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METAL HYDRIDES: PROPERTIES AND PRACTICAL APPLICATIONS. REVIEW OF THE WORKS IN CIS-COUNTRIES

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Abstract—A short review of R&D in the field of hydrogen hydride technologies in Russia and CIS countries is presented. As a result of basic research of physical and chemical features of intermetallic alloys and their hydrides, their structural peculiarities, absorption kinetics, thermal processes, etc., methods have been developed for creation high efficient alloys for different applications in metal hydride technology. Original devices such as hydrogen accumulators, thermal compressors, heat transformation systems and other experimental devices with unique characteristics have been created. The results of reviewed R&D demonstrate high efficiency of metal hydride technologies. These investigations and developments give reliable scientific and technical background for the development of the new research projects in the framework of the new Russian program of R&D in hydrogen energy and technology and for international cooperation. © 1998 International Association for Hydrogen Energy

1. INTRODUCTION

R&D in the field of hydrogen hydride technologies in Russia and CIS-countries are being conducted since the early 70s. The reviews of the research done in the USSR up to the middle of 80s are presented in [1–5]. The following review covers mainly the works carried out in the last 15 years in more than 20 laboratories. During this period, more than 200 papers were published by the authors from the CIS countries, and of course it is not possible to present these works in full. Due to the limited volume of the review, we will briefly describe the contents and the main results of the research that are most important from our point of view. However, we give rather detailed references in order to compensate to some extent the brevity of the review.

2. BASIC RESEARCH

2.1. *Interaction with hydrogen, sorption characteristics, thermodynamics of alloys, structural investigations, electronic properties*

The development of principles of metal–hydride technology requires a detailed study of hydrogen interaction with metals and alloys in wide ranges of pressure and temperature, investigation of factors controlling the kinetics and thermodynamics of hydride formation, and analysis of crystal structure of hydrides. A physicochemical investigation of metal–hydrogen systems using X-ray, neutron diffraction, DTA, microscopy, electroconductivity and magnetic measurements, calorimetry and isotope exchange methods has been done for the solution of these problems.

Thermodynamic investigation of the reaction of hydrogen with intermetallic compounds (IMC) of different structures types (RT, RT_2, RT_3, RT_5 where R—rare-earth, Ti, Zr; T—transition metal) using methods of physicochemical analysis, has allowed to develop the knowledge of the mechanism of hydrogen interaction with metal matrix, the hysteresis phenomenon, and to propose the models for calculation of hydride formation enthalpies [6–20]. Calorimetric measurements of RT_5–H_2 and RT_2–H_2 systems have disclosed the existence of intermediate hydride phases in some systems and states with a pronounced dependence of absorption (desorption) reaction enthalpy on temperature.

It has been stated that the absorption of hydrogen can proceed in several different paths and that the reversible absorption–desorption reaction is not the only possibility. Actually for a number of IMC, especially at high temperatures and large hydrogen pressures, the reaction of hydrogenolysis (disproportionation) of metallic matrix due to hydrogen influence becomes thermodynamically

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preferable [21–24]. In some cases, the hydrogenolysis reaction is preceded by a stage of intermetallic hydride amorphisation with the formation of metastable products with high hydrogen content.

The investigation of hydrogen absorption kinetics by alloys of $LaNi_5$, $Ti(Zr)Cr_2$, FeTi type allows to propose the following mechanism: at the initial stage, the reaction rate is described by a "contracting sphere" model and limiting stage is the rate of hydride phase nucleation. At the final stage of reaction, the limiting process is the rate of hydrogen diffusion through the hydride layers [25–31].

A great importance for the development of the conception of hydride structure and of the metal–hydrogen bonding nature involve the works on neutron diffraction investigation and description of hydride crystal structure [32–38], magnetic measurements [39–45] and X-ray emission spectroscopy [46–47]. In these works the crystal, magnetic and electron structure of several intermetallic hydrides have been described for the first time.

Rather detailed study of the interaction of hydrogen with multicomponent alloys of RT_5 and RT_3 types has been performed in the works [48–50]. The enthalpies of reaction, the influence of different constituents of the alloys on the properties of hydrides have been stated.

The investigation of hydrogen interaction with alloys of Ti-V–M (M—Al, Fe, Co, Ni) systems [51, 52] provided for the first time to obtain and to discuss the properties of ternary hydrides and to determine the character of hydriding reactions of alloys belonging to 2- and 3-phase regions of metallic system states.

