Capacity Degradation Mechanisms in Nickel/Metal Hydride Batteries

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Abstract: The consistency in capacity degradation in a multi-cell pack (>100 cells) is critical for ensuring long service life for propulsion applications. As the first step of optimizing a battery system design, academic publications regarding the capacity degradation mechanisms and possible solutions for cycled nickel/metal hydride (Ni/MH) rechargeable batteries under various usage conditions are reviewed. The commonly used analytic methods for determining the failure mode are also presented here. The most common failure mode of a Ni/MH battery is an increase in the cell impedance due to electrolyte dry-out that occurs from venting and active electrode material degradation/disintegration. This work provides a summary of effective methods to extend Ni/MH cell cycle life through negative electrode formula optimizations and binder selection, positive electrode additives and coatings, electrolyte optimization, cell design, and others. Methods of reviving and recycling used/spent batteries are also reviewed.

Keywords: nickel/metal hydride (Ni/MH) battery; failure analysis; electrochemistry; hydrogen storage alloys; self discharge; capacity degradation

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1. Introduction

Nickel/metal hydride (Ni/MH) batteries are widely used in many energy storage applications. Cycle stability is one of the key criteria in judging the performance of rechargeable battery technology. The general observations regarding failed Ni/MH cells are summarized in Figure 1. In order to further investigate the mechanisms of capacity degradation and their relevant solutions to extend cycling under normal and abuse conditions, we have chosen to begin with a review of the significance of the Ni/MH battery in the overall battery market, its basic structure and chemistry, and the analytical tools used to study and characterize its performance, and to mainly focus on academic publications and reports. Patents detailing solutions for extending cycle life are reviewed in two separate articles [1,2].

1.1. Significance of Nickel/Metal Hydride Batteries

Ni/MH batteries using an alkaline KOH electrolyte have been commercialized for more than 25 years [3]. Because of its durability, abuse tolerance, compact size, and environmental friendliness, Ni/MH battery applications have steadily expanded from the traditional consumer market to include propulsion and telecommunications. However, due to its relatively low gravimetric energy density compared to the rival Li-ion battery, the Ni/MH battery lost part of its market share in portable electronic devices, such as notebook computers, cell phones, and digital cameras. In the meantime, Ni/MH battery technology also invaded the primary alkaline battery market because of its voltage compatibility and

low self-discharge [4–6], as well as the NiCd power tool market for its non-toxicity [7,8]. The success of Ni/MH in powering hybrid electric vehicles (HEV) developed by a handful of automobile manufacturers stems from its wide temperature range, abuse tolerance, superb cycle stability, high charge and discharge rate capabilities, and environmental friendliness [9]. One analyst even predicted a fourfold increase in Ni/MH battery sales for HEV and EV markets from 2014 to 2020 [10]. Although the current industries making pure battery-powered electric vehicles embrace Li-ion battery technology, a Ni/MH pouch cell developed under a five million dollar Advanced Research Projects Agency-Energy (ARPA-E) program has demonstrated a specific energy of 127 Wh·kg^{-1} at the cell level with an estimated target of 148 Wh·kg^{-1} [11]. With the recent breakthrough of high-energy Si-negative electrodes capable of storing 3635 mAh·g^{-1} (about ten times the current A_2B_7 alloy) [12], the future of Ni/MH in the EV application appears very bright. From the beginning of their competition, Ni/MH batteries have had higher volumetric energy density than Li-ion batteries, due to the high density active materials (rare earth metal (RE) and transition metal *versus* carbon-based products in the anode and nickel hydroxide *versus* lithiated transition metal oxides in the cathode). With the improvements in specific energy, a resurgence in Ni/MH batteries for applications that place a premium on space rather than weight, such as portable displays, wearable electronic devices, and medical devices, can be expected. As for large-scale high-power temporary energy storage applications, the GIGACELL, made by Kawasaki Heavy Industries, demonstrates superior performance using the Ni/MH chemistry [13,14]. In stationary applications, its excellent cycle stability and wide operating temperatures, combined with the low cost and easy manufacturability, have made Ni/MH the best choice [15]. The overall outlook for Ni/MH battery technology shows that it has tremendous potential in various energy storage applications following these new scientific discoveries and process improvements—a far cry from being the 25-year obsolete veteran in the battery business.
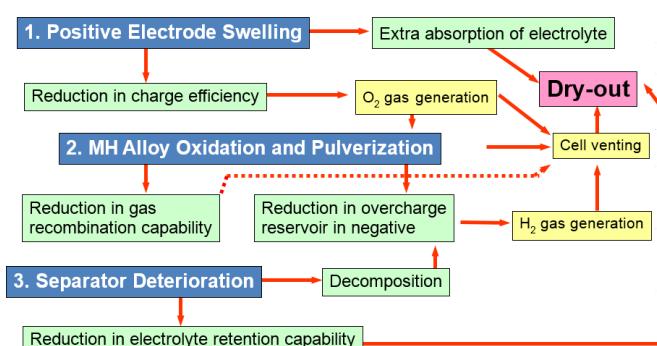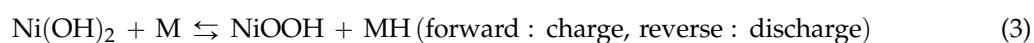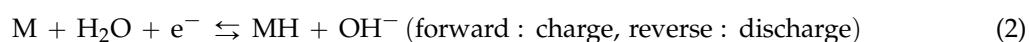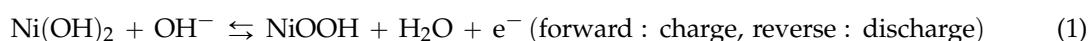

Figure 1. Schematic diagram of three key factors leading to the major failure mode of nickel/metal hydride (Ni/MH) cells—electrolyte dry-out.

