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Received 27.06.2025

Revised 12.10.2025

Accepted 16.10.2025

[https://doi.org/10.53360/2788-7995-2025-4\(20\)-64](https://doi.org/10.53360/2788-7995-2025-4(20)-64)

MPHTI: 29.19.03



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THE INFLUENCE OF MECHANICAL ACTIVATION ON THE STRUCTURAL-PHASE STATES AND HYDROGEN ABSORPTION PROPERTIES OF THE INTERMETALLIC COMPOUND LaNi_5 – REVIEW

Abstract: The article provides an overview of a number of studies on the use of alloys and intermetallic compounds for hydrogen storage. Among them, a particularly important place is occupied by the inter-metallic compound LaNi_5 type. The latest achievements in the development of intermetallic compounds of the AB_5 -type are analyzed. These compounds, in particular LaNi_5 and its alloyed counterparts, are widely used due to the ability to regulate their properties by replacing elements. The article also provides an overview of the methods of synthesis and modification of AB_5 alloys aimed at improving their efficiency in hydrogen technologies. Both traditional production methods and modern technological approaches, including Spark plasma sintering and mechanical activation, are considered. A review of the scientific literature has shown that mechanical activation is an effective way to modify the LaNi_5 intermetallic compound to improve its hydrogen absorption properties.

According to a number of studies, the effect of high-energy ball milling leads to significant changes in the microstructure of the material. It is shown that to increase the effectiveness of practical applications of LaNi₅ in hydrogen energy, additional comprehensive studies are needed to establish the relationship between mechanical processing parameters, structural characteristics, and functional properties of the material. The aim of this work is to use mechanical activation to modify the microstructure of a material, reduce the size of crystallites, increase the density of defects, and promote the formation of amorphous or nanostructured states, which together can significantly affect the structure and interactions with hydrogen.

Key words: hydrogen storage, intermetallic compounds, hydrides, mechanical activation.

Introduction

Among the many metal hydride materials, the intermetallic compound LaNi₅ attracts special attention due to its high reversible hydrogen capacity, stability in multiple absorption-desorption cycles, and relatively low saturated hydrogen vapor pressure at room temperature. However, one of the main limitations of its practical application in hydrogen storage systems is the insufficiently high kinetics of hydrogen absorption and the need to activate the material before the first use. The main focus of this work is on studying the effect of mechanical activation on the change in the structural-phase states and hydrogen absorption characteristics of LaNi₅. Thus, metal hydrides such as LaNi₅ play an important role in modern hydrogen storage technologies, offering safe and efficient solutions for various applications. Despite significant progress in this area, there are still many unresolved issues, such as optimization of the sintering process, increasing the stability and strength of materials. The prospects for using these materials in integrated circuits open up new horizons for energy and hydrogen storage microsystems, which makes further research in this area very relevant.

Mechanical activation of intermetallic compound is an effective method to improve its hydrogen absorption properties. These results open up new perspectives for the development of highly efficient hydrogen storage materials, which are of great importance for applications in energy and clean technologies.

The IPS method is a promising method for obtaining materials capable of efficiently absorbing and desorbing hydrogen, such as intermetallic compounds, hydrides and porous metals.

Application of AB₅ type hydrogen storage materials and the prospects of LaNi₅

In this paper, the effect of mechanical activation on the structure, phase composition, and hydrogen absorption properties of LaNi₅ is considered. Particular attention is paid to the relationship between microstructural changes occurring as a result of mechanical action and the improvement of hydrogen absorption parameters. The obtained data are considered important for optimizing the characteristics of metal hydride hydrogen storage systems and, accordingly, increasing the efficiency of energy devices. As is known from the works, hydrides of intermetallic compounds are very well studied and are currently widely used in research. Intermetallic compounds of the AB₅ (e.g., LaNi₅), AB₂ (e.g., ZrV₂), A₂B (e.g., Ti₂Ni) and AB (e.g., TiFe) type with atoms of rare earth and/or transition metals used for hydrogen storage are considered. Metal hydrides are easily formed when these compounds interact with hydrogen at 290-475K and a pressure of 0.1-1.0 MPa. Hydrides have a high bulk density for hydrogen, but their mass capacity does not exceed 3%. Partial replacement of the main components A and B with various elements (Ti, V, Cr, Mn, Ni, Fe, Co, Y, Ce, Cu, Zn, etc.), even with impurities of oxygen, nitrogen, carbon (for example, up to Ti₂Ni), allows to increase the sorption capacity, improve the characteristics and reduce the cost. [1].

According to sources [2-3], hydride-forming materials (LaNi₅, TiFe, TiCr₂, Mg₂Ni and others) have a number of advantages as materials for storing hydrogen: high volumetric storage density, safety due to the formation of a hydride solid phase, selectivity of hydrogen absorption, etc. LaNi₅ is a typical representative of hydride-forming IMS class AB₅.

Hydrogen stands out as an efficient and environmentally sustainable energy carrier. Metals that can form hydrides exhibit high selectivity for hydrogen and exhibit very low permeability to other gases. The hydrogen transport mechanism begins with the decomposition of molecular hydrogen (H₂) into atomic hydrogen on the metal surface, followed by diffusion of these atoms through the metal layer, their re-integration into molecules, and finally desorption.

Among the intermetallic compounds containing rare earth elements, alloys of the LaNi₅ type are among the most studied materials. The discovery in 1970 of the ability of the LaNi₅ compound to form hydrides stimulated the rapid development of research in this area. This discovery paved the way for extensive research into hydrogen-absorbing intermetallic systems (IMS), which ultimately

contributed to the commercialization of nickel – metal hydride (Ni – MH) batteries and the development of hydrogen storage technologies.

