A New Metallurgical Process for Recycling of Discarded Nickel-Metalhydride-Batteries

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Abstract

The demand on Nickel-Metalhydride-Batteries (NiMH) is increasing rapidly since their market launch in the early 1990ies. Nowadays there is no suitable recycling process existing. The discarded batteries are used in the steel industry for alloying because of their Nickel content. Cobalt and Rare Earth (RE) are slagged and lost for recycling. The value of the metals in discarded Ni-MH is estimated at 3-4 €/kg.

In a german governmental funded research project the IME cooperates since Mai 2001 with two industrial partners for a processing and feasibility study. The target of this 3 years project is the development of a new pyrometallurgical recycling process for metal recovery from Ni-MH. After comminution steel and organics are separated. The separated Ni-MH is smelted in an electric arc furnace. Different fluxes are investigated in order to reach best separation of the two phases. First laboratory scale experiments have been successfully implemented. The products are a Nickel-Cobalt alloy and a slag phase enriched with RE-oxides for further treatment to MischmetalBoth the Nickel-Cobalt alloy and the 'Mischmetal' will be directly used in battery production.

1. Introduction

In the recent ten years the demand on light, energy-rich and rechargeable batteries has been grown intensively. This trend is expected to continue due to increasing requirements in relation to mobility within the areas of communication, video and audio applications, and computers. Furthermore the use of rechargeable batteries in the automobile is predicted to increase sharply. Nickel-Metallhydride-Batteries are voltage compatible, high in energy density and compared to Ni-Cad-systems environmental friendly. So far no sustainable closed-loop recycling circle is existing. State of the art is the use of discarded Ni-MH-Batteries in the steel industry using the Nickel content as alloying element. But the contained Cobalt and Rare Earths Metals (RE) are neither used nor recycled. They are lost in the slag. Hence, it is timely to develop a novel technology to enable recycling of all metallic components, thereby also enabling recovery of non-metallic components, such as plastics.

The object of the German government funded work reported here is to develop a simply structured, environmental friendly recycling process for discarded Ni-MH-Batteries. After collection a combination of comminution and metallurgical processing is intended (Figure 1).

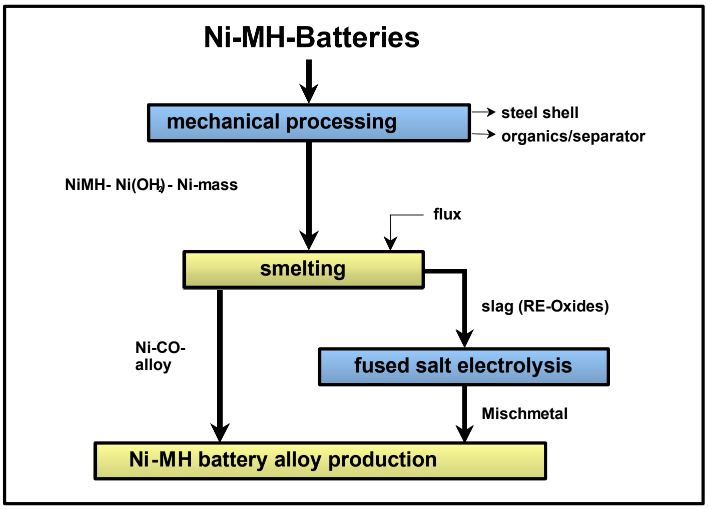


Figure 1: Flowsheet of the intended investigation on NiMH-battery recycling

Within the comminution step it is expected to separate the steel shell and organics from the Ni-MH mass. Using an electric arc furnace and fluxes a Nickel-Cobalt alloy should be generated whereas the Rare Earths Metals should be oxidised and slagged. The Nickel-Cobalt alloy is pyrometallurgically refined and a product directly useable in the battery industry. The slag is processed mechanically and hydrometallurgical in order to transfer the RE-oxides into RE-chlorides, which are fed into a fused salt electrolysis. The produced Mischmetal is also direct usable as a battery alloy component.

2. Market of Nickel-Metallhydride-Batteries

2.1. Battery types, design and applications

Batteries are galvanic elements, where chemical energy is transferred into electrical energy. A battery consists of several, parallel galvanic cells, battery packs contain additionally plastic housings and electronic elements. The cell consist of a positive (cathode) and a negative (anode) electrode, which are embedded in a liquid, pastry or solid electrolyte. If the battery is discharged (=used) the cathode is reduced by electrode consumption, whereas the anode is oxidised. The electrons are leaving the cell at the anode entering the outside current circuit.