1.2. Basic Structure of Nickel/Metal Hydride Battery

There are basically seven different types of Ni/MH batteries: cylindrical with metal cases, stick (bubble gum shape), prismatic with metal cases, prismatic with plastic cases, button cell, pouch cell [16], and flooded cell [17] (Figure 2). All but button cell and pouch cell have a safety valve installed to prevent explosions from gas build-up. A simple comparison between various construction types is shown in Table 1. They all share some common parts: positive electrode, negative electrode, separator, electrolyte, case, and safety valve (except button and pouch cells). The basic electrochemistry reactions for the positive electrode, negative electrode, and full cell are:

$$Ni(OH)_2 + OH^- \leftrightarrows NiOOH + H_2O + e^- \text{ (forward : charge, reverse : discharge)} \tag{1}$$

$$M + H_2O + e^- \leftrightarrows MH + OH^- \text{ (forward : charge, reverse : discharge)} \tag{2}$$

$$Ni(OH)_2 + M \leftrightarrows NiOOH + MH \text{ (forward : charge, reverse : discharge)} \tag{3}$$

where M is the hydrogen storage metal/alloy and MH is the hydride of metal M. During the charge process, bi-valent Ni is oxidized into the tri-valent state while metal M is reduced by the absorbed hydrogen atom. The most commonly used positive electrode in the current Ni/MH battery technology consists of active materials made of co-precipitated spherical hydroxides from Ni, Co, and Zn and some binders, pasted onto Ni-foam via a wet method. Recently, a dry application of spherical powder onto Ni-foam with no binder followed by immediate compaction has also been used to increase the energy and power density of the cell. In some high-temperature/high-rate applications, old sintered-type positive electrodes, based on fibrous Ni on stainless steel plate, are still in commission. Co-coating of the spherical particles and additives such as metallic Co and/or CoO and rare earth element (RE) oxides in the positive electrode paste are also popular. A review of the synthesis and properties of $Ni(OH)_2$ was recently reported [18].
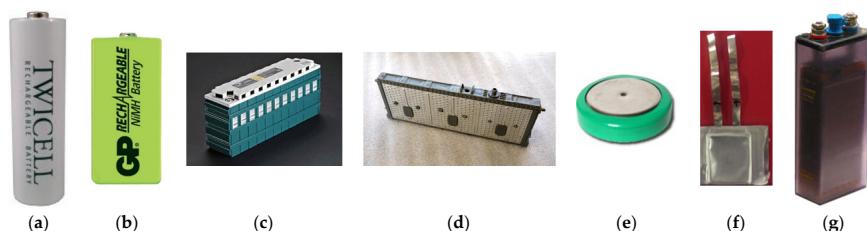

Figure 2. Ni/MH batteries in: (**a**) cylindrical; (**b**) stick; (**c**) metallic prismatic; (**d**) plastic prismatic; (**e**) button; (**f**) pouch; and (**g**) flooded configurations.

Table 1. Comparison of different types of Ni/MH battery packaging.

Shape	Case material	Sealed	Manufacturability	Cost	Energy density	Heat dissipation	Abuse tolerance
Cylindrical	Metal	Yes	Easy	Low	High	Easy	High
Stick	Metal	Yes	Medium	Low	High	Easy	High
Prismatic	Metal	Yes	Medium	High	Low	Easy	Med
Prismatic	Plastic	Yes	Medium	High	Low	Hard	Med
Button	Metal	Yes	Easy	Low	Low	Easy	Low
Pouch	Al foil	Yes	Easy	Low	Very high	Easy	Low
Cylindrical/prismatic	Plastic/flooded	No	Easy	Low	Low	Hard	High

The most common metal hydride (MH) alloy used in the negative electrode is a RE-based AB_5 alloy. A typical atomic composition is $La_{10.5}Ce_{4.3}Pr_{0.5}Nd_{1.4}Ni_{60.0}Co_{12.7}Mn_{5.9}Al_{4.7}$. Recently, RE- based A_2B_7 MH alloys have gained popularity in high-energy and low self-discharge consumer type applications [4–6]. A typical atomic composition of this type is $La_{6.7}Pr_{6.3}Nd_{6.3}Ni_{72.8}Al_{4.0}$. Recent progress in MH alloys for Ni/MH battery applications can be found in the following review article [19]. The negative electrode can be prepared by dry-compacting the MH powder directly onto a Ni-mesh, Cu-mesh, expanded Ni, Ni foam, or expanded Cu substrates without the use of a binder, or by wet-pasting a slurry with MH alloy, binder, and/or additives onto nickel plated perforated stainless steel (NPPS).

A 30% KOH solution is widely used as the electrolyte for Ni/MH batteries due to the balance of conductivity and freezing point temperature. Performance comparisons for other concentrations [20] and alkaline metal hydroxides [21] are available. A small amount of LiOH ($1.5~g \cdot L^{-1}$), which has higher chemical reactivity, is added to boost low-temperature performance, while in high-temperature applications, part or all of the KOH is replaced by the less reactive (corrosive) NaOH to reduce corrosion. In the current standard mass production of Ni/MH cells, no other specific additive is added to the electrolyte.

Grafted polypropylene (PP)/polyethylene (PE) non-woven fabric is today's standard separator material, and an overview has been published by Kritzer and Cook [22]. While the regular separator is white in color, the sulfonated separator is brownish and offers benefits to low self-discharge due to its ability to trap redox shuttle substances, especially the nitrogen-containing compounds [23]. Both types of separators can be found in current NiMH batteries.

1.3. Experimental Methods Used in Failure Analysis

A few analytic tools are frequently used to identify the failure mode of a cycled Ni/MH battery [24–26]. Scanning electron microscope (SEM) with X-ray energy dispersive spectroscopy (EDS) capability is commonly used to examine the degree of pulverization, phase segregation, degree of oxidation, physical size changes, and trapping of particulates. The different features found between the secondary electron image and the backscattering electron image can be extrapolated into the changes in the average atomic weight of the area of interest. EDS mapping is especially useful in studying elemental distribution (for example, oxygen) in a relatively large area (10–100 μm scale). While gas chromatography (GC) is used to identify the gas composition in the cell, inductively coupled plasma (ICP) is used to examine the metallic composition of any solid (electrode, separator, tap, *etc.*) or liquid (remaining electrolyte and solution attained through Soxhlet extraction) content from the autopsy of a cycled cell. Titration is another method to determine the content of a specific element [27]. X-ray diffraction (XRD) is an important tool to study oxide formation, phase changes, and microstructure changes in both negative [25,26,28,29] and positive [30] electrodes.