At room temperature and slightly above atmospheric pressure (1.6 atm), this compound forms the hydride LaNi_5H_6 . The hydrogen content in this form is about 1.38 wt.%. The hydride decomposes easily at a slight increase in temperature or decrease in pressure, regenerating the original intermetallic compound (IMC) and hydrogen. The volume fraction of hydrogen atoms in the LaNi_5 compound is approximately 1.5 times higher than in liquid hydrogen. The process of hydrogen saturation of this intermetallic compound takes less than 10 minutes at room temperature, and the main part of the gas is absorbed in the first 3-5 minutes. Like other intermetallic compounds of the AB_5 composition, LaNi_5 is distinguished by its high density, resistance to impurities in a gas environment, and endurance to multiple cycles of hydrogenation and dehydrogenation. To determine the structural parameters of the hydride phases, hydrogen-saturated samples are kept under pressure in liquid nitrogen, and then stored at a temperature of 77 K (in air) for 1-2 hours. Such treatment contributes to the formation of a passivation layer on the surface of the sample, preventing the decomposition of the hydride for several hours. This time is sufficient for X-ray phase analysis. During the mechanical activation process, the morphology of intermetallic powders changes according to the principles for all studied intermetallic compounds (Fig. 1). The effect of hydrogen causes a significant crushing of IMS particles. With an increase in the processing time, the size of amorphous fine particles increases in the ball mill.

The effect of mechanical activation on the structural characteristics and parameters of IMS samples was studied by X-ray phase analysis. The X-ray diffraction patterns of the initial and mechanically activated alloys, as well as their hydrides, are presented in Figure 2. These results clearly demonstrate the changes in the crystal structure of the materials, i.e. the evolution of the phase composition and lattice parameters after mechanical processing. The values of the structural parameters calculated for these samples are given in Table 1.

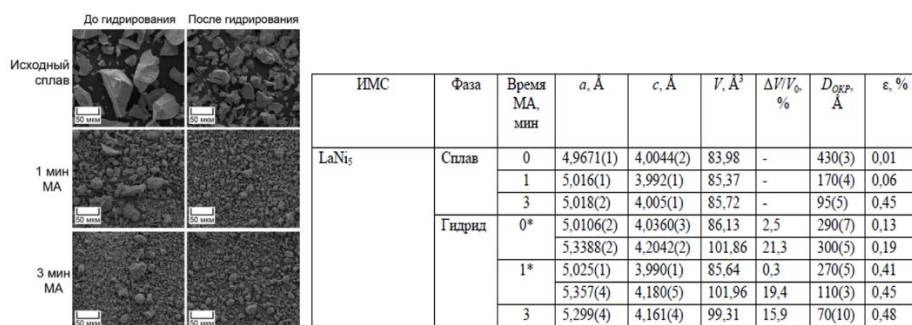


Figure 1 – Shows SEM (scanning electron microscope) images of LaNi_5 alloy in the initial state and after mechanical activation, which underwent a three-cycle hydrogenation-dehydrogenation process. In addition, the table shows the crystal structure and microstructure of the hydride-forming intermetallic compounds, as well as the parameters of the corresponding hydride phases before and after mechanical activation [3]

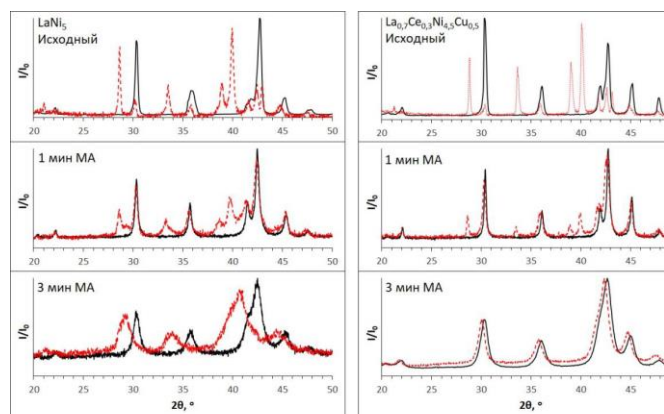


Figure 2 – Diffraction patterns of hydride-forming intermetallic compounds, as well as the corresponding hydride phases, before and after mechanical activation:

LaNi_5 (a), $\text{La}_{0.7}\text{Ce}_{0.3}\text{Ni}_{4.5}\text{Cu}_{0.5}$. Black lines are alloys, red lines are hydrides [3]

The data in the table describe the increase in structural defects of the alloy as a result of mechanical activation and the change in lattice parameters under the influence of the hydride formation process.

The identified characteristic peaks are consistent with literature data [3]. All studied compounds, including the hydrides, possess a hexagonal structure of the CaCu_5 type, belonging to the $P6/mmm$ space group. The addition of other metals to the LaNi_5 composition does not lead to the formation of any additional phases. In all intermetallic compounds, the c lattice parameter remains nearly unchanged, while the a parameter increases when nickel is replaced by aluminum, cobalt, or manganese, and decreases when lanthanum is substituted with cerium.

The positions of the characteristic diffraction peaks remain unchanged during the mechanical activation process, indicating that the crystalline structure of the intermetallic compound is retained after 1 and 3 minutes of ball milling. However, with increasing milling time, the diffraction peaks broaden. This broadening is attributed to a reduction in the size of the coherent scattering regions (DOC) and an increase in the level of microstrain (ϵ) (Table 1). These effects become particularly pronounced after 3 minutes of mechanical treatment.