In general batteries have to be divided into primary and secondary (or rechargeable, accumulator) batteries. In primary cells the discharge reaction is irreversible contrary to secondary cells. Thus secondary cells can be recharged up to 3000 times depending on their type and mode of use. The most popular accumulator battery systems nowadays are Nickel-Cadmium-, Nickel-Metallhydride-, and Lithium-Ion-Batteries. Typical designs of consumer-type batteries are shown in Figure 2.

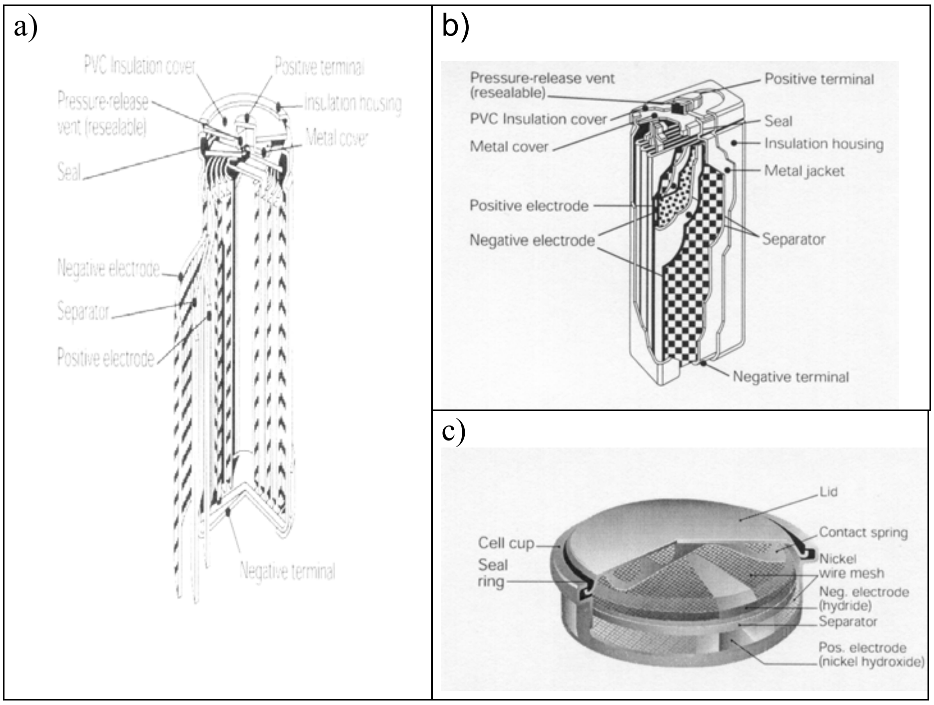


Figure 2: Schematical design of different Ni-MH-Battery types: a) cylindrical cell, b) prismatic cell, c) button cell [3]

Cylindrical cells are used in various applications like walkman, cameras or camcorders. Prismatic cells are designed for use in special devices, where cylindrical cells are limited due to their shape. They are mostly used as battery packs, for instance in mobile phones. Button cells are adopted to computer, phones or televisions devices for bridging operations [1, 2].

2.2. Components of Ni-MH-Batteries

The main parts of a the battery are an aqueous alkaline electrolyte, the positive and negative electrode, separator and cell cup with lid. The positive electrode is consisting of Cobalt doted Nickel(II)-hydroxide, which is oxidised during charging to Nickel-oxi-hydroxide. The negative electrode can based on a nickel-plated, punched steel sheet or Nickel-foam. The sheet is plated, the foam impregnated with mixture of a hydrogen-storing-alloy/Teflon binder/Carbon- or Nickel-powder.

Two intermetallic alloy types are used for hydrogen-storage in batteries. Due to their larger stability and lower self-discharge is AB_5 the most common, very rare is AB_2. A is derived from IIIa, IVa group metals and B is derived from Va, VIa, VIIa, VIIIa group metals. For

AB_5 $LaNi_5$ is mainly used, but improved by substitution of Nickel with Cobalt, Manganese and Aluminium [4]. Lanthanum is partly replaced by the cheaper Mischmetal (Mm), composed e.g. of 50-55 % Cerium, 18-28 % Lanthanum, 12-18 % Neodymium and 4-6 Praseodymium. A typical alloy nowadays is $MmNi_{3,5}Co_{0,7}Mn_{0,4}Al_{0,3}$.
A typical AB_2 alloy is represented by $Zr(Mn_{0,25}Cr_{0,1}V_{0,05}Ni_{0,6})_2$. The separators between the electrodes are made of alkaline resistant plastics, mainly PA or PP. The cell cup is consisting of Nickel or nickel-plated steel, which is resistant in alkaline solutions. [6] The electrolyte is mainly diluted potassium hydroxide (20-30 % KOH) containing some parts of Lithiumhydroxide. The reactions occurring at the electrodes are:

$$\text{Negative Electrode:} \qquad MH + OH^- \quad \overset{disch\arg e}{\underset{ch\arg e}{\rightleftarrows}} \quad M + H_2O + e^- \tag{1}$$

$$\text{Positive Electrode:} \qquad NiOOH + H_2O + e^- \quad \overset{disch\arg e}{\underset{ch\arg e}{\rightleftarrows}} \quad Ni(OH)_2 + OH^- \tag{2}$$

$$\text{Overall:} \qquad MH + NiOOH \quad \overset{disch\arg e}{\underset{ch\arg e}{\rightleftarrows}} \quad M + H_2O + e^- \tag{3}$$

During discharge atomic hydrogen is disorbed at the negative electrode and reacts with OH^- ions (released at the positive electrode) forming water. This process is reversible and allows thousands of charge/discharge-cycles.

2.3. Market situation and economical potential

In the recent years the production of Ni-MH-Batteries has grown steadily (Figure 3a). Nowadays they are established in the market in various applications. About 5200 t Ni-MH-Batteries were sold within Europe in 2000 including 1699 t in Germany (Figure 3b) [6]. For further information see [7]

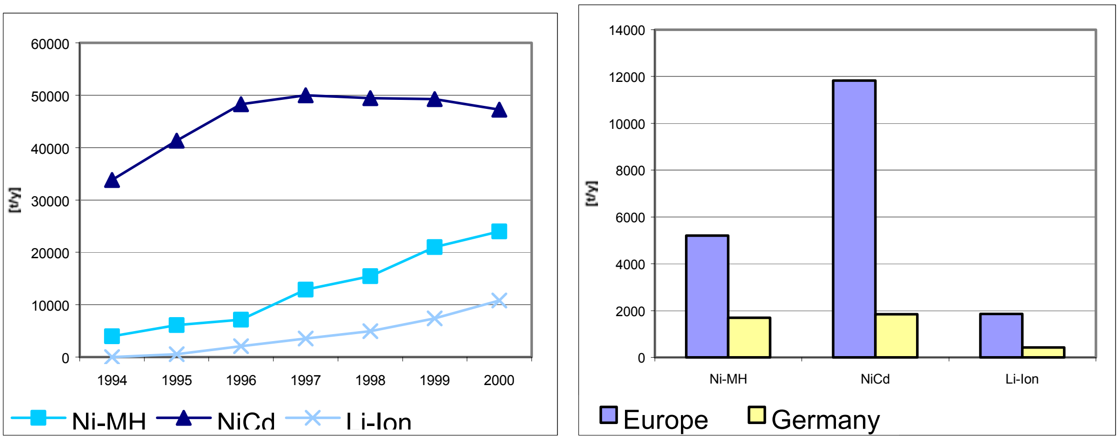


Figure 3: a) World battery production (left), b) sold batteries in 2000 (right) [6, 7]

The most important criterion for the evaluation of recycling processes is the raw materials situation and the raw materials price of the recycled material. According to today's knowledge the reserves of Nickel, Cobalt and Rare Earth metals are sufficient for the next 100 years [8,9]. But recycling is economically interesting due to the relatively high metal prices of Nickel, Cobalt and Rare Earth metals. In order to determine the economical content of Ni-MH-battery scrap the composition are summarized in Table 1:

Table 1: Composition of Ni-MH-cell scrap [6]

component	**mass percentage %**			
	AB_5-button cell	**AB_5-cyln. cell**	**AB_5-pris. cell**	**AB_2-cyln. cell**
Ni	29-39	36-42	38-40	37-39
Fe	31-47	22-25	6-9	23-25
Co	2-3	3-4	2-3	1-2
La, Ce, Nd, Pr	6-8	8-10	7-8	-
Zr, Ti, V, Cr	-	-	-	13-14
Carbon black, Graphite	2-3	<1	<1	-
Organics	1-2	3-4	16-19	3-4
Potassium	1-2	1-2	3-4	1-2
Hydrogen and Oxygen	8-10	15-17	16-18	15-17
others	2-3	2-3	3-4	1-2

The metal value of 1 kg Ni-MH-Batteries (type AB_5) is represented in Table 2. Base of the calculation are the mass percentages of Table 1.

