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Study of the Process of Hydrogen Accumulation in Nickel-Cadmium Batteries Taking into Account the Service Life

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Abstract: This research addresses a critical aspect of battery management and automation in energy storage systems: hydrogen accumulation in nickel-cadmium batteries during operational cycles. From an automation perspective, understanding hydrogen generation mechanisms is crucial for developing intelligent battery monitoring and control systems. The study employs advanced thermal decomposition techniques and volumetric gas analysis to quantify hydrogen accumulation in battery electrodes over time. By systematically investigating KSL-15 and KPL-14 batteries with a service life of 0 to 10 years, we found a direct proportional relationship between hydrogen volume and service life, and identified where in the electrode hydrogen accumulates. Key findings have significant implications for automated battery management technologies. The research demonstrates that hydrogen accumulates predominantly in cadmium and nickel oxide electrodes, with volumes reaching up to 290 liters in specific battery configurations. This knowledge enables the development of more sophisticated automated monitoring algorithms that can predict battery degradation and potential safety risks. The study's methodology provides a framework for integrating real-time hydrogen detection and measurement into battery management systems, potentially enhancing predictive maintenance strategies and improving overall energy storage system reliability through advanced technological process automation.

Keywords: Nickel-cadmium, Hydrogen accumulation, Service life

Introduction

Technological process automation in battery systems involves sophisticated monitoring of critical parameters such as hydrogen accumulation, charge-discharge cycles, and thermal characteristics. Understanding the intricate mechanisms of hydrogen generation and accumulation within battery electrodes is paramount for developing intelligent battery management systems. These automated systems can potentially predict battery degradation, prevent thermal runaway, and optimize charging protocols.

A significant operational challenge associated with batteries is the phenomenon of aging, which is closely linked to electrolyte degradation and gas evolution. Several factors can contribute to the occurrence of thermal runaway in batteries, including overcharging, short circuits, manufacturing defects, and physical damage to the battery. When a battery overcharged, the voltage at the electrodes increases, leading to the production of excess heat. This can cause the electrolyte in the battery to decompose, releasing gases that can lead to further heating

and potentially cause the battery to explode (Galushkin et al., 2015). Short circuits can also cause thermal runaway by creating a path for a large current to flow through the battery, leading to the rapid generation of heat. Manufacturing defects, such as contamination of the electrolyte or improper sealing of the battery cells, can also contribute to thermal runaway.

Once thermal runaway begins, it can be difficult to stop or control. The heat generated can lead to a chain reaction, as the increased temperature causes more heat production, leading to a rapid and uncontrolled increase in temperature. This can ultimately result in the battery exploding and potentially causing damage to the surrounding environment.

There are very few studies of this phenomenon in the literature on this topic. In our opinion, the lack of studies of thermal runaway of alkaline batteries can be explained by the following reasons. There are many hypotheses about how thermal runaway occurs in batteries, including chemical reactions inside the battery, uneven charge distribution, overheating, and other factors. However, despite the various theories, the exact mechanism of thermal runaway is still a subject of research. The authors of these scientific studies did not consider it appropriate to provide direct evidence of the mechanism under consideration based on experimental data.

The studies presented in (Abada et al., 2016; Feng et al., 2014; Kim et al., 2014; Lee et al., 2015; Popov et al., 2021; Ren et al., 2014; Rezvanizaniani et al., 2014) demonstrated that the uncontrolled process of thermal instability in nickel-cadmium batteries is accompanied by the active release of previously accumulated hydrogen. Experimental studies (Fleischhammer et al., 2015; Friesen et al., 2016; Grutzke et al., 2015; Kraft et al., 2015) devoted to the thermal destruction of nickel-cadmium battery electrode samples revealed that the hydrogen recorded during these experiments was in the electrodes even before the onset of the thermal destruction process. The present study is aimed at a quantitative analysis of the hydrogen content that accumulates in the electrodes of nickel-cadmium battery cells during their service life.

Possible sources of hydrogen formation in electrode materials of nickel-cadmium batteries are analyzed. The first possibility arises from various technological processes employed in the production of these battery electrodes. According to the second hypothesis, hydrogen accumulation can occur during battery discharges throughout the charge-discharge cycles (Li et al., 2014; Park et al., 2015). Consequently, it is hypothesized that hydrogen accumulation in the electrodes of the studied nickel-cadmium batteries is a gradual process resulting from extended battery operation. Therefore, the volume of accumulated hydrogen in the electrodes should be proportional to the battery's service life. As an example, consider a KSL-15 battery, which operates for 6 hours with a charging current of 3.8 A. This results in an overcharge of 1.52 times the battery's nominal capacity. Consequently, the overcharging of the KSL-15 battery (with a nominal capacity of 7.8 Ah) leads to the decomposition of electrolyte water, releasing hydrogen and oxygen.