Based on the results of these studies, the model for "design" of compositions of alloys with pre-set hydride dissociation pressures has been proposed. Table 1 shows the characteristics of three groups of alloys which reversibly absorb considerable amounts of hydrogen [53–56]. The P–C-isotherms for some selected systems are shown in Figs 1–3.

A great deal of consideration has been given to interaction with hydrogen of some magnesium IMC and alloys, mainly due to their large hydrogen absorption capacity (up to 7.6 mass.% H_2), and to the fact that these compounds seem to be promising for preparing high-temperature hydrogen storage systems. The main obstacle for magnesium hydride application is the extremely low rate of interaction with hydrogen even in view of various methods of metal activation [57–61]. Most effective appears to be the methods of magnesium alloying with rare-earth metals, calcium and nickel. To determine the hydride formation mechanism and to optimise the conditions of synthesis, the interaction of hydrogen with IMC, solid solutions and multiphase alloys in the Mg–Ln(La, Ce, Er, Yb), Mg–Ca–M(Al, Zn, Cu, Ce, Ni), Mg–Ln(La, Ce, Sc, Y, Mn)–M(Al, Ni) systems has been investigated [62–75]. The role of phase composition and alloy microstructure in hydriding reactions has been studied. Based on the kinetic measurements, the values of activation energy of hydriding–dehydriding reactions in multicomponent magnesium-based systems have been calculated. It has been shown that doping of magnesium by small amounts of nickel and rare-earth metals allows to increase the rate of magnesium hydride nucleation by several times: 90% of transformation takes place in the period of 1–2 min at 300°C (Fig. 4). A model for reaction of MgH_2 formation in the presence of rare-earth metals has been proposed. It is based on the consideration that lanthanide hydride inclusions act as active conductors of dissociated hydrogen to the magnesium surface and also as magnesium hydride nucleation centres (Fig. 5) [75].

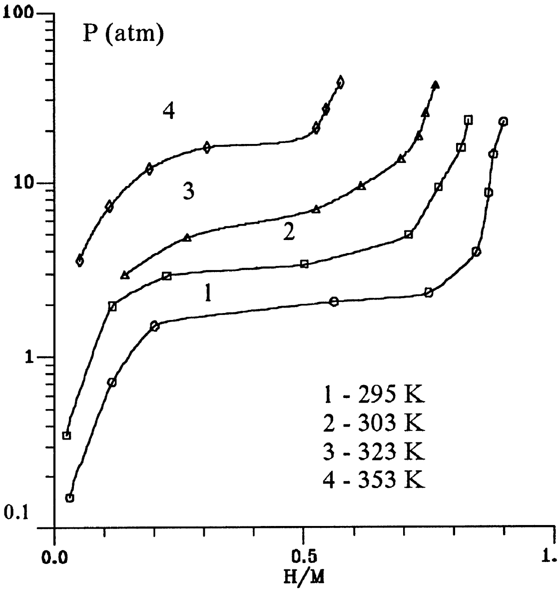

Fig. 1. Desorption isotherms for the $Ti_{0.48}Fe_{0.47}V_{0.025}Mn_{0.025}$–$H_2$ system.

Table 1. Characteristics of hydrogen storage alloys

Alloy type	Substituents	Absorption–desorption temperature range °C	Mass % H_2
FeTi	V, Mn	20–100	1.7
Ti–V–M	Al, Fe, Co, Ni	200–400	2.0–3.8
$Ti_xZt_{1-x}MnCr$	V, Fe, Co, Ni	0–200	1.7–2.0
$MmNi_5$	Co, Fe, Mn, Al, Cu, La, Ce	0–100	1.3–1.5