Table 2: Value contained in 1 kg Ni-MH-round cell consumer battery-scrap (type AB_5)

component	proportion in AB_5-cyln. cell [mass-%]	metal price [€/t] [15]	value [€/kg]
Ni (January 2002)	36-42	5888	2,12-2,47
Co (January 2002)	3-4	20956	0,63-0,84
Mischmetal (January 2000) (La, Ce, Nd,Pr)	8-10	6716	0,54-0,67
sum			3,29-3,98

Taking the mass of sold batteries in 2000 (Figure 3b) the total economical potential of Ni-MH-battery scrap is calculated to 5.6-6.7 million Euro for Germany respectively 17.1-20.7 for Europe.

3. State of the art in Ni-MH-Battery recycling

3.1. Assumption for recycling

Recycling of used products and materials is demanded by the EU-politics because of a raised environmental consciousness in the recent years. Objectives of recycling are to relieve the environment, safe raw materials and energy and to reach a high recycling rate of valuable materials. From today's point of view recycling is less a technological problem but an economical. In particular metal recycling is depending on economical variations, which often leads to bankruptcy of recycling companies. With expected increasing costs for depositions of wastes the need for recycling is obvious. But recycling has to be always economically feasible.

Assumptions for the mentioned requirements with respect to a recycling process for Ni-MH-Batteries are:

- Knowledge of present and estimation of future rate of returns for discharged Ni-MH-Batteries by installation of suitable collection systems
- sufficient purity of the scrapped battery by sorting technologies to avoid disturbing impurities

- fundamental knowledge about directives, laws, ordinances, regulations
- estimation of future raw material situation on world market
- feasibility study of recycling process and comparison with primary production

A recycling concept for Ni-MH-Batteries is expected to be profitable, if it recovers Nickel, Cobalt and RE-Metals for direct re-use in battery production (closed loop process). A minimum of process steps and of non useable by-products like plastics ore waste water has to be guarantied.

3.2. Hydrometallurgical recycling processes

Several hydrometallurgical recycling processes were investigated. The most important processes are describe by Yoshida [10], Lyman et al. [11], Pietrelli et al.[12] and Varta [13]. In all described processes the first step is mechanical processing. Subsequently different leaching and solvent extractions steps are necessary in order to obtain Rare Earth metals and Nickel and Cobalt dissolved. The resulting RE salts are useable in a fused salt electrolysis whereas Nickel and Cobalt are won via precipitation, ion-exchange or electrowinning. All described processes were investigated in laboratory scale without transfer into pilot plant tests.

3.3. Pyrometallurgical recycling processes

A closed loop recycling process for Ni-MH-Batteries is not existing. At the moment Nickel and the steel shell of the batteries are open loop recycled in the steel industry. Cobalt is neither paid nor recovered. The RE are ending in the slag. Besides this no recycling processes or research projects are described in literature.

4. Experimental Methodology

4.1. Comminution

The main design criteria for the comminution process is to obtain a powder-fraction not polluted by iron. A shredder with one rotor and equipped with perforated discharge was used for crushing the batteries. The ground material was wet screened, obtaining a fine fraction with the valuable components Ni, Co, and SE and an oversize fraction consisting of a mix of iron chips, Fe-based-electrodes grids, plastic chips, paper, and flocculent material of separators. After shredding the steel shell and the various fractions of battery materials were carefully separated by mechanical processing. The processed material was supplied by a project partner (UVR-FIA GmbH, Freiberg, Germany).

4.2. Slag systems

Any pyrometallurgical process relies on a suitable slag system. In this specific case it has to allow separation of Nickel and Cobalt as metal from the RE almost quantitavely collecting the RE as well as unnoble impurities e.g. as oxides. Alternatively the RE can be forced to react with compounds or elements into chlorides or fluorides, which might be more suitable than oxides for the subsequent slag treatment and fused salt electrolysis.

The important demands of the slag system are:

- poor solubility for Nickel and Cobalt

- high solubility for RE as oxide, chloride or fluoride
- density significant smaller than metal phase density (< 5 g/cm^3)
- low viscosity to allow an easy slag/metal separation
- moderate melting temperature (< 1450 °C)

Several slag systems are suitable to the mentioned assumptions. A slag system with 45 % SiO_2/40 % CaO/15 % MgO has a melting point at 1400°C, a density of 2,6 g/cm^3 and a viscosity between 1,8 and 2,5 poise [14]. Pure silica flux is also suitable. Between 1400°C and 1700°C RE-silicates are generated. These RE-silicates are later extractable with hydrochloric acid in order to chlorinate RE-oxides.