The aforementioned calculation illustrates the potential for hydrogen accumulation in the electrodes of nickel-cadmium batteries with varying service life. Accordingly, it is necessary to determine the relationship between the accumulated hydrogen volume in the electrodes and the battery's operational duration. The main objective of this study is to analyze the correlation between the volume of hydrogen accumulated in the electrodes of nickel-cadmium batteries and their service life, as well as to determine the location of hydrogen accumulation using the example of KSL-15 batteries.

Materials and Methods

Experimental studies were performed on KSL-15 batteries with a service life of 0 to 7 years. In addition, KPL-14 batteries with a service life of 0 to 10 years were also used in the study. The alkaline nickel-cadmium battery consists of positive nickel oxide and negative cadmium electrodes separated by plastic separators, which ensure a stable interelectrode gap and free circulation of the electrolyte, (Figure 1).

Structurally, KSL-15 batteries are blocks of positive and negative electrodes placed in a case, where the negative terminal of the battery terminal was connected to the case, and the positive one to a terminal insulated from the case. The battery design includes metal-ceramic positive electrodes and pressed negative electrodes. A fibrous polypropylene separator provides separation. The active materials are: for the positive electrode - nickel hydroxide $Ni(OH)_2$, for the negative - cadmium hydroxide $Cd(OH)_2$. The electrolytic medium is represented by a solution of technical potassium hydroxide with a density of 1.22 g/cm³ with the inclusion of technical lithium hydroxide. The fundamental reactions occurring in the battery are reflected by the equation (1):

$$2Ni(OH)_2 + Cd(OH)_2 \underset{\text{discharge}}{\overset{\text{charge}}{\rightleftarrows}} 2NiOOH + Cd + 2H_2O \quad (1)$$

During the process of full charging of the battery under study, hydrogen is released at the negative electrode in accordance with the reaction (2):

$$2H_2O + 2\ e \rightarrow 2OH^- + H_2 \quad (2)$$

As the positive electrodes charge during the NiOOH formation process, oxygen is released, with its quantity increasing towards the final stage of battery charging in accordance with the reaction (3):

$$4OH^- - 4\ e \rightarrow 2H_2O + O_2 \quad (3)$$

The oxygen that is released oxidizes the negative electrodes according to the reaction (4):

$$2Cd + O_2 + 2H_2O \rightarrow 2Cd(OH)_2 \quad (4)$$

As a result, the pressure inside the accumulator does not exceed the permissible limits.

The battery KPL-14 is an electrode block consisting of positive and negative plate electrodes separated from each other by a frame plastic separator. The electrode block is placed in a rectangular tank and insulated from it by vinyl plastic gaskets. The battery cover is welded to the tank along the perimeter. Under normal operating conditions, the batteries use an electrolyte - potassium oxide hydrate with a density of 1.19 g/cm^3 with the addition of (20±1) g/l lithium oxide hydrate; at negative temperatures - potassium oxide hydrate with a density of 1.26 g/cm^3 without the addition of lithium oxide hydrate.