Other tools are used less frequently in failure analysis. For example, transmission electron microscope (TEM) is sometimes used to study the microstructure and composition of the surface oxide from a cycled cell [31–33]. Magnetic susceptibility (MS) measurements can be used to monitor the evolution of the count and size of metallic Ni-clusters embedded in the surface oxide [26,34,35]. Both X-ray photoelectron spectroscopy (XPS) [36–40] and Auger electron spectroscopy (AES) [41] have been used to study the surface composition, with the former being able to identify the oxidation state. The acoustic emission (AE) technique has also been used to study the volume change and pulverization of the MH alloy [42,43]. Fourier transform infrared spectroscopy (FTIR) has been used to study the OH^- ligand in $Ni(OH)_2$ [44–47]. Raman spectroscopy (RS) is another optical measurement used to characterize the changes in the separator and positive electrode [37,46–48]. Electrochemical impedance spectroscopy (EIS) or AC impedance measurements are usually used to isolate components with different degrees of degradation [49–52]. Polarization curves [53–56] and cyclic voltammetry (CV) [57–60] are other electrochemical tools that can be used to study the evolution of electrode surface changes. Besides experiments with real batteries, empirical capacity degradation models have also been previously developed [61–63].

2. Capacity Degradation

Battery failure can be separated into two categories: accidental and long-term degradation. The former includes fire, electrical short-circuit, and physical damage. In Table 2, we have listed a few common symptoms and possible causes that originated the failure of the batteries. Long-term capacity loss under various test conditions is discussed in the remainder of this section.

Table 2. Common Ni/MH battery failure symptoms and possible causes.

Symptom	Reasons	Possible causes
Battery short-circuit	Direct conducting path between two electrodes developed	• Separator punch-through • Conducting debris from Cu-impurities • Deformation of electrode causing direct contact between taps
Battery open-circuit	Breakage of inside connection	• Electrode breakage due to expansion/distortion • Broken tap connection • Complete electrolyte dry-out
Battery abuse	Over-discharge and overcharge	• Unbalanced capacity in positive and negative electrode • Mismatched charger
Capacity decrease	Electrode degradation	• Pulverization/oxidation of MH alloys in negative electrode • Pulverization in spherical particle due to formation of γ-NiOOH phase • Decrease in the Co-conductive network in the positive electrode

Table 2. *Cont.*

Symptom	Reasons	Possible causes
Power decrease and impedance increase	Electrolyte dry-out	• Venting from improper cell-balance • Consumption due to oxidation
	Electrode degradation	• Reduction in electrode active materials • Increase of the surface oxide of negative electrode • Loss of co-conductive network in positive electrode
	Separator degradation	• Increase in fiber diameter • Reduction in pore volume • Impurity trapped internally • Decomposition
Overheat during charge	Micro-shorting	• Conductive debris accumulation in separator
White deposits	Electrolyte leak from venting	• Improper closing of the cell • Off-balance in the remaining electrode capacity • Deterioration of gas recombination capability at the surface of MH alloy • Heavily oxidized electrode and/or electrolyte • Failure in the safety vent

2.1. Capacity Loss During Normal Cycling at Room Temperature

There are two types of capacity loss during cycling: reversible and irreversible. The reversible part is also called self-discharge, which mainly occurs through six pathways: shuttling effects from nitrogen containing compounds [64], shuttling effects from soluble ions of multi-valence transition metals [65], micro-shorts [66] from conducting/semiconducting deposits trapped in the separator [67,68], hydrogen gas desorption from MH alloys [69–73], direct reaction between hydrogen gas and NiOOH [69,73,74], and CoOOH protective/conductive coating breakdown due to contamination from leached MH alloys [6]. Self-discharge accelerates with rises in the environmental temperature. There is basically no self-discharge at below $-5\,^{\circ}$C [75]. Before the low self-discharge Ni/MH battery was introduced (the Eneloop cell from Sanyo using a combination of improved MH alloy, separator, and positive active materials [6]), cells initially had a monthly 20%–30% capacity reduction, which was then improved to a monthly loss of 5%–10% at room temperature [76]. Modern low-self discharge Ni/MH consumer batteries have self-discharge rates of less than 20% per year [6]. An automatically triggered re-charging algorithm may be necessary for large-scale applications [77]. Common methods used to suppress self-discharge in Ni/MH batteries are summarized in Table 3. The irreversible capacity loss, which leads to failure of the battery, covers the majority of this review.

Table 3. Summary of common methods used to suppress self-discharge in Ni/MH batteries. The star system used in the effectiveness column in Tables 3–9 was meant to show the relative strength in each method to address the problem based on authors' own experience. Interested readers are encouraged to read the original article and to form their own opinions. PP: polypropylene; PTFE: polytetrafluoroethylene; and CMC: carboxymethyl cellulose.

