspond to surface interaction products with oxygen or moisture, such as LiOH and Li<sub>2</sub>O. The interplanar distances dhkl were calculated



**Figure 3** X-ray diffraction of metallic lithium after processing in a hydrogen atmosphere at a pressure of 12 atm and a temperature of 700°C

based on Bragg's law. For the (111) LiH peak at  $2\theta \approx 38.2^\circ$ , the angle  $\theta = 19.1^\circ$ , resulting in  $d_{111} \approx 2.356 \text{ \AA}$ . The cubic lattice parameter is  $\sim 4.081 \text{ \AA}$ , which is close to the reference value for LiH ( $\sim 4.084 \text{ \AA}$ ), confirming the formation of a stoichiometric hydride. (see Table 2)

**Table 2** Diffraction maxima and calculated LiH parameters

No.	$2\theta$	$\theta$	Interplanar distance (Å)	Miller indices (hkl)	parameter a (Å)	Note
1	38.22	0.3335	2.353	(111)	4.076	The main peak of lithium
2	44.39	0.3874	2.039	(200)	4.078	The second reflex
3	64.58	0.5636	1.442	(220)	4.078	The maximum intensity
4	77.72	0.6782	1.228	(311)	4.073	The additional reflex
5	81.85	0.7143	1.176	(222)	4.074	The additional reflex

The X-ray diffraction pattern (Figure 3) confirms the successful synthesis of lithium hydride with a cubic structure, high crystallinity, and stability of the hydride phase. The presence of additional peaks indicates the sensitivity of the material to oxygen and moisture, which can form surface hydroxides and oxides. These results provide valuable information about the kinetics and thermodynamics of the lithium hydride process. In the case of lithium, the hydride reaction follows the equation

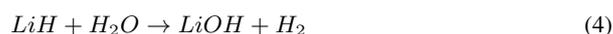


Therefore, both elevated temperatures (about 700°C) and high pressures (>15 atm) are required to achieve high absorption levels (>6% by weight). Metal hydrides are formed as a result of the interaction of metals with hydrogen, usually at significantly elevated pressures. The mechanism of metal hydride formation includes the adsorption of molecular hydrogen on the metal surface, the dissociation of  $H_2$  into individual atoms, and the diffusion of these hydrogen atoms into the metal crystal lattice. LiH has been found to decompose when heated to high temperatures (around 700°C) or when it reacts with water.

The thermal decomposition produces metallic lithium and hydrogen:



In its pure form, LiH begins to release hydrogen only near its melting point (688°C). Heating experiments in a vacuum show that significant desorption begins only at 600°C. When lithium hydride comes into contact with water, it undergoes an intense reaction that produces lithium hydroxide and releases hydrogen:



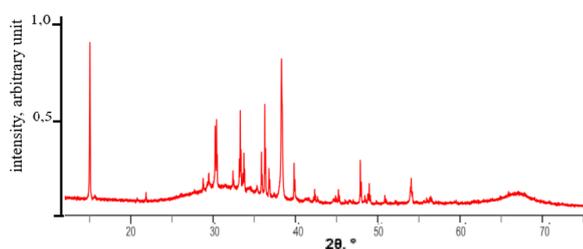
Experiments have shown that approximately 290 grams of hydrogen can be produced from 1 kg of lithium hydride when it reacts with water. This corresponds to a yield of 2.8 liters of hydrogen per 1 gram of the substance when it reacts with water. In the case of titanium hydride, it has been found that complete decomposition of  $TiH_2$  occurs at temperatures above 600-650°C. It has also been found that pre-grinding titanium hydride to a particle size of 100 nm reduces the activation energy of hydrogen desorption by 15-20%. Calculations show that the density of hydrogen in  $TiH_2$  is around  $90 \text{ kg/m}^3$ , which is higher than that of liquid hydrogen ( $70 \text{ kg/m}^3$ ). In the case of sodium, it has been found that direct hydrogenation of sodium is most effective at

temperatures between 350 and 400°C and a hydrogen pressure of around 10 atm. The yield of the product is 98%. It has also been found that the active decomposition of NaH begins at a temperature of 425°C in an inert atmosphere.

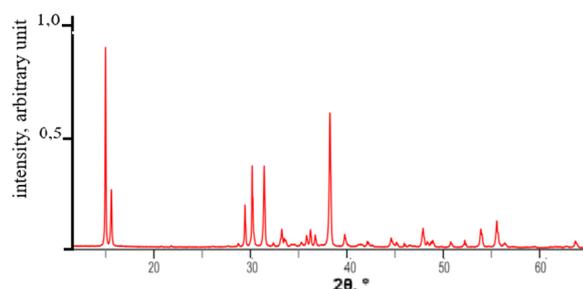
The analysis of the X-ray diffraction patterns in Figures 4, 5 and 6 shows that after treatment in a hydrogen atmosphere at a pressure of 12 atm and a temperature of 400°C, sodium metal is converted into sodium hydride.

Sodium hydride (NaH) is an ionic crystalline compound with a cubic face-centered (FCC) lattice similar to sodium chloride (NaCl), with the space group  $Fm\bar{3}m$ . The lattice parameter is approximately  $a = 0.488$  nm (4.88 Å) at room temperature. Ions  $\text{Na}^+$  and  $\text{H}^-$  form a structure where each ion is surrounded by 6 ions of the opposite sign. It decomposes at a temperature of about 300–425°C.