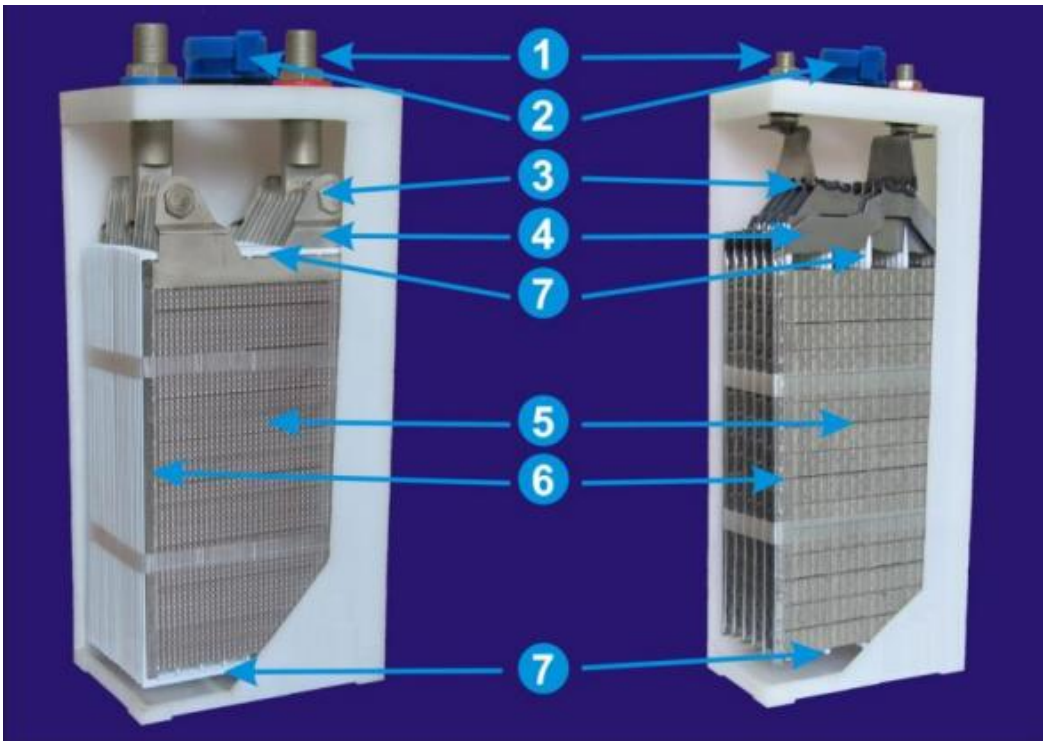

Figure 1. Construction of alkaline nickel-cadmium battery: 1. Current collector - acts as a current collector element and serves as a contact unit for the inter-battery connection; 2. Plug - is designed for convenient filling of the tank with electrolyte, unimpeded gas removal during charging and preventing electrolyte splashing and the release of aerosol particles; 3. Electrode connection - combines the electrodes into a single system and transmits electric current from the electrodes to the current terminal (conductor); 4. Contact strips - are fixed to the electrode by welding and carry out the current drain from the electrode elements; 5.Electrode; 6. Rib - gives the electrode mechanical strength and ensures conductive connection with the contact strip; 7. Separator - isolates the positive electrode from the negative and creates conditions for the free movement of electrolyte in the interelectrode space

The hermetically sealed thermal chamber for the thermal decomposition of the electrodes consists of a cylinder 1.8 m long and 2 cm in diameter. One end of the cylindrical apparatus is sealed, while a rubber stopper equipped with a gas outlet tube is inserted into the arcuate end. The sealed end is positioned within a muffle furnace. The gas generated during heating within the thermal chamber is directed into a measurement container after passing through a standardized coil. For experiments involving the thermal decomposition of battery electrodes, the electrodes are placed in a specialized cartridge, which is then inserted into the thermal chamber.

This configuration facilitates the convenient removal of the test electrode from the thermal chamber following thermal decomposition, which occurs at a temperature of 800 °C. The temperature for thermal decomposition was determined experimentally. It was established that substantial gas emission occurs from both electrode types when the temperature exceeds 740 °C. Consequently, an optimal temperature of 800 °C was selected for the experiment.