Method	Direct impact	Environmental impact	Cost impact	Effectiveness	References
Use of a sulfonated separator	Removal of N-containing compounds	None	Modest	★★★★★	[22,78,79]
Use of an acrylic acid grafted PP separator	Reduction in Al- and Mn-debris formation in separator	None	None	★★★★	[80]
Removal of Co and Mn in A_2B_7 MH alloy	Reduction in debris formation in separator	None	None	★★★★★	[6,81]
Increase of the amount of electrolyte	Reduction in the hydrogen diffusion in electrolyte	None	None	★★★★	[82]
Removal of Cu-containing components	Reduction in micro-short	None	None	★★★★★	[83–85]
PTFE coating on positive electrode	Suppression of reaction between NiOOH and H_2	None	Negligible	★★★★	[86]

Table 3. *Cont.*

Method	Direct impact	Environmental impact	Cost impact	Effectiveness	References
CMC solution dipping	Suppression of oxygen evolution	None	Negligible	★★★★	[87]
Micro-encapsulation of Cu on MH alloy	Decrease in H_2 released from MH alloy	None	Modest	★★★	[88]
Ni-B alloy coating on MH alloy	Formation of a protection layer	None	Modest	★★★	[89]
Alkaline treatment of negative electrode	Reduction of leach-out of Mn and Al	None	Modest	★★★★	[90]
Addition of LiOH and NaOH into electrolyte	Reduction in electrolyte corrosion capabilities	None	None	★★★★	[75]
Addition of $Al_2(SO_4)_3$ into electrolyte	Reduction in MH alloy corrosion	None	Negligible	★★	[91]

Irreversible capacity losses under regular cycling conditions (temperature between 20 °C and 30 °C, rated below 2C with one or a combination of reasonable cut-off schemas during over-charge, such as those used in [26,29]) can be categorized into five main categories: degradation of negative electrode active material (MH alloy), degradation of positive electrode active material (spherical Ni(OH)$_2$), disintegration of the negative electrode, disintegration of the positive electrode, and venting of cells.

Degradation in the negative electrode includes MH alloy pulverization due to lattice expansion during hydrogenation [92–95] which results in poor electrical and protonic conduction [49,95,96], alloy surface oxidation hampering electron and proton conduction [36,52–54,93,94,97–101], and surface fluoride formation [36]. The corrosion processes of AB$_5$ MH alloys have been characterized by Maurel and his coworkers using XRD, SEM, and TEM [102]. In the La-only A$_2$B$_7$ superlattice MH alloy, the pulverization due to different sequences of hydrogenation between Mg-containing A$_2$B$_7$ and Mg-free AB$_5$ phases dominates the failure mode [25,103].

Degradation in the positive electrode includes swelling from γ-NiOOH formation [67,104], breaking of the Co-conductive network [105], formation of less electrochemically rechargeable γ-NiOOH [25,93,106], Co dissolution and migration from the conductive network in the positive electrode [107], contamination from leach-out products (Al and Mn) in the negative electrode, deteriorating Co-conductive coating [27,104], and pulverization of positive electrode spherical particles causing detachment of active material [68,108]. The increased surface area in the positive electrode as a result of pulverization also deprives electrolyte from the separator, which increases cell resistance [109].

The mechanical disintegration of the negative electrode may include breakage of the NPPS substrate due to increased stress from electrode expansion/distortion and MH alloy powder detachment from the substrate. The mechanical disintegration of the positive electrode may include breakage of the Ni-foam substrate due to large amounts of stress from electrode expansion/distortion [110], especially in a small wounded cylindrical cell [111], and separation of spherical particles from the substrate [112]. Venting occurs when high pressure (mostly H_2) is built up inside the cell primarily from inadequate gas recombination capabilities of the MH alloy surface and/or unbalanced capacity distribution [113], which results in reduced electrolyte content [114].

2.2. Capacity Loss During Long-Term Room Temperature Storage

The irreversible capacity loss during long-term room temperature storage can be attributed to the dissolution of the surface CoOOH conducting network [115,116], corrosion/passivation of the negative electrode [23,117–119], decomposition of the positive electrode [115], decomposition of the separator [116], and poisoning of the positive electrode from cations that originate from the negative electrode [68,80,115].

2.3. Capacity Loss During High-Temperature Storage

Temperature is one of the key factors affecting cycle stability [120]. In addition to the regular capacity losses described in Section 2.1, high-temperature environments (⩾45 °C) will accelerate the cell degradation through the following pathways: oxidation rate increases at the surface of the MH

alloy particles [121,122], dissolution of Co-compounds in the Co-conductive network [113,123], higher self-discharge rates that lower the cell voltage and result in further alloy oxidation, and separator degradation [124]. The charging method used in the high-temperature range has to be specially designed. First, the cell voltage tends to be lower at higher temperature, which demands that a lower cut-off voltage be adopted during charging to prevent over-charge [122] as it can be directly correlated to capacity degradation [125]. Next, the oxygen gas evolution potential in the positive electrode tends to decrease with increased temperature, which forces the positive electrode to finish charging prematurely and for which the $-\Delta V$ cut-off method is less effective [122,126,127]. Ni/MH batteries are also more sensitive to over-charge at elevated temperatures. Ni/MH batteries overcharged at rates of 0.2C, 0.5C, and 1.0C for one month show irreversible capacity losses of 12%, 30%, and 40%, respectively [126]. Different from the irreversible capacity losses during high-temperature cycling, losses in capacity observed during low-temperature cycling are recoverable when returned to room temperature [74].

2.4. Capacity Loss Due to Low-Temperature Cycling

As stated above, low-temperature storage of Ni/MH batteries causes no apparent damage to performance. However, Chen *et al.* [128] reported capacity degradation during a $-20\ ^{\circ}$C cycling experiment with MH alloy pulverization, but the alloy corrosion was less serious compared to results from room temperature and high temperature. At low temperatures, a special "surface icing" appears to form on the MH alloy, further hindering electrochemical reactions and then disappearing at higher temperature [129].

2.5. Capacity Loss Due to High-Rate Cycling

Fast charge acceptance is controlled by solid-state hydrogen diffusion [130], and the diffusion coefficient of hydrogen decreases with increasing current density [131]. The increase in the degradation rate with fast charging typically originates from an improper termination method for detecting the end of charge, which leads to a large degree of over-charge especially within an aged cell [132]. The heat generated from the internal resistance of the cell and the hydrogen-oxygen recombination reaction cannot be dissipated quickly enough, and this results in an increase in the cell temperature. Both the high rate and the high temperature conditions reduce charging efficiency [133] and therefore both conditions facilitate similar failure mechanisms, except that a high-rate cycled cell also shows electrode disintegration from extraordinarily fast gas release [134] (mostly H_2 [135]) as well as gas venting due to the insufficient time for hydrogen-oxygen recombination [136,137]. As such, fast charging of a large-sized Ni/MH battery is not recommended unless special temperature monitoring devices are installed [138,139].