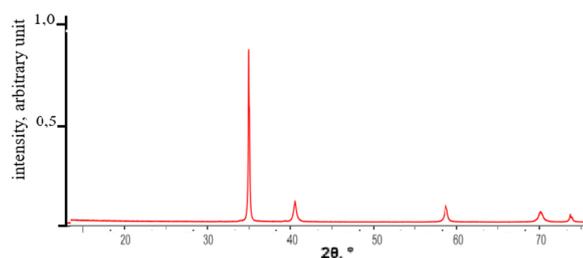
Titanium hydride ( $\text{TiH}_2$ ) has a crystal structure that depends on the temperature. Below 37°C (310 K), it has a tetragonal lattice (I4/mmm) with parameters  $a = 0.4528$  nm and  $c = 0.4279$  nm. When the temperature is increased (above 37°C), the structure changes to a face-centered cubic (fcc) phase. This structure allows titanium hydride to reversibly absorb hydrogen, making it an important material for hydrogen storage and metallurgy.



**Figure 4** X-ray image of the initial sample of metallic sodium before exposure to hydrogen



**Figure 5** X-ray diffraction pattern of the initial sample of metallic sodium after processing in a hydrogen atmosphere at a pressure of 12 atm and a temperature of 400°C



**Figure 6** X-ray diffraction pattern of the initial sample of metallic sodium after processing in a hydrogen atmosphere at a pressure of 12 atm and a temperature of 650°C

Based on the experimental results, a table of summary data for lithium, sodium, and titanium hydrides can be compiled. (see Table 3)

**Table 3** Summary of experimental data

Parameters	Hydrides		
	LiH	NaH	TiH <sub>2</sub>
Sorption temperature, °C	650–700	350–400	600–650
Desorption temperature, °C	600–700	380–430	450–650
Hydrogen pressure during synthesis, atm	1–5	10–50	1–2

## 4 Discussion

The synthesis of lithium, sodium and titanium hydrides is realized through the direct hydrogenation of molten metals under specific temperature and pressure conditions, and the formation of each hydride has its own characteristic reaction conditions and structural properties. Lithium hydride requires a relatively high temperature (650-700°C) and pressure (12 atm) for formation, and has a high hydrogen content (11 mass.%), which is related to the strong interaction between lithium and hydrogen atoms and the formation of a stable NaCl-type cubic lattice structure. The slight reduction of hydrogen absorption level of titanium powder at 700°C compared with the theoretical value may be related to the reaction temperature and pressure conditions, and the hydrogen absorption level can reach the theoretical value of 4.04 wt.% for TiH<sub>2</sub> when the temperature is adjusted to 650°C and the pressure is 12 atm.

Sodium hydride can be efficiently synthesized at a lower temperature (300-450°C), and the product yield can reach 98% at 350-400°C and 10 atm hydrogen pressure, which is due to the relatively active chemical properties of sodium and the formation of a stable FCC lattice structure similar to sodium chloride. The crystal structure of titanium hydride has a temperature-dependent phase transformation characteristic, which is the key reason for its reversible hydrogen absorption performance, and its hydrogen density is higher than that of liquid hydrogen, showing unique advantages in hydrogen storage application.

The surface of the synthesized hydrides is prone to react with oxygen and moisture in the air to form hydroxides and oxides, which is reflected in the weak impurity peaks in the X-ray diffraction patterns, indicating that the hydride materials have high sensitivity to the external environment and need to be protected during the synthesis and storage processes. The hydrogen desorption temperature of each hydride is closely related to its thermal stability: lithium hydride has the highest hydrogen desorption temperature (700°C), followed by titanium hydride (450-650°C), and sodium hydride has the lowest (350-400°C). Pre-grinding titanium hydride to nanoscale (100 nm) can effectively reduce the activation energy of hydrogen desorption, which provides an effective way to optimize the hydrogen desorption performance of hydride materials.

Lithium hydride has a special decomposition characteristic that it can react with water to produce a large amount of hydrogen, which makes it have potential application value in on-site hydrogen production, while the thermal decomposition of sodium and titanium hydrides is the main way of hydrogen desorption. The mechanism of metal hydride formation is a multi-step process including hydrogen adsorption, dissociation and atomic diffusion, which is the common theoretical basis for the synthesis of various metal hydrides, and the adjustment of temperature and pressure is the key to control the progress of this process and improve the hydrogenation degree.

## 5 Conclusion

It is shown that lithium hydride is formed at a temperature of 650-700°C and a pressure of 12 atm. Lithium hydride contains 11 wt.% of hydrogen. Desorption of hydrogen from lithium hydride occurs at a temperature of 700°C. Lithium hydride decomposes in water to form lithium hydroxide and hydrogen. Sodium hydride is formed at a temperature of 300-450°C with a hydrogen absorption level of up to 4 mass%. Hydrogen desorption from sodium hydride occurs at a temperature of 350-400°C. Titanium hydride is formed at a temperature of 600-650°C with a hydrogen absorption level of up to 4 mass%. Hydrogen desorption from sodium hydride occurs at a temperature of 450-650°C. Lithium, sodium, and titanium hydrides can be used in hydrogen storage and transportation processes.

## Conflicts of Interest

The authors declare that they have no conflict of interest.

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