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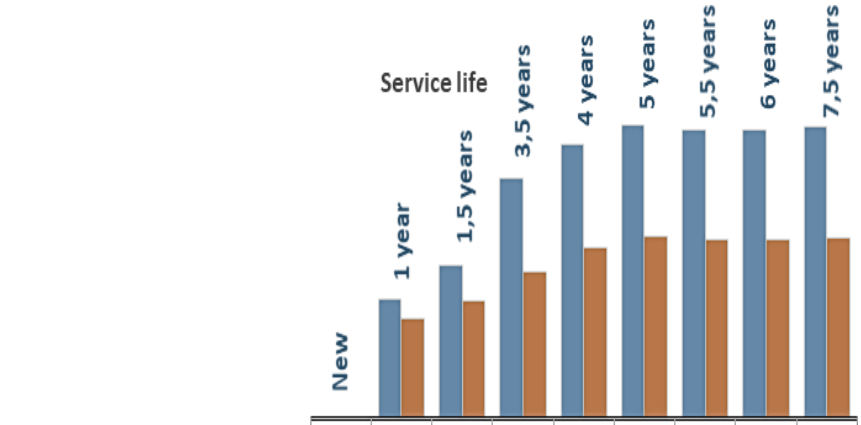
During the experiment, each electrode underwent thermal decomposition. For this purpose, the electrode was rolled into a cylindrical shape and placed in a cartridge, which was then inserted into a thermal chamber. The thermal decomposition process was terminated when the volume of the gas mixture released per day was less than 100 milliliters, in accordance with the experimental conditions. Thermal decomposition of the nickel oxide electrode was carried out for two weeks, eleven hours a day. Thus, on the first day of the experiment, 5.5 liters of gas were extracted from the nickel oxide electrode. 3.5 liters of gas were released from the cadmium electrode during the first day. Theoretically, the following substances can be released as result of thermal runaway: water vapor, hydrogen and oxygen due to the decomposition of water and oxides in the electrodes, as well as separator combustion products. The gas accumulator was cooled and the water vapor was separated. To determine the quantitative composition of the released gas, the VOG-2M volumetric optical gas analyzer was used in the study. It is capable of determining the content of carbon dioxide, oxygen and carbon monoxide using the gas volume method, and when determining methane, the device operates by measuring the shift of the interference pattern. It consists of a burette, an absorption vessel, a pressure vessel for the sealing liquid, a pressure vessel for water, a vessel, a distribution tap, a tube «Heat Pipe», hopcalite, silica gel, a tube for activated carbon, a pressure gauge, a burette with a capacity of 275 ml. The results of the experimental studies are presented in Figures 1 and 2 for the KSL-15 and KPL-14 accumulators, respectively.

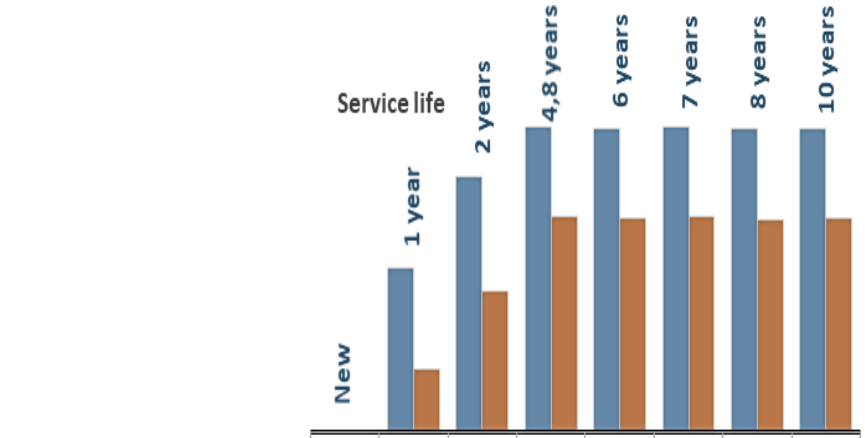
Before putting the new batteries into operation, 2 training cycles were reported for full operation of the mode: charge with a current of 0.2 C_n for 10 hours on the first cycle and 8 hours on the second cycle, discharge with a current of 0.2 C_n for 4 hours on the first cycle and to 1.0 V on the second cycle. Then a control cycle was reported with the mode: charge with a current of 0.2 C_n for 8 hours, a break of 1 hour, and discharge with a current of 0.2 C_n to 1.0 V. After that – charge with a current of 0.2 C_n for 10 hours. Upon completion of charging, the battery was disconnected from the charger. Charging was carried out from a source of direct or rectified current, the maximum working voltage of which is not less than (2*n) V, where n is the number of batteries connected in series. Continuous operation in the buffer mode leads to a decrease in the capacity of the batteries. This process is reversible. To restore capacity, it is recommended, if necessary, to re-train the batteries in a mode similar to that used for commissioning. Due to thermal decomposition of the electrodes from new KSL-15 batteries at the temperature set above, about 160 ml of a mixture consisting of hydrogen and air was released. It is interesting to note the fact that when heating the thermal chamber without an electrode, approximately 130 ml of air is observed in the measuring container due to the expansion of air in the thermal chamber itself, which makes it difficult to determine the initial moment of hydrogen release from the battery electrodes and its entry into the measuring container. In this regard, during the primary thermal decomposition of the battery electrodes, 170 ml of gas is removed from the experimental setup under study, which determines the accuracy of measuring the accumulated hydrogen in the battery electrodes.