2.6. Capacity Loss in a Multi-Cell Module

Thus far, the discussion in this section has focused on the cell-level where most of the capacity degradation occurs. In a single Ni/MH cell, both the over-charge (with a state-of-charge (SOC) greater than 100%) and over-discharge (depth of discharge (DOD) greater than 100%) conditions can be avoided by the proper monitoring of the cell voltage. Because of the low risk of operating Ni/MH cells under disadvantageous conditions, a multi-cell module or pack does not require voltage monitoring at the cell-level whereas the Li-ion battery does. With the proper design of the negative-to-positive capacity (n/p) ratio, the size of the over-discharge reservoir [113] and the anticipated rates of capacity degradation in both electrodes, the over-charge or the over-discharge of the cells only results in small amounts of oxygen gas or hydrogen gas evolution, respectively, in the positive electrode [17]. The small amounts of generated oxygen gas can be recombined with the hydrogen stored in the negative electrode in case of over-charge, and the small amounts of generated hydrogen gas can be stored in the negative electrode in case of over-discharge [113]. Repetitive gas evolutions from the positive electrode can result in both mechanical disintegration of the electrode and cell venting to relieve the pressure, causing a loss in capacity and an increase in cell impedance. The DOD in a multi-cell pack also plays

an important role in the cycle life performance. For example, an increase of DOD from 10% to 90% in a HEV Ni/MH pack can reduce cycle life from 5000 cycles to 500 cycles [61]. For high-rate operation, as in a HEV, large swings in the SOC can result in premature MH alloy pulverization.

3. Methods to Improve Cycle Stability

There are many academic publications and issued patents offering, at least, partial solutions to the capacity loss problem during cycling. While patents addressing cycle stability are reviewed in two other papers [1,2], the strategies issued from the academic research community reviewed here fall under six general categories: (1) cell designs guided mainly by n/p ratio, electrolyte loading, and electrode thickness parameters; (2) active binder and additive material designs in the negative electrode; (3) composition, coating, and paste additives in the positive electrode; (4) choice of separator; (5) electrolyte; and (6) other components. Other systematic maintenance protocols for battery packs using Ni/MH cells were reported by Zhu and his coworkers [140].

3.1. Cell Design

In good Ni/MH cell design, an appropriate n/p ratio is critical to the balance of the various performance requirements in a specific application. For instance, a high energy consumer cell, a general purpose cell, and a high-rate cell may have n/p ranges of 1.05–1.2, 1.4–1.6, and 1.8–2.2, respectively. Adequate distribution of the extra negative electrode capacity into the over-charge-reservoir (OCR) and the over-discharge-reservoir (ODR) to avoid cell-venting is especially critical, particularly near the end of service life [116]. Nearly all cases of venting are due to short-circuits in the OCR that arises from material oxidation and γ-NiOOH formation that overwhelm the ODR. Other important design parameters that impact cycle life performance are electrolyte loading and electrode thickness. The amount of electrolyte added to the cell is proportional to cycle life, but too much electrolyte will eliminate the gas recombination centers and cause venting during formation. Optimal electrolyte loading is about 1.7–1.9 g·A^{-1}·h^{-1} [141]. Thicker electrodes can improve the gravimetric and volumetric energy densities of the battery at the expense of high-rate discharge capability and mechanical integrity of the electrode. Methods for improving cycle performance through cell design are summarized in Table 4.

Table 4. Summary of cycle stability improvement methods related to cell design. ODR: over-discharge-reservoir; and n/p: negative-to-positive capacity.

Method	Direct impact	Environmental impact	Cost impact	Effectiveness	References
Pre-charge of the positive electrode	Reduction of ODR	None	Negligible	★★★★★	[142,143]
Increase in the n/p ratio	Trade-off of capacity for longer life	None	None	★★★★★	[134,144]
Optimization of electrolyte loading	Balance between cycle life and production yield	None	None	★★★★	[141]
Optimization of positive electrode thickness	Reduction in electrode breakage	None	None	★★★★	[145]
Pre-charge during the formation process	Protection of MH alloy	None	Negligible	★★★	[146]

3.2. Negative Electrode

While studies of degradation in MH alloys such as AB_2 [147–153], Mg-Ni [154–157], and V-based body-center-cubic [158] are available, we will singularly focus on the discussion of misch-metal based AB_5 and A_2B_7 superlattice MH alloys and their related electrode properties. In this section, improvements in cycle stability related to the negative electrodes are summarized in Table 5 and are categorized by alloy formula, alloy preparation, alloy post-treatment, electrode additives, and different electrode types.

Table 5. Summary of cycle stability improvement methods related to negative electrode. PVA: polyvinel alcohol; HEC: hydroxyethyl cellulose; and RE: rare earth metal.