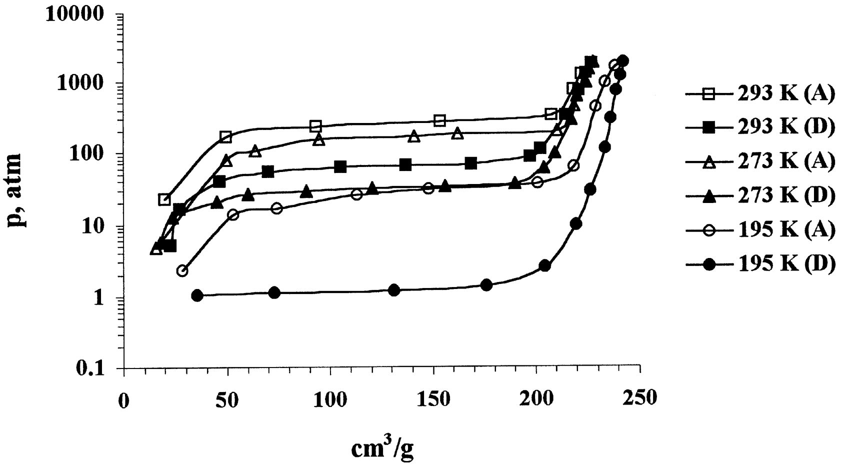

Fig. 2. Absorption–desorption isotherms for the $Ti_{0.9}Zr_{0.1}Mn_{1.4}Cr_{0.45}Fe_{0.15}$–$H_2$ system.

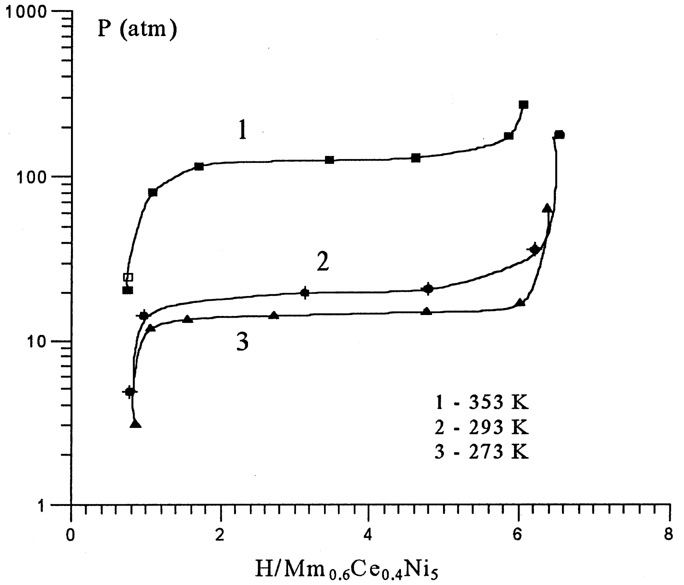

Fig. 3. Desorption isotherms for the $Mm_{0.6}Ce_{0.4}Ni_5$–H_2 system.

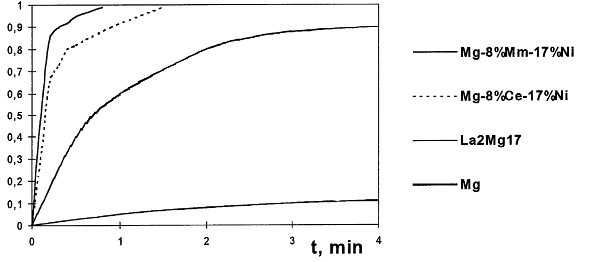

Fig. 4. Kinetic curves of hydrogen absorption for Mg-based alloys at 300°C and 1 MPa.

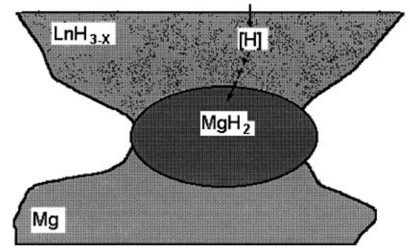

Fig. 5. Scheme of magnesium hydride formation in the presence of lanthanide hydride.

Investigations at high gaseous and quasihydrostatic pressures have resulted in synthesis of new binary metal hydrides and hydrides based on IMC, and descriptions of their transformations [76–84]. The phenomenon of hydride phase stabilisation, while hydrogen absorption at high pressures for $TiCr_2$–H_2 and RNi_2–H_2 systems, is also presented.

The study of decrepitation of alloys during hydrogen absorption–desorption cycles have been instrumental to explain the processes of metal matrix destruction, to obtain the quantitative data on the dependence of particle size on the created parameters, and to propose an empirical formula for the calculation of the degree of decrepitation [85, 86].

A large amount of data on physicochemical properties of hydrides and hydrogen absorbing alloys is collected in several databooks [87, 88].