Analysis showed that the gas released was only hydrogen. The absolute error of the percentage concentration is 0.3-0.5 %. The results of the experiments are presented in (Figure 2) (KSL-15), (Figure 3) (KPL-14). The relative error of the data in Fig. 2 and 3 is 5–6 %. The next step is to determine the location of the hydrogen: whether it is concentrated in the active mass or in the metal-ceramic frame of the electrode. Experimental work was carried out using nickel oxide electrodes from KSL-15 batteries that had been in operation for six years in identical modes. Sulfuric acid with a concentration of 22.6% was used in the experiment.



№ battery	1	2	3	4	5	6	7	8	9
Amount of gas released from the nickel oxide electrode, l	0	13	16,7	26,3	29,9	32	31,5	31,6	31,9
Amount of gas released from the cadmium electrode, l	0	11	12,8	16,1	18,7	20	19,5	19,5	19,8

Figure 2. Mean hydrogen content in nickel oxide and cadmium electrodes of KSL-15 batteries as a function of operational duration



№ battery	1	2	3	4	5	6	7	8
Amount of gas released from the nickel oxide electrode, l	0	19,9	30,9	37	36,9	37,1	36,8	36,9
Amount of gas released from the cadmium electrode, l	0	7,7	17,2	26,1	25,9	26,1	25,8	25,9

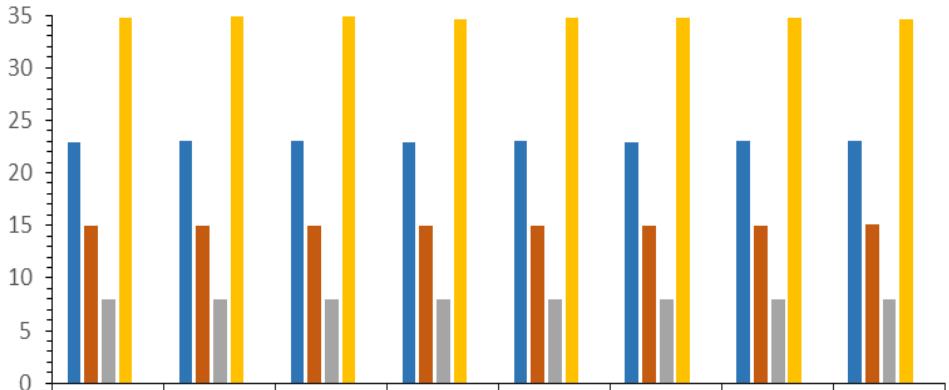
Figure 3. Mean hydrogen content in nickel oxide and cadmium electrodes of KPL-14 batteries as a function of operational duration

First of all, let us consider the probability of hydrogen accumulation in nickel hydroxide. When a nickel oxide electrode is immersed in sulfuric acid, a soluble compound $NiSO_4$ is formed. Based on this, when hydrogen is concentrated in nickel hydroxide either in unbound form or by forming unstable compounds with nickel hydroxide soluble in sulfuric acid, its yield will be equivalent to the amount obtained during high-temperature decomposition of the nickel oxide electrode (Figure 2 and 3). If hydrogen creates strong chemical structures

with nickel hydroxide that are not subject to destruction by sulfuric acid, then the mass of the nickel oxide electrode after acid action will be greater than the initial mass of the metal base.

Additionally, it was found that after 20-minute treatment with sulfuric acid, the mass of the nickel oxide electrode stabilized. In our studies, a treatment time of 30 minutes was used to ensure complete extraction of nickel hydroxide from the nickel oxide electrode. In addition, direct experimental data demonstrated that the sintered nickel base weakly interacts with sulfuric acid, since its mass remained unchanged after 20-minute acid treatment (the error of the scales was ±5 mg). Hydrogen release was also not observed within the measurement accuracy of ±5 ml.

All electrodes of the KSL-15 battery were cleaned with distilled water, dried, and their weight was determined. Then the electrodes were placed in sulfuric acid (in a sealed vessel with a gas outlet tube connected to the measuring container). After the 30-minute acid treatment, the electrodes were again washed with distilled water, dried, and weighed. The obtained data are presented in (Figure 4).