Most of metal chlorides and fluorides have got low melting and boiling points. Thus they are not suitable as slag base for the expected working temperature of 1500-1700 °C. Calcium fluoride (Bp 2513°C) and Calcium chloride (Bp 1600 °C) may be useable as fluxes in order to generate RE-fluorides respectively RE-chlorides during the melting process. The boiling point of RE-fluorides is about 2300°C and for RE-chlorides between 1700 and 1800 °C. Within expected working temperature area the RE-chlorides respectively RE-fluorides can not be evaporated selectively. Alternatively a mixture of Calcium fluoride and Calciumoxide has potential, as the mixture has got an eutectic melting point of 1360°C (for 15 % CaO, 85 % CaF_2).

4.3. Metallurgical Processing

Experiments Tamman Furnace

Trial tests were made in a Tamman-type furnace (Figure 4, left). The experiments were performed in order to screen the slag systems and to find a suitable crucible-material, which resists the aggressive conditions of chlorides and Rare Earth Metals. The input was consisting of pure negative electrode mass originated from production wastes. Type and mass of additive slag was varied as represented in Table 3. Pouring took place always at 1600 °C measured with a EL18 thermocouple. Different crucibles were tested (graphite, MgO, Al_2O_3).

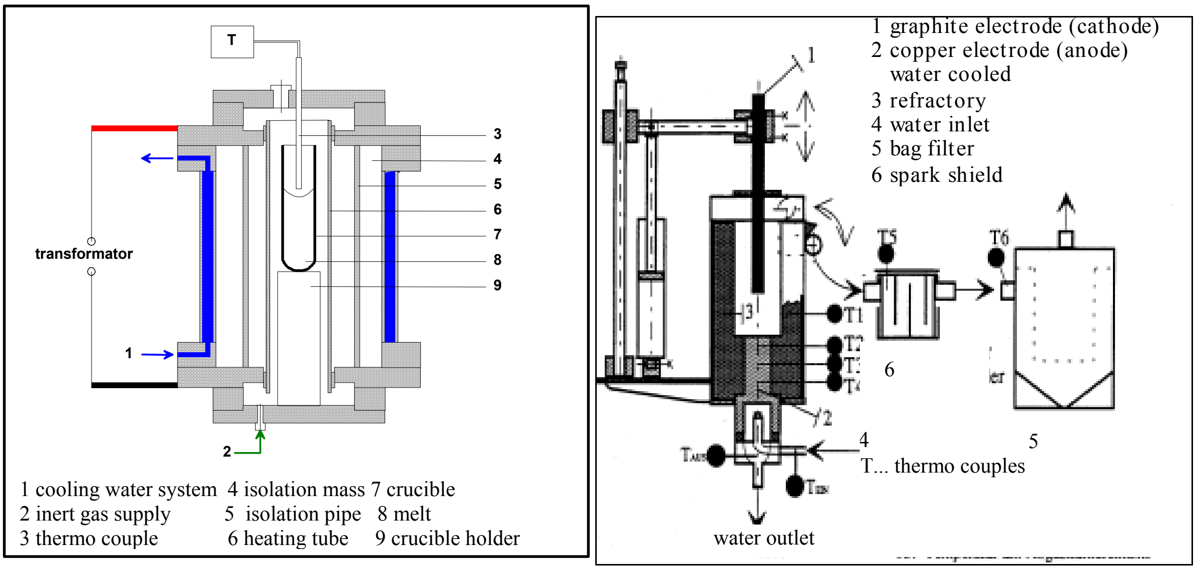


Figure 4: Principle sketch of Tamman furnace (left), electric arc furnace (right)

Table 3: Overview Tamman furnace experiments

exp.	flux composition					charge composition	
	SiO_2	CaO	MgO	CaF_2	$CaCl_2$	flux	Ni-MH
1	45 %	40 %	15 %			33 %	67%
2	45 %	40 %	15 %			29 %	71 %
3	45 %	40 %	15 %			26 %	74 %
4	45 %	40 %	15 %			15 %	85 %
5	45 %	40 %	15 %			23 %	77 %
6	45 %	40 %	15 %			18 %	82 %
7				100 %		23 %	77 %
8				100 %		19 %	81 %
9				100 %		16 %	84 %
10		40 %		60 %		19 %	81 %
11		40 %		60 %		20 %	80 %
12	100 %					11 %	89 %
13	100 %					15 %	85 %
14	100 %					10 %	90 %
15					100 %	21 %	79 %
16					100 %	23 %	77 %