2.2. *Catalysts and electrocatalysts*

Much attention is paid to the development of the physicochemical principles of creation of catalysts based on intermetallic hydrides. It has been shown that the unique property of IMC- and hydride-based catalysts to activate hydrogen, including its dissolution in crystal lattice of catalyst, favours the process of heterophase exchange of hydrogen between gaseous and solid phases in catalytic conditions and maintains the high stability of catalytic action. The main principles of controlling the catalytic properties of such systems have been formulated. The wide possibility of use of these phenomena for the preparation of effective and selective catalysts for different reactions of hydrocarbons, synthesis of methanol and of higher alcohols from carbon monoxide and hydrogen, utilisation of highly toxic nitrogen-containing organic compounds, in particular, residuals of rocket fuel based on dimethilhydrasine (in co-operation with NPO "Salut") have been shown. The mechanism of catalytic action of intermetallic hydrides in the reaction of hydrogenation of unsaturated compounds in different solvents has been studied. The optimal conditions for liquid phase hydrogenation of compounds with unsaturated bond and of nitrogen containing aromatic compounds have been determined [86–93].

The absorption and electrochemical properties of IMC $CeNi_{3-x}Co_x$ and $YNi_{2.5}M_{0.5}$($M = Al, Fe, Cr, Cu, Co, Mn$) have been studied for the first time. It has been shown that the $CeNi_3$ system possesses a good reversibility and stability during cycling processes in alkali solutions. The thermodynamic parameters of the reaction with hydrogen and the diffusion coefficients of hydrogen in different IMC have been determined. A high electrocatalytic activity of $CeNi_3$ in the reaction of hydrogen evolvement has been noted.

A comparative investigation of electrode materials based on AB_5-type IMC: $LaNi_5$, $LaNi_{2.5}Co_{2.5}$, $LaNi_{2.5}Co_{2.4}Mn_{0.1}$, $La_{0.8}Ce_{0.2}Ni_2Co_3$, $La_{0.7}Nb_{0.3}Ni_{2.5}Co_{2.4}Cr_{0.1}$, $MmNi_{3.5}Co_{0.7}Al_{0.8}$, $Mm_{0.5}La_{0.5}Ni_2Co_3$ prepared by compacting with copper or PTFE binding have been done. It has been shown that the IMC hydride, stable at normal pressure and ambient temperature in alkali solutions, contain 4.1–2.1 H/f.u. and the equilibrium dissociation pressure is below the atmospheric one.

A new effective method of electrode material activation has been proposed. The main feature of the method is the combination of potentiodynamic and potentiostatic modes of electrode charge-discharge cycling.

While studying the porosity of electrode materials based on IMC with polymer binding, it has been found that during cathode polarisation—due to the intensive hydrogen desorption in bulk material—there arises a pressure gap and the electrolyte fills partly hydrophobic pores. This leads to the increase of the effective electroconductivity of material [94–98].

A model for calculation of galvanostatic charge-discharge curves for separate spherical grain of hydrogen absorbing metal has been proposed [97]. Assuming that electrode potential is the monovariant function of the absorbed hydrogen quantity, the model allows to achieve semiquantitative correlation of experimental and calculated charge-discharge curves for AB_2-type intermetallic electrodes by varying the adsorption equilibrium constant.

2.3. *Hydrogen activation*

The peculiarities of processes of electrotransfer in hydrogen, desorbed from hydrides, points to the fact that this very hydrogen is much more exposed to ionisation, dissociation and excitation than molecular hydrogen [99–101]. The effect of activation of hydrogen by hydrides stimulates the low potential barrier of hydrogen transfer from metal matrix to ions, which makes it possible to use hydrogen in several energy devices [102–104].

2.4. *Thermophysical processes*

The peculiarities of heat- and mass transfer in metal hydride porous beds as well as thermophysical properties of Me–H systems have been investigated theoretically and experimentally [105–117]. On this basis, several mathematical models have been developed for calculation of thermophysical processes in metal–hydride porous beds and elements of metal hydride accumulators of hydrogen for different applications [118–126].