In [4], the effect of mechanical activation in a high-energy planetary ball mill on the hydrogen sorption and structural properties of the intermetallic compound $\text{LaNi}_{2.5}\text{Co}_{2.4}\text{Mn}_{0.1}$ was studied in relation to its use as a filler in composite metal-polymer membranes for hydrogen separation from gas mixtures. High-energy mechanical activation significantly influences not only the physical properties of metal hydrides but also their hydrogen absorption and desorption behavior. While the enhanced kinetics of hydrogen uptake and release are primarily attributed to the reduction in particle size and the creation of additional reactive surface area, the changes in the thermodynamic characteristics of the hydrogenation process are more complex in nature. The choice of the object in this work is based on a number of important parameters that play a decisive role in the adaptation of metal hydride materials for membrane gas separation processes, several key factors must be considered. First, the equilibrium pressure for hydride formation should not exceed 1 atm within the operating temperature range (up to 60 °C). Second, the specific volume expansion during hydrogen absorption must be minimal to ensure compatibility at the metal-polymer interface. Third, the particular structure of the metal hydride phase diagram is advantageous for maintaining stability during repeated hydrogenation-dehydrogenation cycles, characterized by an enlarged solid solution region and a short plateau.

The intermetallic compound LaNi_5 is often used, including in their studies [5], as a reference material for testing installed facilities and for training students to determine the properties of hydrogen storage. Fig. 3 shows the isothermal dependences characteristic of the $\text{LaNi}_5 - \text{H}_2$ system. Upon formation of the hydride phase – resulting from the incorporation of hydrogen atoms into octahedral and tetrahedral sites – the crystal structure of the metal lattice remains unchanged; however, the lattice parameters and the unit cell volume increase by approximately 20-25% (Fig. 4).

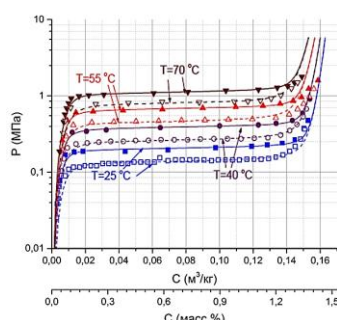


Figure 3 – Isotherms « H_2 pressure – hydride phase composition» in the $\text{LaNi}_5 - \text{H}_2$ system: solid lines – absorption, dashed lines – desorption [5]

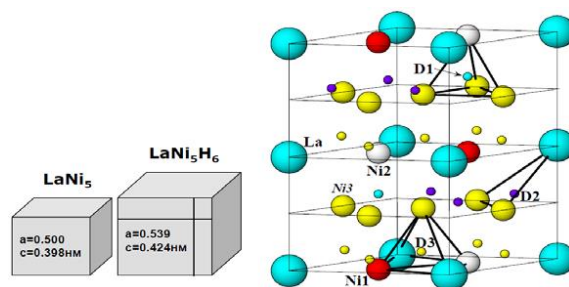


Figure 4 – Diagrammatic representation and lattice parameters of the unit cells of LaNi_5 and LaNi_5H_6 (left) along with the arrangement of D atoms within the LaNi_5D_6 lattice (right) [5]

The structures of AB_5 -type intermetallic compounds and their corresponding hydrides have been extensively investigated and documented in the literature [6]. These compounds adopt the CaCu_5 structural type, featuring a hexagonal lattice within the $P6/mmm$ space group. The

introduction of dopant elements into these intermetallic compounds alters the size of the structural voids due to differences in the crystallographic radii of the constituent metal atoms. To evaluate the impact of these structural modifications on hydrogen storage properties, the unit cell volumes provide a convenient metric. Among the AB₅-type intermetallics, the most promising systems are based on rare-earth metals (La, Ce, and unalloyed REM mixtures-mischmetal, Mm) alloyed with nickel and other transition metals.

The greatest difference between hydrogenation and dehydrogenation pressures occurs during the initial cycles, which is attributed to the fragmentation of larger particles down to the micron scale (Fig. 5).

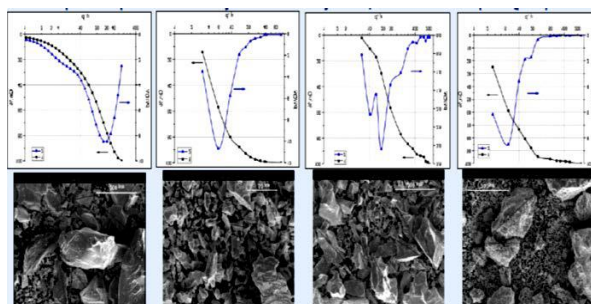


Figure 5 – SEM micrographs and particle size distributions of LaNi₅ after 10 cycles (A) and 18,180 cycles (B), as well as a material based on La_{0.5}Ce_{0.5}Ni₅ after 10 cycles (C) and 18,180 cycles (D) [6]

In many studies, the compositions of metal hydride alloys of the AB₅ type have been widely studied from the point of view of hydrogen sorption properties and various alloy compositions with elements of microalloying of the LaNi₅ alloy base – cerium, aluminum, manganese, etc. – have been developed. In this direction, the authors' task was to develop the technological foundations for obtaining these alloys and the supply of pilot industrial small-tonnage lots. The manufacture of metal hydride alloys of the AB₅ type was carried out as follows. Initially, the alloys were smelted in ingots weighing up to 30 kg. A larger ingot mass can lead to liquation heterogeneity of microalloying additives (Ce, Mn, Al) and, as a consequence, to a decrease in the properties of the alloys or the appearance of the effect of their heterogeneity [7].