Experiments electric arc furnace

Table 4: Overview electric arc furnace experiments

exp.	flux composition				charge composition			pouring temperature	crucible
	SiO_2	CaF_2	CaO	MgO	flux		Ni-MH	[°C]	
1	100%				25%		75%	1550	MgO
2	100%							1700	SiC
3		100%						1350	
4		100%						1420	
5		65%	35%					1555	
6		65%	35%					1592	
7	45%		40%	15%				1450	
8	45%		40%	15%				1415	
		CaF_2	CaO		CaO	CaF_2	Ni-MH		
9		84,20%	15,80%		7,50%	40,00%	52,50%	1420	graphite
10		87,00%	13,00%		7,50%	50,00%	42,50%	1392	
11		84,20%	15,80%		7,50%	40,00%	52,50%	1520	
12		100,00%				50,00%	50,00%	1360	
13		66,70%	33,30%		15,00%	30,00%	55,00%	1430	
14		76,90%	23,10%		15,00%	50,00%	35,00%	1360	
15		84,20%	15,80%		7,50%	40,00%	52,50%	1650	
16		100,00%				30,00%	70,00%	1480	
17		100,00%				40,00%	60,00%	1360	
18		80,00%	20,00%		7,50%	30,00%	62,50%	1290	
19		72,70%	27,30%		15,00%	40,00%	45,00%	1540	
20		84,20%	15,80%		7,50%	40,00%	52,50%	1420	

The most promising slag systems (Table 4) were investigated in a lab-scale electric arc furnace (Figure 3, right). Objective was to evaluate the technical feasibility of these slag systems in a larger volume of scale. The Ni-MH mass was briquetted with the flux (25 % of charge) in order to avoid dust losses. The total input was 6500 g for each trial. Two different furnace refractories (MgO, SiC) were used, which were identified to resist the chemical aggressive impact of RE. A special emphasis was given to the CaO-CaF_2 system (Table 5). As refractory wear was too high for MgO and SiC a graphite crucible was mounted into the furnace. Total input mass was 5000 g. The temperature was measured with a commercial lance. Samples were taken from the cast metal as well as from the slag. Two trials were executed for each slag system.

5. Results and discussion

5.1 Tamman furnace

Only the graphite crucible is resisting the thermal and chemical stress. But wear is still occurring after several uses. The crucibles made of MgO and Al_2O_3 are not suitable. According to the chemical reaction

$$2\ RE + MeO \rightarrow RE_2O_3 + 3\ Me \qquad (4)$$

the lining oxides (MeO) were reduced by the rare earth metals as it can be expected thermodynamically.

The evaluation of the experiments showed a good separation of metal and the corresponding slag composition is suitable in regard to viscosity. For selected slag systems the final metal and slag analysis are given in Figure 4.