3. APPLICATIONS

3.1. *Alloys production*

Research and design in the field of hydrogen-absorbing alloys and the co-operation of scientific centres with industry resulted in the pilot alloy production in Russia and Ukraine. this concerns first of all the alloys based on FeTi, $La_{1-x}Ln_xT_y$, $Mm_{1-x}Ln_xT_y$ (T—Al, Co, Fe, Mn, Cr, Cu), $Ti_{1-x}Zr_x(Mn, Cr)_2$ for the metal–hydride technology and for the production of fuel cells [56, 127–135]. The technical documentation on alloys and the technology of their production have been worked out. This technology utilises the conventional induction and skull melting methods as well as powder metallurgy [128], and a new method of direct reduction of fluorides in the self-propagating high-temperature synthesis [135]. The alloys obtained using the developed technologies do not yield the best Western results. The dissociation pressures range from parts of atmosphere to several tens atmospheres at temperatures from 0–100°C.

3.2. *Accumulation of hydrogen*

The realisation of metal–hydride method of hydrogen accumulation is implemented mainly in two directions. First is the development of diminutive (small) storage systems for R&D laboratories and for apparatuses consuming small amounts of hydrogen. Second is the development of storage systems for vehicular applications and energy devices. Laboratory accumulator is usually a cylindrical vessel filled with alloys based on $LaNi_5$, $MmNi_5$ or $(Ti, Zr)(Mn, Cr)_2$ with a capacity of 300–1000 l of hydrogen. The working pressure in such accumulators is about 0.5–5.0 atm in the temperature range 20–100°C [102, 136–141].

A series of laboratory storage systems for different applications has been developed at the Institute for Problems of Mechanical Engineering of Ukrainian National Academy of Sciences. Their characteristics are presented in Table. 2.

Accumulators for vehicular and energy applications can store about 2–20 $STPm^3$ hydrogen and are rather complex devices, equipped with heat-exchangers with hot (cold) water or gas inlets and outlets [1, 5, 142–148]. A rather original solution of the problem of controlling the content of hydrogen in the accumulator is the transducer, described in [149].

The trend of the storage system development is the creation of module systems and maintenance of needed hydrogen capacity by a certain number of modules [56]. The characteristics of such modules are presented in Table 3.

Table 4 presents the characteristics of storage systems developed in the Institute for Problems of Mechanical Engineering of Ukrainian National Academy of Sciences. These accumulators are also designed according to module technology and can be easily redesigned to fit preset conditions, since they can meet different hydrogen application functions.

3.3. *Energy transforming systems*

The characteristics of thermosorption compressors (TSC), designed and constructed in the Institute for problems of Mechanical Engineering of Ukrainian National Academy of Sciences are presented in Table 5. These devices do not yield the same results as those of foreign manufacturers in specific features (energy consumption for compression, metal consumption, dimensions). Low productivity TSCs outrun the conventional mechanical compressors of hydrogen in their functional ability. They are widely used as the sources of pure hydrogen in microcryogenic apparatuses and installations for testing of metals in hydrogen atmosphere, etc. [150–154].

Much attention has been paid to the utilisation of metal hydrides in the energy accumulating and transforming systems. The different types of hydrogen accumulators

Table 2. Characteristics of laboratory hydrogen accumulators

N	Device	Hydrogen capacity kg	Working pressure MPa	Outlet flow rate g/s	Precision of Pressure maintenance %	Impurities vol.%	Dimensions mm	Mass kg
1	Laboratory accumulator “Omega-10”	0.18	0.1–10.0	2.0×10^{-1}	±10	10^{-2}	490 × 530 × 120	40
2	Sorption chamber for inlet of deuterium	1.95	0.1–10.0	2.6×10^{-1}	±10	10^{-2}	∅400 × 600	110
3	Hydrogen storage and inlet system	1.7	0.2–2.0	2.4×10^{-1}	±20	10^{-2}	∅1320 × 1550	260
4	Hydrogen accumulator for masers	0.0026	0.12	8.0×10^{-9}	±20	10^{-4}	∅40 × 100	0.3
5	Hydrogen generator	0.0036	0.15–1.0	$(0–5) \times 10^{-2}$	±5	10^{-2}	165 × 65 × 65	0.8
6	Hydrogen storage purification and programmable inlet system	0.0063	0.01–0.3	$(0–2) \times 10^{-4}$	±1	10^{-3}	490 × 530 × 120 (two modules)	30
7	Gas inlet system	0.0045	0.05–0.6	2.0×10^{-2}	±5	10^{-3}	160 × 135 × 120	1.6
8	Hydrogen accumulator A-1,5	0.0018	0.15–0.2	5.0×10^{-3}	±5	10^{-3}	∅80 × 138	1.2
9	Metal-hydride storage, purification and inlet system for Tokomak	0.018	0.05–0.4	$(0–5) \times 10^{-4}$	±5	10^{-3}	∅50 × 440	3.5
10	Laboratory hydrogen accumulator	0.081	0.15–15.0			10^{-2}	∅126 × 780	23.4