№ electrode	1	2	3	4	5	6	7	8
Initial mass, g	22,91	22,98	23,00	22,92	22,98	22,96	23,02	23,00
Final mass, g	14,96	14,97	14,97	15,00	14,99	14,99	15,01	15,03
Mass loss, g	7,95	8,01	8,03	7,92	7,99	7,97	8,01	7,97
Mass loss, %	34,70	34,86	34,91	34,55	34,77	34,71	34,80	34,65
Amount of hydrogen, ml	0	0	0	0	0	0	0	0

Figure 4. Etching performance in sulfuric acid of nickel oxide electrodes of KSL-15 battery

Based on the experimental results (Figure 4), the following conclusions can be formulated:

- hydrogen does not accumulate in nickel hydroxide either in free or weakly bound form, which is confirmed by the absence of gas evolution during acid etching within the measurement accuracy of ±5 ml;
- according to the technical characteristics of the KSL-15 battery manufacturer, the content of active mass in the nickel oxide electrode is 35±1%.

On the experimental results (Figure 4), it can be hypothesized that hydrogen is concentrated in the metal-ceramic framework of the electrode, similar to processes in other transition metals used for hydrogen accumulation. Based on the experimental results (Figures 2 and 4), the proportion of active material in the positive electrode of a fresh nickel-cadmium battery was estimated.

The average mass of the positive electrode was 23 g (Figure 4). During the six-year operation of the KSL-15 battery, 32 l of hydrogen accumulated in its positive electrode (Figure 2), which corresponds to a mass of $32 \times 2/22.4 = 2.857$ g of hydrogen (where 22.4 l/mol is the molar volume of hydrogen, and 2 g/mol is its molar mass). Therefore, the initial mass of the positive electrode without taking into account the accumulated hydrogen was $23 - 2.857 = 20.143$ g. The mass loss of the positive electrode during treatment with sulfuric acid (which corresponds to the mass of the active material) averaged 8 g (Figure 4). Based on this, the active material content in the new positive electrode was $(8/20.143) \times 100 = 39.71\%$. According to the experimental results shown in Figure 4, the percentage of active material in the positive electrode (when converted to the original hydrogen-free electrodes) was in the range of 34.55% to 34.91%. The obtained values were consistent with the technical specifications of the KSL-15 battery manufacturer. The relative error of the experiment was 4%.

Thus, hydrogen is concentrated in the metal-ceramic frameworks of nickel oxide electrodes. As a result, during high-temperature decomposition of the metal-ceramic framework of the oxide-nickel electrode, only hydrogen is released from it, and only nickel remains. This experiment directly confirms that the metal-ceramic framework of the oxide-nickel electrode after six years of operation contains two components: hydrogen and nickel, i.e. it is a nickel hydride.

Results and Discussion

Figure 2 and 3 conclusively demonstrate that the electrodes of newly developed batteries do not contain hydrogen.

During the course of the research, a correlation was established between the volume of hydrogen accumulated in the electrode materials of nickel-cadmium batteries and their operational lifespan. It was determined that this relationship exhibits a direct proportionality. However, considering the existence of a saturation phase, it was demonstrated that the quantity of hydrogen retained in batteries after more than five years of operation remains constant. These findings are substantiated by experimental data. Furthermore, it was confirmed that the gas accumulated within the battery electrodes is predominantly hydrogen, accounting for approximately 98% of the total gas volume.

For instance, in the KSL-15 battery design, which consists of five cadmium and six nickel oxide electrodes (as illustrated in Figure 2), it can be estimated that approximately 290 liters of hydrogen will be accumulated. Similarly, for the KPL-14 battery design, comprising four cadmium and five nickel oxide electrodes (as shown in Figure 3), an accumulation of approximately 289 liters of hydrogen is anticipated. These estimated volumes of accumulated hydrogen align with previously reported values for other types of nickel-cadmium battery configurations (Feng et al., 2014; Galushkin et al., 2015; Golubkov et al., 2015; Metzger et al., 2016)The calculations presented in this example demonstrate that nickel-cadmium batteries are capable of accumulating such volumes of hydrogen over their operational lifespan.

Consequently, in order to achieve full charge, KSL-15 batteries must be recharged to 1.5 times their nominal capacity. This process results in the consumption of approximately 7.7 Ah during the charging cycle, where hydrogen and oxygen are produced through the electrolyte water decomposition reaction. Experimental observations revealed that during the charging of a KSL-15 battery, approximately 3 liters of hydrogen and 1.5 liters of oxygen are generated. This indicates that the total volume of hydrogen accumulated in KSL-15 battery electrodes, amounting to 290 liters, can be produced through thermal decomposition within approximately 97 charge-discharge cycles. Given that KSL-15 batteries typically undergo several orders of magnitude more charge-discharge cycles throughout their service life, it can be theoretically concluded that they are capable of accumulating 290 liters of hydrogen.