Method		Direct impact	Environmental impact	Cost impact	Effectiveness	References
A. Alloy formula	Increase in Al-content	Increase in unit cell volume and reduction in lattice expansion during hydrogenation. Formation of Al_2O_3 protection layer on MH alloy.	None	None	★★★★★	[159–162]
	Increase in Co-content	Reduction in hardness and prevention of La-migration onto surface	None	Modest	★★★★★	[163]
	Use of misch-metal instead of pure La	Increase in degree of disorder	None	Reduction	★★★★★	[164,165]
	Increase in Ce and Nd content	Increase in oxidation resistance	None	Modest	★★★★★	[166]
	Zr addition	Decrease in pulverization rate	None	Negligible	★★★★★	[167,168]
	Ti addition	Decrease in pulverization rate	None	Negligible	★★★★	[168,169]
	Use of hyper-stoichiometry	Reduction in pressure-concentration-temperature hysteresis and pulverization	None	None	★★★★	[92,163]
B. Alloy preparation	Fast quenching-gas atomization	Distribution of stress from lattice expansion	None	Modest	★★★★★	[40,170–173]
	Fast quenching-melt spin	Improvement in alloy homogeneity	None	Modest	★★★★★	[174,175]
C. Surface treatment	Ni surface plating	Protection of alloy surface from oxidation and reduction in inner pressure	None	Modest	★★★★★	[176,177]
	Cu coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[178–181]
	Co coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[182]
	Pd coating	Protection of alloy surface from oxidation	None	High	★★★★	[183]
	Ni-B alloy coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[89]
	Ni-P alloy coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[184]
	Ni-S alloy coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[185]
	Ni-Cu alloy coating	Protection of alloy surface from oxidation	None	Modest	★★★★	[186]
	Alkaline pre-activation	Formation of a Ni-rich surface	None	Modest	★★★★★	[187]
	KBH_4 treatment	Formation of a Ni-rich surface	Toxic in contact with skin	Modest	★★★★★	[187,188]
	Surface fluorination	Protection of alloy surface from oxidation	None	Modest	★★★★★	[189–192]
	Cu and HF surface treatment	Formation of CuF_2 protective layer on the surface	None	Modest	★★★	[193]
D. Other treatments	AB_5 annealing	Improvement in Mn homogeneity and reduction in inner pressure	None	Modest	★★★★★	[166,194–196]
	La-A_2B_7 annealing	Improvement in phase homogeneity	None	Modest	★★★★★	[197]
	Magnetization	Improvement in mechanical integrity	None	Modest	★★★	[198]
	Ultrasound treatment	Reduction in pulverization	None	Modest	★★★	[128]
E. Additives	Ni fine powder	Increase in mechanical integrity	None	Negligible	★★★★	[199]
	Cu fine powder	Increase in mechanical integrity	None	Negligible	★★★	[200]
	Co-compounds	Increase in oxidation resistance	None	Modest	★★★★	[60,201–203]
	CMC:PVA (3:2)	Increase in mechanical integrity	None	Negligible	★★★★	[204]
	Ratio of binder to conductive additives	Increase in mechanical integrity	None	None	★★★★	[205]
	PTFE	Improvement in hydrogen gas absorption capability to reduce pressure	None	Negligible	★★★★	[206]
	Teflonized carbon	Creation of 3D conductive network	None	Negligible	★★★★	[207]
	HEC	Improvement in hydrogen gas absorption capability to reduce pressure	Very low toxicity if swallowed	Negligible	★★★★	[127,195]
	BC-1 (irigenin)	Improvement in gas recombination rate	None	Negligible	★★★★	[208]
	Carbon nanotube	Increase in mechanical integrity	None	Modest	★★★★	[209,210]
	Y_2O_3	Improvement in corrosion resistance	None	Modest	★★★★	[211]
	Oxides of light RE	Improvement in corrosion resistance	None	Modest	★★★★	[212]
	Oxides of heavy RE	Improvement in corrosion resistance	None	Modest	★★★★	[213,214]
F. Electrode type	Use of a pellet electrode	Increase in mechanical integrity	None	Reduction	★★★	[215]
	Use of a sintered type electrode	Increase in mechanical integrity	None	Reduction	★★★★	[216]

3.3. Positive Electrode

Currently, the most commonly used positive active material is a spherical hydroxide co-precipitated from sulphates [45] of Ni, Co, and Zn [217]. Ni has been in active use for more than one hundred years due to the chemical reversibility between Ni^{2+} and Ni^{3+} and a voltage slightly above the oxygen gas evolution potential that maximizes energy density for aqueous chemistries. Both Zn and Cd [218] are good suppressors of γ-NiOOH formation, which causes swelling of the positive electrode and consequently premature failure, but Cd is highly toxic to the environment. The element Co is interesting in that it has oxides with different oxidation states (CoO, Co_2O_3, Co_3O_4, β-$CoOOH$, β-$H_{0.5}CoO_2$ [219], and Co^{4+} [220]). The mechanism of reaction for Co in alkaline solution is rather complicated [38,221], but a simplified version for electrochemical engineers can be used as a guideline. Co in a +2 state is not a good conductor for electrons or protons, and it is only slightly soluble in 30% KOH. Co can be oxidized into the +3 state through solid-state reaction [222], and it is a good conductor for both electrons and protons due to the half-filled proton plane between two Co-O layers in the CoOOH crystal structure; however, the reaction is not easily reversible. The presence of Co^{4+} through a solid-state reaction can be detected at charge rates greater than C/5, and it can be reduced back to Co^{3+} at a potential of 1.05 V *versus* Cd-electrode [220]. There are three general methods for incorporating Co into the positive electrode of Ni/MH batteries, which leverage the irreversible oxidation of Co^{2+} in the normal operation voltage range (>0.63 V *versus* Cd-electrode [220]). First, Co co-precipitated with the spherical hydroxide particles form Co^{3+} to enhance the electron and proton conductivities for $Ni(OH)_2$. Second, the addition of Co, CoO, or other Co-compound into the electrode paste allows the formation of a CoOOH conductive network that surround the spherical $Ni(OH)_2$ particles. This Co-conductive network is crucial for the operation of Ni/MH batteries, especially at high rate conditions, but they can have issues with distribution, thickness uniformity, and severe degradation at high-temperatures [223]. A third method involves adding a pre-coating of CoOOH onto the spherical particles prior to making the slurry for the electrode paste, which can involve a wet-precipitation [224–226], a mud-slurry [227], or a dry mixing method. The use of Co in the pre-coating form is the most effective and economical method, and thus is indispensable in high-end Ni/MH consumer products. Suggestions to improve cycle stability related to the positive electrode are summarized in Table 6 and are categorized by spherical particle composition and size, coatings, additives, fabrication process, and substrates.

Table 6. Summary of cycle stability improvement methods related to the positive electrode. NPPS: nickel plated perforated stainless steel.