Table 3. Technical characteristics of the typical metal–hydride accumulator module

Hydrogen outlet pressure in discharge mode (MPa)	0.5–5.5
Module working hydrogen capacity	
absolute (STPm^3)	9.4
specific mass (%)	1.13
specific volume (STPm^3/m^3	620
Purity of discharged hydrogen (%)	$\geqslant$99.999
Maximum stationary flow rate in discharge mode (l/s)	$\geqslant$20
Working temperature range (°C)	0–32
Hydrogen pressure in charge mode (MPa)	35
Charging time (h)	$\leqslant$3
Guaranteed number of chargings (without loss of capacity)	$\geqslant$150
Module mass (kg)	74
Alloy mass in module (kg)	58
Alloy type	Mm-Ce(La)–Ni–Fe
Module dimensions (mm)	
diameter	75
length	3870

for the chain: Energy source–Electrolyser—Hydrogen accumulator—Electrogenerator (sun, wind) have been discussed and investigated [1, 5, 126, 144, 145, 155–160]. The working principle of such systems is in the transformation of electric power, produced by wind or solar batteries, into chemical energy accumulator—hydrogen, with its consequent utilisation as an energy carrier. The calculations and experimental studies show the perspective of such schemes for autonomous devices. A unique feature of the metal–hydride systems is the possibility of their utilisation not only as a hydrogen accumulator, but also as a heat accumulator and heat pump in the energy devices [126, 144]. This allows the design of fully autonomous systems of energy maintenance for final users situated in hard-to-reach regions and to provide absolute ecological compatibility of processes of production and utilisation of energy.

3.4. *Hydride decrepitation*

The property of IMC and alloys to disintegrate during the hydrogen absorption–desorption cycling is the ground for the development of methods of hydride decrepitation, being used in different industrial fields. A new approach has been developed in full for the production of constant magnets and Ti-based alloys. The main feature of hydride decrepitation of Sm–Co-, Nd–Fe–B- and R_2Fe_{17}-type alloys is the production of powders with well developed nonoxidised surface free of "naklep" and plastic deformation effects [161–165]. This results in the considerable improvement of characteristics of constant magnets, produced from powders prepared using this technology. It has been stated that preliminary activation by hydride decrepitation of R_2Fe_{17}-type alloys allows to conduct the consequent process of nitrogenation for the production of $R_2Fe_{17}N_3$ materials in considerably milder conditions [166].

The problems of practical application of consequences of hydrogen treatment of titanium alloys and hydride decrepitation method for embrittling of titanium alloys for the production of powders have been described in [167]. The studies allow us to effectively determine the groups of alloys and the conditions of their treatment for the utilisation in metallurgical and mechanical engineering industries.

3.5. *Purification and activation of hydrogen*

Studies in the field of membrane purification of hydrogen and investigations of the hydrogen influence on

Table 4. Characteristics of hydrogen storage systems

N	Device	Hydrogen capacity kg	Working pressure MPa	Outlet flow rate g/s	Precision of Pressure maintenance %	Impurities vol.%	Dimensions mm	Mass kg
1	Hydrogen accumulator for GAZ-24	2.1	0.1–0.5	1.8×10^{-1}	±20	10^{-2}	∅390 × 1430	250
2	Hydrogen accumulator for RAF-2203	2.2	0.1–0.5	6.7×10^{-1}	±20	10^{-2}	322 × 375 × 1165	245
3	Hydrogen accumulator for forklift 4091	2.4	0.1–0.3	2.2×10^{-1}	±20	10^{-2}	460 × 640 × 1060	496
4	Feeding systems for vehicular hydrogen accumulators	0.061	0.5–2.5	5.0×10^{-3}	±10	10^{-2}	490 × 530 × 120	25
5	Transportable hydrogen source	0.0015	0.15–0.5	$(0–5) \times 10^{-3}$	±5	10^{-4}	180 × 80 × 100	1.5
6	Hydrogen accumulator for autonomous gas-turbine device	3.5	0.1–0.4	8–16		10^{-3}	1060 × 770 × 2050	3120