In this context, the literature [8-11] highlights the widespread use of hydride-forming intermetallic compounds in the development of hydrogen batteries and compressors, heat pumps, and Ni-MH-type energy devices. The key properties of metal hydride materials include hydrogen storage capacity, reversibility, phase transition characteristics (such as hysteresis, plateau slope, enthalpy, and entropy), cyclic stability, hydrogenation kinetics, and hydrogen diffusion. The performance of metal hydride electrodes is influenced by multiple factors, including interfacial phase transitions, hydrogen migration through the surface layer, diffusion within the bulk, and the transformation from the α -phase solid solution to the β -phase metal hydride. Cyclic absorption and desorption of hydrogen have two major physical and irreversible consequences for intermetallic compounds such as LaNi₅: defect formation and sputtering. Both phenomena can significantly affect the properties of these compounds depending on the application. While defect formation leads to amorphization and a decrease in capacity after long-term cycling. Also in the article [12] Based on the analysis of literary data, hydride-forming intermetallic compounds (La, Ce) Ni₅ were chosen as working materials for hydrogen compression. They are easily activated, have a high rate of reversible hydrogen sorption and narrow hysteresis, are cyclically stable, are not poisoned by impurities in electrolytic hydrogen [12]. Metals and intermetallic compounds that form hydrides often require a pre-treatment known as activation. In some cases this is as simple as soaking in H₂ to remove surface contaminants. Most of the literature on LaNi₅ is devoted to the P-C phase diagrams and crystal structure models. This paper presents a comprehensive study of the activated state formation using P-C manometric measurements, X-ray and neutron diffraction, optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [13]. In this work, the proposed method is used to determine the sorption characteristics of LaNi₅ IMC with a hexagonal structure. For the first time, the phase diagrams of the LaNi₅-H₂ system are constructed both in the two-phase region (α + β) and at supercritical parameters [14]. In particular, the LaNi₅ alloy is of great technological interest for such application areas as compressors, heat pumps, rechargeable batteries and energy conversion materials. In addition, the LaNi₅-H system is considered to be a

prototype of hydrogen absorption materials due to its excellent hydrogenation properties. The hydrogen storage capacity of LaNi_5 is about 1.38 wt%, which is larger than the storage capacity of liquid hydrogen [15].

Much research is being conducted on materials that store hydrogen in a chemically bound state, such as metal hydrides or intermetallic compounds (IMCs). Since the ability of metals to store hydrogen was first discovered, scientists have been particularly interested in this class of compounds [16]. Although AB₂ alloys are considered to have higher specific energy than AB₅ alloys, AB₅ alloys are mainly used in commercial NiMH batteries. AB₅ alloys are based on LaNi_5 with various La and Ni substitutes. The systematic effects of alloy modification and the causes of these effects are current research topics [17]. Another study used pure powders of intermetallic compounds Mg, Ni, LaH, MgH and LaNi_5 . Powder mixtures with a given composition were mechanically crushed in an argon medium was carried out in a Spex 8000 ball mill. In order to monitor structural changes, small amounts of powder were taken at certain intervals [18]. According to the work, magnesium alloys in the field of triple eutectic (composition 70-75% by weight Mg, 6-9% by weight (La)Mm, 19-21% by weight Ni), consisting of phases Mg, Mg_2Ni and $\text{La}(\text{Mm})_2\text{Mg}_7$, are of great interest for hydrogen storage. These alloys are capable of absorbing up to 5.4-5.8% H₂ mass fraction at temperatures and pressures of 520-550 K of 1.0-1.5 MPa. Hydrogen is released from the formed hydride phases at a temperature of 610-620 K and a pressure of 0.15-0.20 MPa. With multiple hydrogenation-dehydrogenation cycles, the sorption characteristics do not change significantly [19]. The intermetallic compound LaNi_5 and its metal-substituted derivatives have been studied by many authors because they are used as anode materials in nickel-metal hydride (Ni-MH) batteries, which in turn can replace the toxic element cadmium in alkaline batteries. However, the binary compound LaNi_5 is subject to severe corrosion during cycling due to the breakdown of the intermetallic compound in alkaline electrolyte, resulting in a rapid decrease in capacity with increasing number of cycles [20]. Nickel-metal hydride (Ni-MH) batteries, which use hydrogen-storing alloys as negative electrodes, have been widely studied due to their environmental safety and high operating efficiency. However, the electrochemical capacity of AB₅-type alloys, which are limited to the hexagonal structure of the CaCu_5 type, is insufficient. In addition, the energy density of Ni-MH batteries is low compared to some new-generation secondary batteries. In this regard, the development of new alloy types with high energy density, fast start-up capability, efficient operation in fast discharge mode, and low production cost has become a pressing issue [21-23]. It is well known that the substitution of La in LaNi_5 – type Ce or Mm alloys (industrial rare earth alloy) significantly affects the equilibrium pressures of hydrogen absorption and desorption, hysteresis parameters and hydrogenation/dehydrogenation enthalpies [24]. In order to improve the performance of nickel-metal hydride batteries, replacing nickel in LaNi_5 -based metal hydride electrodes with other alloying elements is of great interest to many researchers. In fact, multicomponent alloys are usually used for practical applications for hydrogen storage. However, many studies devoted to the effect of alloying elements were mainly based on individual and discrete additives. Among the elements replacing Ni, Co, Mn and Al are the three most important elements added to hydrogen storage alloys. The beneficial effects of these elements have been studied extensively. Taking into account the operating conditions and cost, the alloying elements and their contents should be adjusted to achieve the desired optimum performance. However, in LaNi_5 -based alloys containing (Co,Mn,Al), changes in the electrochemical properties of metal hydride electrodes with a fairly wide range of Ni contents are rare [25]. As demonstrated by numerous studies, partially substituting nickel atoms with aluminum in the LaNi_5 alloy significantly lowers the equilibrium pressure in the plateau region and reduces the hysteresis between hydrogen absorption and desorption pressures. However, partial replacement sharply reduces the hydrogen absorption capacity of the intermetallic compound. Especially at low temperatures, desorption slows down significantly. In addition, the presence of only 2 at.% manganese in the alloy reduces the equilibrium pressure in the system by almost half compared to LaNi_5 , and even when the manganese content increases to 5 at.%, the hydrogen absorption capacity remains practically at the level of the original LaNi_5 compound. The conducted X-ray phase analysis confirmed the single-phase nature of the obtained sample and showed that the studied alloy has a hexagonal crystal lattice characteristic of the CaCu_5 structural type with the parameters $a = 5.045 \pm 0.005 \text{ \AA}$ and $c = 4.027 \pm 0.005 \text{ \AA}$ [26]. It was shown in the works that even at a temperature of about 320 K, during long-term holding, ordering of dislocations in the metal occurs and experimentally, using precision equipment, it is possible to record a change in the