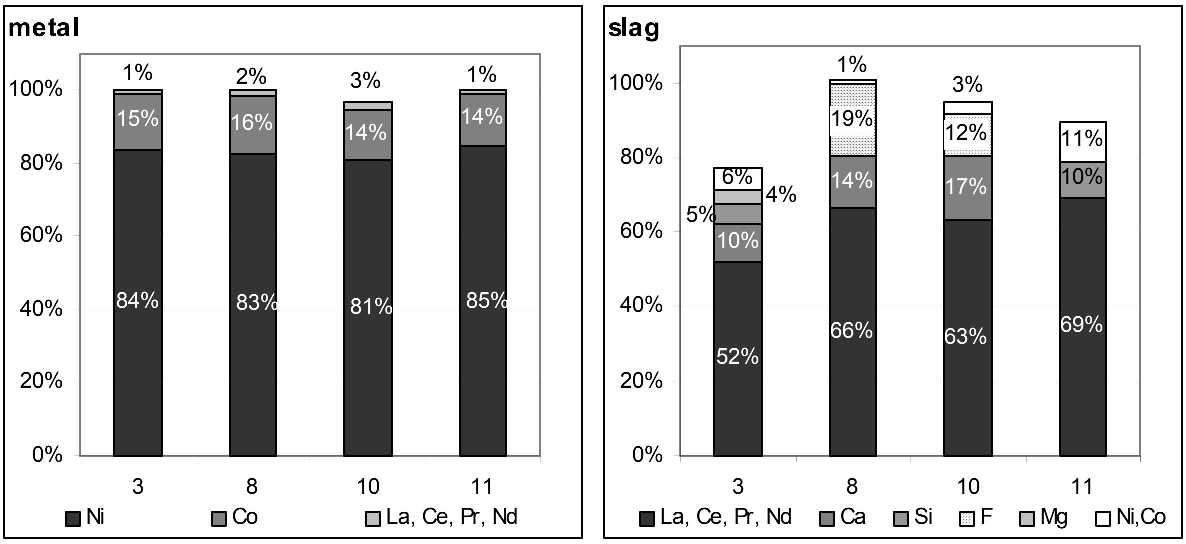


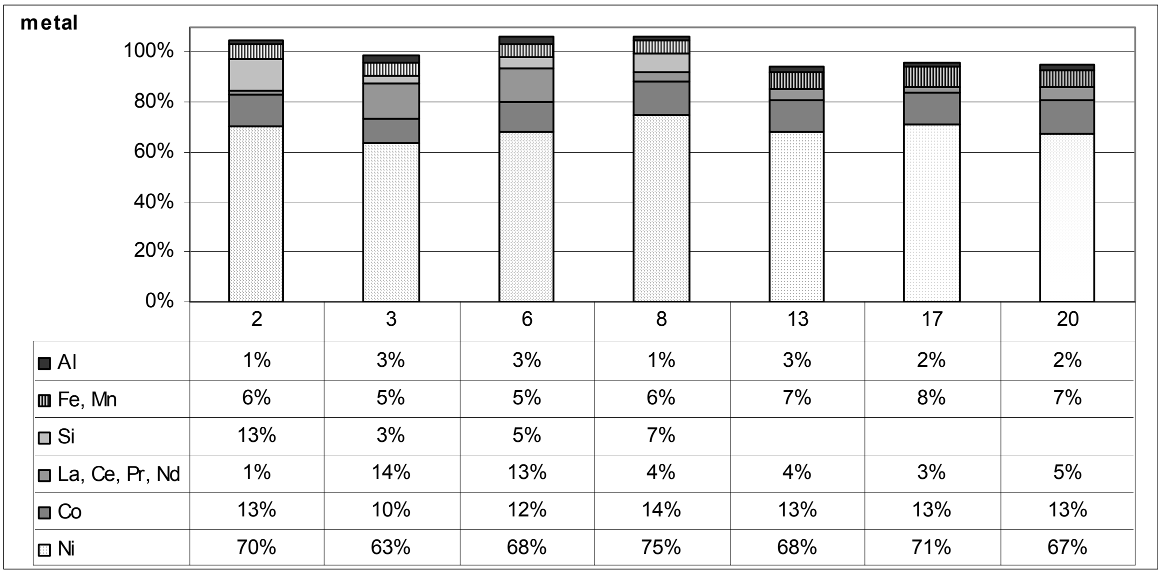
Figure 4: Metal and slag compositions for selected Tamman-furnace experiments

Nickel and Cobalt are mainly collected in the metal phase whereas RE end up in the slag phase. Slag systems containing SiO_2 and/or MgO have got a higher Nickel content in slag in comparison to the CaF_2-CaO systems. The SiO_2 and/or MgO systems are expected to have a higher viscosity and thus the separation by gravity is hindered. Furthermore the volume of

flux (slag/metal-ratio) must be sufficient in order to secure separation. From these experiments it can be shown that at least 22,5 % flux are needed for metal-slag separation.
The trials with chloride-based slags are not successful. At temperatures larger than 1500°C calcium chloride is already evaporated due to its high vapor pressure. Intensively smoke development was observed during these experiments. A separation was not visible.