Table 5. Characteristics of thermosorption compressors

	TSC-6.0-0.4	TSC-X	TSC-70	TSC-150-2.5	TSC-300-2.5	TSC-80	TSC-150-10.0
Suck pressure MPa	0.4	0.6–1.0	0.5–0.8	1.0	1.0	0.3–0.5	0.3–0.5
Force pressure MPa	6.0	5–10	5–7	15	30	5–8	15.0
Productivity g/s (10^{-3}	56	25–30	38–44	35	18	36–41	250
Heating source				electric			
Total consumed power kW	1.3	2	3.6	3	8	3.2	27
Cooling source		water		natural convection	forced air	water	forced air
Number of modules	3	2	3	3	7	4	60
IMC	TiFe			$LaNi_5$		$(La,Ce)(Ni,Al)_5$	$LaNi_5$ $La(Ni,Mn)_5$
System volume dm^3	20	90	85	106	170	98	2200
Mass kg	18	72	64	46	142	47	660

properties of materials are presented to the full in the works of the Donezk Polytechnic Institute [168–177]. The studies on the creation of hydrogen diffusion filters have led to implementing their industrial production.

Original equipment for cyclic absorption–desorption processes for hydrogen purification and compression from hydrogen-containing gases of nitrogen and petrochemical industry are described in [178, 179]. Basing on the results obtained, the pilot installation for TSC–hydrogen separation has been worked out and is under testing at the Pilot Factory of State Institute for Nitrogen Industry (Vidnoye, Moscow region).

The use of metal–hydride systems of hydrogen filling allows us to increase the effectivity of physico-energetical devices. Preliminary tests have shown [104] that the addition of such devices to the gas-discharge chamber of ion-sources, increases the output of ions in the H^+-generation mode by 30–50% and in H^--generation mode by 15–20%.

3.6. *Fuel cells and batteries*

Now the germetic disk and cylindrical Ni-MH batteries are being produced in Russia at several enterprises. They have capacities 1.5–2 times higher compared to analogous (in type and dimensions) Ni–Cd batteries. They are reserved for the direct current feeding of transmitters, radio-receivers, measuring devices, medical equipment, shavers, home audio-video, computer games for children, etc. Their characteristics are presented in Table 6.

All types of batteries having the maximum discharge current value of 4 C_H, have rather low value of self-discharge and are operational in the temperature range $-10-+50°C$ for 300–400 cycles. Investigations aimed at the creation of Ni–MH batteries have also been started in Ukraine.

Familiarisation with the production of Ni–MH batteries has begun at NIAI (St.-Peterburg), "Accumulator" Joint Stock Company (Kursk), and "Autonomous Current Sources" (Saratov).

3.7. *Safety maintenance of nuclear energy devices*

The use of hydrides of lithium, zirconium, titanium and other metals as radiation and biological shields has been a common practice for many years. A production of hydride retarding blocks based on above mentioned materials has been launched in Russia recently. The problem of holding hydrogen in these materials for 3–5 years

Table 6. Characteristics of Ni-MH batteries

Analogue	Type	Capacity C_H Ah	Voltage V	Recommended discharge current, A	Diameter mm	Height mm	Mass g
D-0.1	NMVD-0.16D	0.16	1.2	0.032	20.0	6.6	6.7
D-0.26	NMVD-0.40C	0.40		0.080	25.2	9.2	15.0
D-0.55	NMVD-0.80	0.80		0.160	34.6	9.8	30.0
RKGZ-0.5	NMVZ-0.95	0.95		0.190	14.0	50.0	28.0

has also been solved in State Scientific Centre "A.A.Bochvar Institute of Inorganic Materials".

4. RESUME

The R&D in the field of metal hydride hydrogen technologies conducted in Russia and CIS countries up to now, give reliable scientific and technical background for creating the new types of current sources and energy transformation and storage systems based on the metal hydrides. Their further development within the Russian R&D program in the field of hydrogen energy and technology will allow us to solve some important problems of autonomous power production, ecologically clean transport and others. Solving these problems could be the subject of efficient international cooperation in the field of hydrogen energy.

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