equilibrium pressure, hysteresis and heat of the hydrogenation reaction. The temperature conditions 313, 320, 353 K. are shown here. The intermediate hydride phase was structurally characterized by neutron diffraction, and changes in the nature of the stress state of the intermetallic matrix, concentration, and type of defects in the crystal structure were identified as the reason for its appearance [27].

Structural and phase characteristics of LaNi₅

Nickel metal hydride (Ni/MH) batteries based on alloy electrode for hydrogen storage have several important advantages over nickel cadmium (Ni/Cd) batteries, these systems have advantages such as high energy density and capacity, resistance to overcharging and deep discharge, as well as the absence of toxic heavy metals and no electrolyte consumption during charge/discharge cycles [28]. Magnesium hydride is a promising material for hydro regulation of the composition in the Mg-LaNi₅ system. It is used in this generation of storage systems due to its high capacity (Fig. 6). However, the slow work we are doing investigating the Mg-50 wt.% LaNi₅ composite on hydration and dehydration kinetics of pure magnesium, as well as the effect of high-intensity milling on the structure, limits its practical application. A number of studies have shown that the hydrogen storage properties are also subject to change. Doping magnesium with catalytic transition elements such as nickel, cobalt, titanium, or iron, as well as with intermetallic compounds like LaNi₅, FeTi, and Mg₂Ni, enhances the hydrogenation process. Additionally, effective experimental results on the dehydrogenation kinetics of magnesium at high temperatures (573 K) have been reported [29].

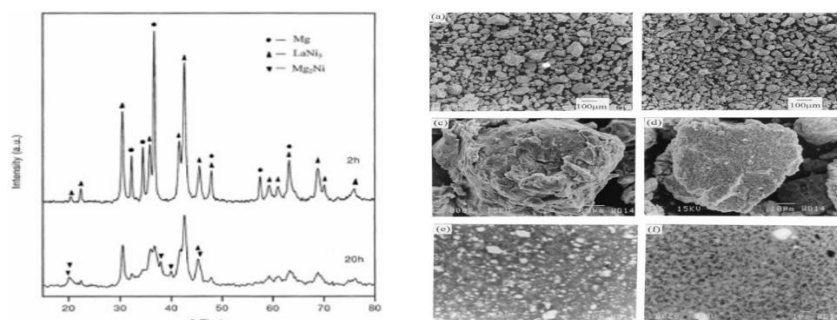


Figure 6 – X-ray spectra of Mg–50 wt% LaNi₅ composite after 2 and 20 h of grinding and SEM microstructures of Mg-50 wt.% LaNi₅ composite before (a, c, e) and after (b, d, f) hydrogenation; (a), (b) low-magnification images and (c), (d) high-magnification images; (e), (f) high-magnification scattered images [29]

In the study by the following authors, a mixture of magnesium (99.95%, 150 mesh) and nickel (99.95%, 250 mesh), purified hydrogen and argon (H₂O and O₂, less than 20 ppm) were used to produce Mg₂Ni. The parameters of ball milling were as follows: the rod to metal powder ratio was 20 to 1, the rotation speed of ball milling was 0.2 s⁻¹, and stearic acid was used as the processing reagent. Detailed TEM analyses were carried out to understand the change in the fine structure of mechanically alloyed Mg₂NiH₄ particles during the above milling steps. The hydrogen absorption properties of the nanocrystalline Mg₂Ni alloy obtained by the ball milling method were better than those of the conventional Mg₂Ni alloy [30].

According to the author of [31], several methods have been used to improve the properties of R–Mg–Ni-based alloys intended for hydrogen storage, such as element replacement in the alloy composition, rapid quenching, composite alloying, and surface modification. Among these methods, element replacement has proven to be an effective way to improve the cyclic stability of R–Mg–Ni-based alloys. The hydrogen storage materials used are rare earth alloy with magnesium and nickel AB₃ with soft hydrogen storage conditions, moderate platform pressure and abundant raw material reserves, etc. The alloys can be produced by induction melting [32]. If we consider magnesium as an alloying element, that is, in the following literature [33] already in the 1970s it was discovered that solid solutions based on magnesium with a minimum content of the second component (up to 1 at.% Ag, Al, Cd, In, Pb, Y, Zn and Al, Ga, In) or mechanical mixtures of magnesium with hydrous metals and intermetallic compounds (Pb, V, LaNi₅, TiFe) were found to surpass individual magnesium in the rate of interaction with hydrogen. Nickel is one of the most effective activating components. It forms a compound Mg₂Ni with magnesium, which is capable of reversibly absorbing hydrogen. The

hydrogen storage alloy $\text{La}(\text{Ni}_{1-x}\text{Fe}_x)_5$ was synthesized by melting stoichiometric mixtures of individual elements. High purity elements including La (99.9%), Ni, Fe and Co (99.99%) were taken in correct stoichiometric proportions and pressed into pellets ($1 \times 0.5 \text{ cm}^2$). The hydrogenation performance of the alloys was evaluated by measuring P-C isotherms and kinetics. The desorption isotherms corresponding to LaNi_5 and $\text{La}(\text{Ni}_{1-x}\text{Fe}_x)_5$ alloy were found to have the maximum storage capacity of 1.93, 1.96, 2.20, 1.78, 1.60 wt%. The X-ray diffraction analysis confirmed the formation of hexagonal structures of the CaCu_5 type for all the studied composition [34].