Method		Direct impact	Environmental impact	Cost impact	Effectiveness	References
A. Composition and particle size	Co-precipitation of Co	Increase in intrinsic conductivity	None	Modest	★★★★★	[228]
	Co-precipitation of Zn	Prevention of γ-NiOOH formation	None	Negligible	★★★★★	[93,229]
	Co-precipitation of Mg and/or Ca	Improvement in high-temperature performance	None	Negligible	★★★	[230]
	New type of Ni-Al double layered hydroxide	High capacity α-Ni(OH)$_2$/γ-NiOOH	None	Negligible	★★★★	[58]
	Increase in Ni(OH)$_2$ crystallite size	Trade-off in activation	None	None	★★★★	[231]
B. Surface coating	CoOOH coating	Enhancement in survival rate after long-term storage	None	Modest	★★★★★	[121,139,223]
	Yb(OH)$_3$ coating	Improvement in high-temperature performance	None	Modest	★★★★	[232]
	Electrode-less plating of Co	Improvement in Co-conductive network	None	Modest	★★★★	[233]
	Co/Yb hydroxide coating	Improvement in high-temperature performance	None	Modest	★★★★	[234]
C. Additives	Nano-sized Ni(OH)$_2$	Increase in electrochemical reaction reversibility	None	None	★★★★	[235]
	Nano-sized ZnO	Increase in the flexibility of the electrode	None	None	★★★★	[236]
	Co in paste	Formation of conductive Co-network	None	Modest	★★★★	[237–239]
	CoO in paste	Formation of conductive Co-network	None	Modest	★★★★	[110,240]
	Co(OH)$_2$ in paste	Formation of conductive Co-network	None	Modest	★★★★★	[195,241,242]
	CoOOH in paste	Formation of conductive Co-network	None	Modest	★★★★★	[243,244]
	CoSO$_4$ in paste	Formation of conductive Co-network	None	Modest	★★★★	[245]
	Co$_3$O$_4$ in paste	Formation of conductive Co-network	None	Modest	★★★★	[246]
	Co and CaCo$_3$	Prevention of oxygen evolution	None	Modest	★★★★	[247,248]
	CuO in paste	Uniform dispersion of Co-conductive network	None	Negligible	★★★	[249]
	ZnO in paste	Prevention of oxygen evolution	None	Negligible	★★★	[250,251]
	Zn(OH)$_2$ in paste	Prevention of electrode swelling	None	Negligible	★★★	[252]
	Na$_{0.6}$CoO$_2$	Formation of better conductive Co-network	None	Modest	★★★★	[253–255]
	RE	Decrease in oxidation rate of MH alloy	None	Modest	★★★★★	[256–259]
	Y$_2$O$_3$	Decrease in oxidation rate of MH alloy	None	Modest	★★★★★	[23,250,260–262]
	Y(OH)$_3$	Decrease in oxidation rate of MH alloy	None	Modest	★★★★★	[263,264]
	Oxides of heavy RE	Improvement in corrosion resistance	None	Modest	★★★★	[213,265,266]
	Calcium metal borate	Prevention of oxygen evolution	None	Negligible	★★★★	[267]
	CaF$_2$	Improvement in high-temperature performance	None	Negligible	★★★	[116]
	Ca(OH)$_2$	Improvement in high-temperature performance	None	Negligible	★★★	[268,269]
	CaS	Improvement in high-temperature performance	Reacts with acid and releases toxic H$_2$S gas	Negligible	★★★	[270]
	Ca$_3$(PO$_4$)$_2$	Improvement in high-temperature performance	None	Negligible	★★★	[271]
D. Electrode process	Use of sintered electrode	Enhancement in survival rate after long-term storage	None	Reduction	★★★★★	[272]
	Use of pasted electrode on NPPS	Increase in mechanical integrity	None	Reduction	★★★	[273]
	Use of granulated particles	Suppression of electrode swelling	None	None	★★★★★	[274]
E. Substrate	Use of 3D Ni-plated steel sheet	Increase in power and cycle stability	None	Modest	★★★★	[275]
	Use of Ni fiber felt	Increase in surface area and flexibility	None	Modest	★★★★	[276]
	Pre-coating of Co-Ce alloy	Increase in contact area between substrate and Ni(OH)$_2$	None	Modest	★★★	[277]

3.4. Separator

The selection of the separator has a strong impact on the discharge capacity, voltage, and cycle stability [278]. Degradation related to the separator under storage and cycling conditions includes: (1) lower rates of electrolyte permeation in the separator; (2) lower electrolyte holding capability; (3) reduction in the separator volume due to electrode expansion; and (4) reduced gas recombination abilities [96]. Degradations (1) and (2) can be attributed to the debris formed in the separator as precipitation products ($ZnMn_2O_4$) of ions leached from the negative and positive electrodes [23,67]. These deposits not only offer a path for self-discharge, but also reduce the ionic conductivity and electrolyte holding capacity by filling the fine pores in the separator [26,29]. Degradation (3) can be traced to swelling of the positive electrode active material that accompanies over-charging, converting β-NiOOH to γ-NiOOH with Al-contamination leached from the negative electrode [29]. Methods to address separator degradation are listed in Table 7.

Table 7. Summary of cycle stability improvement methods related to the separator. EVOH: ethylene-vinyl alcohol copolymer; and AMPE: alkaline microporous polymer electrolyte.

Method	Direct impact	Environmental impact	Cost impact	Effectiveness	References
Sulfonated separator	Reduction in N-compound shuttling effects	None	Modest	★★★★★	[23,279–282]
Grafted acrylic acid/PP	Improvement in electrolyte holding capability	None	Negligible	★★★	[283]
Polymer gel-type	Improvement in durability	None	Negligible	★★★	[284,285]
Hydroentangled CMC composite	Improvement in integrity	None	Negligible	★★★	[286]
EVOH	Improvement in integrity	Cytotoxic	Modest	★★★	[287,288]
AMPE	Improvement in voltage window	None	Modest	★★	[289]
Addition of a K-conducting solid oxide film	Elimination of cross-contamination from the negative electrode	None	High	★★	New idea

3.5. Electrolyte

The earliest indication of performance degradation is a decrease in cell voltage, which can be traced to a reduction in the amount of electrolyte stored in the separator [50]. Electrolyte losses can be traced to: (1) electrode active material expansion and pulverization, causing an increase in surface area and the wicking of electrolyte away from the separator [54,290]; (2) venting of the cell; and (3) oxidation of metal [98,290]. The contamination in/through the electrolyte is also crucial for the life of both electrodes. Strategies involving the modification of electrolyte that can enhance cycle life performance in Ni/MH batteries are listed in Table 8.