In the case of KPL-14 batteries (as depicted in Figure 3), which are charged with a current of 2.5 A for a duration of 10 hours, according to the technical specifications, the accumulated volume of hydrogen in their electrodes can be achieved within 68 charge-discharge cycles.

During charging, electrolytic water undergoes decomposition, accompanied by the release of hydrogen and oxygen. However, the experimental results obtained (shown in Figure 2 and 3) did not confirm the supposed accumulation of oxygen in the battery electrodes. Instead, only hydrogen was observed to accumulate. An intriguing observation was made concerning the presence of hydrogen in the electrodes of nickel oxide and cadmium batteries, particularly in significant quantities. This phenomenon is likely attributed to the high diffusion permeability of hydrogen. Specifically, the diffusion coefficient of hydrogen in nickel at a temperature of 20°C is approximately 1000 times greater than that of oxygen, as documented in references (Abe et al., 2019; Galushkin et al., 2015; Lei et al., 2017). Consequently, during the decomposition of electrolyte water into its constituent gases, hydrogen diffuses into and accumulates within the electrodes, while oxygen is more likely to escape into the atmosphere. In this case, to study the accumulation of hydrogen, oxide-nickel electrodes of KSL-15 batteries with a six-year period of operation in identical modes were used.

The active mass was extracted from the electrodes by acid etching. Then the nickel frames were subjected to high-temperature decomposition. The volume of released hydrogen corresponded to the data in Figure 4 with a relative experimental error of 3 %.

Conclusion

It is established that hydrogen gas is produced at the cadmium electrodes during the charging process of nickel-cadmium batteries. Experimental data, as illustrated in Figures 2 and 3, indicates that hydrogen accumulates on both the cadmium and nickel oxide electrodes. This phenomenon is likely due to the dense packing of sintered electrodes in KSL-15 batteries and KPL-14, allowing hydrogen generated at the cadmium electrodes to permeate into the nickel oxide electrodes and subsequently accumulate within them.

At the same time, the obtained results require in-depth experiments and theoretical justification. The research work revealed that the concentration of hydrogen in the electrode system of nickel-cadmium power elements increases during their long-term operation. The laboratory tests of hydrogen accumulation helped to clarify the process and areas of hydrogen concentration in the oxide-nickel electrodes of batteries after their long-term operation. The main results of this study:

1. The location of hydrogen accumulation was determined. It was experimentally proven that hydrogen does not accumulate in the active mass of the electrode (nickel hydroxide) either in a free or weakly bound state. This is evidenced by the absence of gas evolution during acid etching of the electrodes with sulfuric acid within the measurement accuracy of ±5 ml.
2. Hydrogen localization in the metal-ceramic frame was established. Hydrogen is concentrated exclusively in the metal-ceramic base of the electrode, forming nickel hydride. This is confirmed by the results of high-temperature decomposition of frames cleaned of the active mass, during which hydrogen is released in volumes corresponding to the data for intact electrodes.
3. The composition of the metal-ceramic frame after operation was confirmed. After six years of operation in identical modes, the metal-ceramic frame of the nickel oxide electrode is a two-component "nickel-hydrogen" system, i.e. nickel hydride.
4. The research methodology is substantiated. The developed method of acid etching with subsequent high-temperature decomposition allows for reliable separation of the contributions of the active mass and the metal-ceramic framework to the processes of hydrogen accumulation.

The obtained results are essential for understanding the degradation processes of NiCd batteries and can be used to develop methods for extending their service life and optimizing the design of electrodes. From the point of view of automation, these studies are of great importance for the control of technological processes in battery management systems. The detailed analysis of hydrogen accumulation in nickel-cadmium batteries provides crucial data for developing intelligent monitoring algorithms and automated diagnostic tools.

The high diffusion permeability of hydrogen, particularly its ability to accumulate in nickel matrices, presents both challenges and opportunities for technological process automation. Future automated battery management systems could incorporate advanced sensors and algorithms to track hydrogen diffusion and accumulation in real-time. Moreover, the research underscores the importance of developing automated technologies that can monitor and mitigate potential risks associated with hydrogen generation in battery systems. Using these insights, engineers can develop more reliable, intelligent, and safe battery management solutions in various EV applications.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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