This study investigates the XRD patterns of LaNi_5 -based alloys, with particular attention to the direction and extent of peak broadening observed after activation and subsequent hydriding–dehydriding processes. Emphasis is placed on the development and accumulation of lattice strain in LaNi_5 , as well as the influence of partial substitution of Ni with other elements on the strain formation within the crystal lattice [35]. A brief overview is provided of both qualitative and quantitative models that connect hydriding behavior – such as stability and hydrogen capacity – with various physical parameters, including formation enthalpy, unit cell volume, interstitial site dimensions, and electronic structure of these intermetallic compounds. The authors' specific contribution lies in establishing a relationship between compound stability and formation enthalpy. Furthermore, analysis of the crystal structures of certain Ni-substituted LaNi_5 compounds and their corresponding hydrides reveals a clear link between hydrogen absorption capacity and the number of hydrogen-occupied sites [36]. The LaNi_5 system adopts a hexagonal crystal structure of the CaCu_5 -type, characterized by the P6/mmm space group, also known as the Haucke structure [37]. Among hydrogen storage alloys, AB_5 -type compounds are the most widely utilized as negative electrode materials in nickel–metal hydride (Ni-MH) rechargeable batteries for portable electronic devices [38]. Observed phase transformations, element segregation, and structural degradation in $\text{La}_2\text{Mg}_{17}$ with 40 wt% LaNi_5 during repeated hydrogenation–dehydrogenation cycles at temperatures reaching 350 °C. Additionally, it was reported in that the hydrogen storage capacity of $\text{La}_{0.5}\text{Ni}_{1.5}\text{Mg}_{17}$ declines progressively with cycling [39-40]. Nanocrystalline Mg_2Ni , synthesized via mechanical alloying of Mg and Mg_2Ni , demonstrates the ability to absorb hydrogen rapidly without requiring prior activation. In contrast, Mg + Mg_2Ni composite materials do require activation; however, once activated, they exhibit faster hydrogen absorption kinetics than pure Mg_2Ni at a relatively low temperature of 150 °C under 12 bar pressure, achieving a high storage capacity of 4.2 wt% [41].

Technologies and methods for obtaining LaNi_5 for use in hydrogen energy

SPS – is a complex multifactorial process, the study of which is at an early stage. Recent literature reviews note that many processes occurring during electric pulse plasma sintering are still not sufficiently clear. As for SPS in the literature, the physical description of SPS is based on the classical sintering theory, however, a number of aspects related to the fact that during high-speed heating the sintering process occurs in significantly nonequilibrium and non-stationary conditions cannot be described on the basis of the classical sintering theory. The main advantages of SPS technology include: – uniform heat distribution over the sample; – high density and controlled porosity; – no need for pre-pressure treatment and binders; – uniform sintering of homogeneous and dissimilar materials; – short working cycle time; – minimal grain growth and effect on the microstructure. The ability to consolidate the material in a short time at relatively low temperatures is a decisive advantage of the technology when working with thermally unstable iron nitrides, ensuring the retention of nitrogen in the material and tight control over grain growth [42]. According to the authors' group of IPS, in order to improve the cyclic stability of La-Mg-Ni alloy for hydrogen storage, the possible sintering temperature of $\text{La}_{0.78}\text{Mg}_{0.22}\text{Ni}_{3.70}$ alloy was investigated by spark plasma sintering (SPS). The fracture morphology and phase structure of the alloy were analyzed by scanning electron microscopy (SEM) and X-ray diffraction (XRD), and the electrochemical characteristics of the alloy were measured by the electrochemical method. The results show that the SPS process consists of expansion and compression stages, and the compression stage lasts longer than the expansion stage, and a higher compressed displacement is observed. The alloy is dense at first, and then loose with the increase of sintering temperature. The alloy consists of LaNi_5 , $(\text{La,Mg})_2\text{Ni}_7$ as the main phases and a small amount of $\text{LaNi}_{2.28}$ phases. The discharge capacity and cyclic stability of SPS alloy at 950 °C are better than those of SPS alloys at 850, 900 and 1000 °C [43].

For instance, in [44], the electrochemical properties of $\text{La}_{0.82}\text{Mg}_{0.18}\text{Ni}_{3.50}\text{Co}_{0.15}$ alloys synthesized via spark plasma sintering (SPS) were investigated using electrochemical