Table 8. Summary of cycle stability improvement methods related to the electrolyte.

Method	Direct impact	Environmental impact	Cost impact	Effectiveness	References
Reduction in KOH concentration	Slow-down in alloy oxidation	None	None	★★★★	[291]
Replacement with NaOH	Slow-down in alloy oxidation	None	Negligible	★★★★★	[292]
ZnO additives	Slow-down in alloy oxidation	None	Negligible	★★★	[293]
LiOH additives	Prevention of K^+ migrating into $Ni(OH)_2$ and suppression of Fe-poisoning	None	Negligible	★★★	[93]
$Al_2(SO_4)_3$ additives	Slow-down in alloy oxidation	None	Negligible	★★★	[91]
NaH_2PO_4 additives	Formation of a Ni-rich surface on MH alloy	None	Negligible	★★★	[294]
$NaBO_2$ additives	Improvement of high-temperature cycle stability	None	Negligible	★★★	[295]
Na_2WO_4 additives	Increase in oxygen evolutionary potential	None	Negligible	★★★	[296]
$K_4Fe(CN)_6$ additives	Prevention of electrolyte decomposition	Highly toxic	Modest	★★★	[297]
Use of gel-type electrolyte	Reduction in corrosion and pulverization in the positive electrode	None	Modest	★★★	[298,299]
Use of polymer electrolyte	Wide voltage window and better mechanical integrity	None	Modest	★★★★	[300–310]

3.6. Other Components

Strategies to improve cycle stability not covered in Sections 3.1–5 are summarized in Table 9, which include charging processes, formation processes, storage conditions, and hardware modifications.

Table 9. Summary of cycle stability improvement methods related to other components. OCV: open-circuit voltage.

Method	Direct impact	Environmental Impact	Cost impact	Effectiveness	References
Install super water absorbing material at cell bottom	Reservoir for additional electrolyte	None	Negligible	★★★★★	[104]
Maintain cell OCV above 1.0 V	Prevention of Co dissolution and migration from the conductive network in the positive electrode	None	None	★★★★	[107,311,312]
Maintain cell OCV above 1.1 V	Prevention of Co dissolution and migration from the conductive network in the positive electrode	None	None	★★★★★	[313]
Reduction of depth of discharge	Prevention of swelling in the positive electrode	None	None	★★★★★	[110]
Reduction of number of shallow depth discharge	Prevention of memory effect	None	None	★★★★★	[314]
Implementation of an improved battery management system	Prevention of abuse	None	Modest	★★★★★	[315]
Pulse charging	Reduction in heat generated	None	Negligible	★★★	[316]
Optimization of formation parameters	Reduction in cell performance variation	None	None	★★★★	[317,318]
Battery sealing under vacuum	Reduction in inner pressure	None	Modest	★★	[319]
Improvement in sealing technology	Prevention of electrolyte leak	None	Negligible	★★	[320,321]

4. Revival of Degraded/Failed Battery

After long-term storage, a few small-current charge/discharge cycles can bring back some of the lost capacity in Ni/MH batteries [322,323]. A more complicated method proposed by Li and Meng [324] involves 33% SOC small-current charge, high-temperature storage (45–60 °C for 20–24 h), and a small current charge/discharge cycle to restore at least part of the lost capacity. Its strategy is to redistribute the Co-conductive network that was destroyed during storage. An alternative method uses ultrasound to disperse active materials from both electrodes in order to create a fresh surface and increase the capacity and power of the used cells [325].

Since the most common failure mode for Ni/MH batteries is electrolyte dry-out, opening cycled cells and refilling with fresh electrolyte can restore the capacity almost to the level before cycling [204]. For re-activation of MH alloy, a patent describes a method of recycling a deteriorated nickel-hydrogen battery by cleaning the cells with a concentrated sulfuric acid containing at least one type of Ni ion, Co ions, and La ions [326]. The concentrated sulfuric acid is poured into the deteriorated nickel-hydrogen battery and maintained at a temperature of 60 ± 10 °C while an electric current is applied to charge the nickel-hydrogen battery. After cleaning, the interior of the nickel-hydrogen battery is filled with an alkaline electrolyte containing a reducing agent. Consequently, γ-NiOOH converts to β-NiOOH, which restores the capacity of the positive electrode, and $RE(OH)_3$, $Al(OH)_3$, $Mn(OH)_2$, and $Co(OH)_2$ dissolve in the concentrated sulfuric acid to activate the negative electrode surface. In addition, the hydrophilic properties of the separator are restored following this method. Recycling used negative electrodes is also possible through the removal of oxide by acetic acid [327]. At the end of usable cycle life, procedures of dismantling, recovery, and reuse of spent Ni/MH batteries have been reported by Nan *et al.* [328,329], Tenorio and Espinosa [330], Bertuol *et al.* [331], Zhang *et al.* [332], Rodrigues and Mansur [333], Muller and Friedrich [334], Rabah *et al.* [335], Santos *et al.* [336], and Larsson *et al.* [337]. U.S. Patents regarding recycling Ni/MH batteries are reviewed in a separate article [2].

5. Conclusions

Various failure modes and capacity degradation mechanisms are reviewed here. Solutions to enhance the cycle stability have been summarized in seven tables covering cell design, negative and positive electrodes, separator, electrolyte, and other hardware. After investigating the capacity-fade issue in a single cell, the next step is to study the consistency in the capacity degradation in a battery module composed of multiple cells.

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