measurements, along with observations of physical parameters and microstructure. The sinterability of $\text{La}_{0.82}\text{Mg}_{0.18}\text{Ni}_{3.50}\text{Co}_{0.13}$ alloys at 900, 950, and 1000 °C is characterized by four stages: initial slight shrinkage, expansion, rapid shrinkage, and minor expansion. The maximum shrinkage displacement increases with increasing sintering temperature. All alloys consist of the $(\text{La}, \text{Mg})_2(\text{Ni}, \text{Co})_7$ phase; in addition, temperatures of 900 and 950 °C are favorable for the formation of the $(\text{La}, \text{Mg})(\text{Ni}, \text{Co})_3$ phase, while the LaNi_5 phase is easily formed in the alloy synthesized by SPS at 1000 °C. Various methods of obtaining LaNi_5 have also been developed. LaNi_5 thin films can also be produced by magnetron ion spraying, powder sintering, composite materials based on intermetallic compounds using powder technology, electrochemical measurements show a significant change in the electrochemical characteristics of alloys associated with an increase in the sintering temperature. LaNi_5 thin films can be produced by magnetron ion spraying [45]. Metal hydrides as multifunctional materials are of particular importance for various hydrogen-related technologies. In addition to the widely used nickel-metal hydride batteries and solid-state storage systems, hydrides have proven their effectiveness in thermosorption compression and heat pump, as sensors and absorbers, in the separation and purification of hydrogen [46]. LaNi_5 and MmNi_5 based alloys have been extensively studied as hydrogen storage materials. These alloys are typically produced by melt casting [47]. This paper presents a study of the activation process of LaNi_5 alloy. To the best of our knowledge, this is the first systematic study on the effects of temperature, constant driving force, and pressure on the activation process [48]. This observation suggests that full activation of LaNi_5 can be achieved after just a single hydrogen absorption–desorption cycle. When LaNi_5 is hydrogenated without prior milling, it takes approximately 50 kiloseconds for complete hydrogen uptake. However, following ball milling in an inert gas atmosphere for only 0.6 kiloseconds, LaNi_5 becomes sufficiently activated. Therefore, ball milling under inert conditions followed by hydrogenation without air exposure proves to be an effective activation method for LaNi_5 [49]. The main factors influencing the performance of hydrogen storage materials include the capacity for hydrogen absorption and release, the thermal stability of the formed hydrides, the kinetics of hydration and dehydration reactions, and their thermodynamic and thermophysical characteristics. Furthermore, the crystal structure and surface-related phenomena, such as segregation and carbonization, are also of significant importance [50].

Conclusion

This paper presents a review of recent studies of the intermetallic compound LaNi_5 , one of the most promising AB_5 -type materials for hydrogen storage. The physicochemical properties of this compound, its ability to form stable reversible hydrides, and current trends in structural modifications to improve hydrogen absorption characteristics are considered.

Particular attention is focused on the effect of mechanical activation on the structural-phase states of LaNi_5 and, consequently, on its hydrogen interaction. Mechanical activation has been found to induce significant changes in the material's microstructure by reducing crystallite size, increasing defect density, and partially amorphizing the material. These changes contribute to the improvement of the kinetics of hydrogen absorption and desorption, as well as to the increase in the specific hydrogen capacity.

Analysis of the latest achievements has shown that the most effective results can be achieved with the complex use of mechanical activation and alloying. Replacing nickel with other elements such as Co, Mn, Al and Fe allows fine-tuning the thermodynamic parameters of interaction with hydrogen and increasing the material's resistance to degradation during repeated cycles.

A review of methods for the synthesis and modification of intermetallic compounds confirmed that modern approaches, including spark plasma sintering and high-energy mechanical milling, are effective tools for improving the performance characteristics.

Thus, the analysis of the work performed shows the prospects of using mechanical activation as a method for increasing the efficiency of LaNi_5 -based materials in hydrogen energy. The data obtained can be used in the development of highly efficient hydrogen storage systems and the creation of materials with specified characteristics.

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This work was carried out within the framework of the implementation of the objectives of the project IRN No. PCF BR21882200 program target financing of the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan.

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МЕХАНИКАЛЫҚ АКТИВТЕНДІРҮДІҢ ҚҰРЫЛЫМДЫҚ-ФАЗАЛЫҚ КҮЙЛЕРГЕ ЖӘНЕ LaNi_5 ИНТЕРМЕТАЛЛ ҚОСЫЛЫСЫНЫҢ СУТЕКТІ СІҢІРУ ҚАСИЕТТЕРІНЕ ӘСЕРІ-ШОЛУ

Мақалада сутегін сақтау үшін қорытпалар мен металлалық қосылыстарды қолдану бойынша бірқатар зерттеулерге шолу берілген. Олардың ішінде интерметаллитті қосылыс LaNi_5 ерекше маңызды орынды алады. АВ₅ түріндегі интерметаллдық қосылыстарды әзірлеу және оңтайландыру саласындағы соңғы жетістіктер талданды. Бұл қосылыстар, атап айтқанда, LaNi_5 және оның легирленген аналогтары элементтерді ауыстыру арқылы олардың қасиеттерін реттеу мүмкіндігіне байланысты кеңінен қолданылады. Сондай-ақ мақалада сутегі технологияларында олардың тиімділігін арттыруға бағытталған АВ₅ қорытпаларын синтездеу және модификациялау әдістеріне шолу берілген. Өндірістің дәстүрлі әдістері де, заманауи технологиялық тәсілдер де, соның ішінде ұшқын плазмасын агломерациялау және механикалық белсендіру қарастырылады. Ғылыми әдебиеттерге шолу көрсеткендей, механикалық активтендіру оның сутегін сіңіру қасиеттерін жақсарту үшін интерметаллитті қосылыстарды өзгертудің тиімді әдісі болып табылады. Бірқатар зерттеулерге сәйкес, жоғары энергиялы шарды ұнтақтаудың әсері материалдың микроқұрылымында айтарлықтай өзгерістерге әкеледі. LaNi_5 -ны сутегі энергетикасында практикалық қолданудың тиімділігін арттыру үшін өңдеу параметрлері, құрылымдық сипаттамалары мен материалдың функционалдық қасиеттері арасындағы байланысты орнату үшін қосымша кешенді зерттеулер қажет. Бұл жұмыстың мақсаты-материалдың микроқұрылымын өзгерту, кристаллит мөлшерін азайту, ақаулардың тығыздығын арттыру және аморфты немесе наноқұрылымды күйлердің пайда болуына ықпал ету үшін

механикалық белсендіруді қолдану, бұл құрылымға және сутегімен өзара әрекеттесуге айтарлықтай әсер етуі мүмкін.

Түйін сөздер: сутекті сақтау, интерметаллитті қосылыстар, гидридтер, механикалық активтендіру.

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ВЛИЯНИЕ МЕХАНИЧЕСКОЙ АКТИВАЦИИ НА СТРУКТУРНО-ФАЗОВЫЕ СОСТОЯНИЯ И ВОДОРОДОПОГЛОЩАТЕЛЬНЫЕ СВОЙСТВА ИНТЕРМЕТАЛЛИЧЕСКОГО СОЕДИНЕНИЯ LaNi_5 – ОБЗОР

В статье представлен обзор ряда исследований по использованию сплавов и интерметаллических соединений для хранения водорода. Среди них особое место занимает межметаллическое соединение LaNi_5 . Проанализированы последние достижения в разработке интерметаллических соединений типа ab_5 . Эти соединения, в частности, LaNi_5 и его легированные аналоги, широко используются благодаря возможности регулировать их свойства путем замены элементов. В статье также представлен обзор методов синтеза и модификации сплавов AB_5 , направленных на повышение их эффективности в водородных технологиях. Рассматриваются как традиционные методы производства, так и современные технологические подходы, включая искровое плазменное спекание и механоактивацию. Обзор научной литературы показал, что механическая активация является эффективным способом модификации интерметаллического соединения LaNi_5 для улучшения его свойств поглощения водорода. Согласно ряду исследований, воздействие высокоэнергетического шарового измельчения приводит к значительным изменениям в микроструктуре материала. Для повышения эффективности практического использования ланидов в водородной энергетике необходимы дополнительные комплексные исследования для установления взаимосвязи между параметрами механической обработки, структурными характеристиками и функциональными свойствами материала. Целью данной работы является использование механической активации для изменения микроструктуры материала, уменьшения размера кристаллитов, увеличения плотности дефектов и стимулирования образования аморфных или наноструктурированных состояний, которые в совокупности могут существенно влиять на структуру и взаимодействие с водородом.

Ключевые слова: хранение водорода, межметаллические соединения, гидриды, механическая активация.

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Received 24.10.2025

Revised 02.11.2025

Accepted 06.11.2025

[https://doi.org/10.53360/2788-7995-2025-4\(20\)-65](https://doi.org/10.53360/2788-7995-2025-4(20)-65)

IRSTI: 30.17.35



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EFFECT OF NANOPARTICLES ON THE COUPLING OF TURBULENCE AND HEAT TRANSFER IN PIPE FLOWS WITH HEAT FLUX

Abstract: Enhancing the efficiency of heat transfer processes remains one of the key challenges in modern energy and thermal engineering. Conventional working fluids, such as water and ethylene glycol, are limited in terms of thermal conductivity and heat capacity, which reduces their potential under high heat flux conditions. One of the promising approaches is the use of nanofluids – suspensions of nanoparticles in a base liquid that can modify its thermophysical properties and improve heat transfer performance.

This study presents a numerical investigation of the flow of water and TiO₂-CuO nanofluid in a U-shaped tube channel under a constant heat flux. Computational fluid dynamics (CFD) was applied to analyze the distribution of turbulent kinetic energy (TKE), pressure variations along the channel, as well as integral heat transfer parameters: the heat transfer coefficient and heat absorption.

The results showed that water exhibits higher turbulent activity, with maximum TKE values reaching $1.9 \cdot 10^{-3} \text{ m}^2/\text{s}^2$, while the overall pressure drop is about 230 Pa. Its relatively low thermal conductivity ($0.6 \text{ W/m}\cdot\text{K}$) leads to a temperature rise of $5\text{--}7^\circ\text{C}$ at the outlet. For the TiO₂-CuO nanofluid, turbulence intensity decreases on straight sections ($10^{-6}\text{--}10^{-5} \text{ m}^2/\text{s}^2$) and the pressure drop increases up to 270 Pa due to higher viscosity. However, improved thermophysical properties – thermal conductivity ($0.702 \text{ W/m}\cdot\text{K}$) and density – ensure more effective heat removal, with outlet overheating reduced to $4\text{--}5^\circ\text{C}$.

Comparative analysis of heat absorption and the heat transfer coefficient revealed the advantage of the nanofluid: $h=68.3 \text{ W}/(\text{m}^2\cdot\text{K})$, $Q=143 \text{ W}$ compared with water ($h=67.6 \text{ W}/(\text{m}^2\cdot\text{K})$, $Q=141.8 \text{ W}$). These results indicate that TiO₂-CuO nanofluid provides higher heat transfer efficiency with an acceptable increase in hydraulic losses, making it a promising coolant for compact and high-load thermal systems.

Key words: nanofluid; TiO₂-CuO; CFD modeling; turbulent kinetic energy; thermophysical properties.

Introduction

Modern heat transfer tasks – from solar energy systems to compact heat exchangers – impose high requirements on the efficiency of working fluids. Conventional liquids such as water, ethylene glycol, and their mixtures are limited in thermal conductivity and heat capacity, which often prevents them from achieving the required heat flux in compact designs. One promising solution to this problem is the use of nanofluids - suspensions of nanoparticles (1-100 nm in size) in a base liquid, which can enhance the thermophysical properties of the system. Research in this field has demonstrated that the addition of nanoparticles can improve thermal conductivity, increase effective heat capacity, and influence heat transfer due to Brownian motion, microscale thermal convection, and other effects [1-6].

Despite numerous experimental and theoretical studies, several key issues and contradictions remain:

1 Ambiguity of turbulence influence. Many studies emphasize the intensification of turbulent pulsations kinetic energy (TKE) as one of the main mechanisms of enhanced convective heat