5.2 Electric Arc Furnace

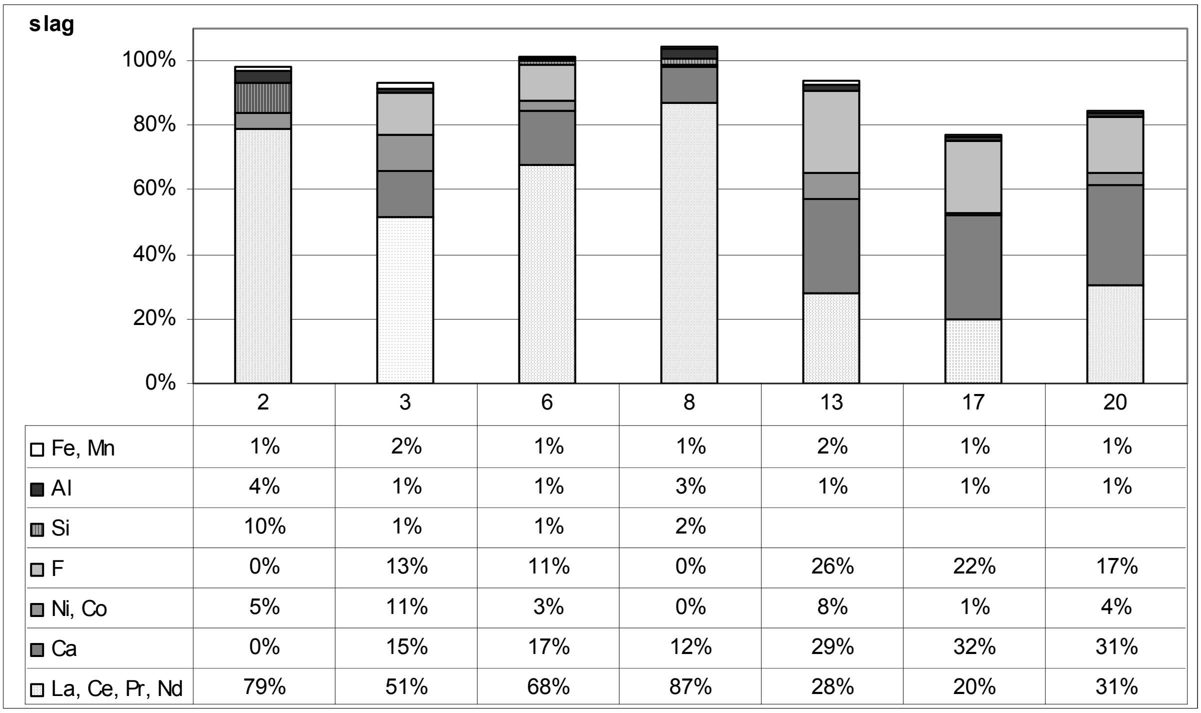
With the slag systems used in experiment 1 to 7 (Table 4) the objective of metal-slag separation could not be performed. A clear separation was failed. In exp. 1 just a single phase was recovered. The melt was high viscose. Decreasing of viscosity by increasing the temperature was not successful. Part of the melt remained in the furnace after pouring. The tested refractory materials were heavily weared by the slag. The exp. 7 and 8 showed expected results. The low viscous melt was poured easily and a clear separation of metal and slag after solidification was observed. The analysis shows for this two experiments the best results follows by experiments 1 and 2 (Figure 5+6).



	2	3	6	8	13	17	20
■ Al	1%	3%	3%	1%	3%	2%	2%
▥ Fe, Mn	6%	5%	5%	6%	7%	8%	7%
□ Si	13%	3%	5%	7%			
■ La, Ce, Pr, Nd	1%	14%	13%	4%	4%	3%	5%
■ Co	13%	10%	12%	14%	13%	13%	13%
□ Ni	70%	63%	68%	75%	68%	71%	67%

Figure 5: Metal analysis of selected electric arc furnace experiments (best results)

The high silicon content in bullion of experiments 1 to 8 shows (Table 4, Figure 5), that silicon is also reduced and dissolved in the metal phase, which makes a subsequent refining step necessary to meet battery alloy specifications. Thus a silicon free refractory is recommended in order to fulfil process cost and purity requirements.



	2	3	6	8	13	17	20
□ Fe, Mn	1%	2%	1%	1%	2%	1%	1%
■ Al	4%	1%	1%	3%	1%	1%	1%
▥ Si	10%	1%	1%	2%			
□ F	0%	13%	11%	0%	26%	22%	17%
▩ Ni, Co	5%	11%	3%	0%	8%	1%	4%
▩ Ca	0%	15%	17%	12%	29%	32%	31%
□ La, Ce, Pr, Nd	79%	51%	68%	87%	28%	20%	31%

Figure 6: Slag analysis of selected electric arc furnace experiments (best results)

In exp. 1 the separation of slag and metal was failed. In exp. 4 to 7 the high RE content in metal phase is obvious as well as a the Nickel content in the slag phase. It is assumed that the flux ratio is to less. The chosen ratio of 25 % is close to the lower limit as proven in the Tamman furnace trials. The trials 9-20 with modified CaO-CaF_2 ratio as well as increased flux/metal-ratio showed an improved melting and pouring behaviour. Transferring Nickel and Cobalt in metal on the one hand and on the other hand to collect RE in the slag phase could be reached best in trial no. 12 and 17, where pure CaF_2 flux was used.

Table 5: Distribution coefficient for selected experiments

exp.	Ni_{met}/Ni_{slag}	Co_{met}/Co_{slag}	RE_{met}/RE_{slag}
2	16	22	0,02
3	7	6	0,28
6	29	19	0,19
8	490	135	0,05
13	9	13	0,16
17	177	98	0,13
20	20	58	0,18

The experiments with CaO-CaF_2 flux did not led to a suitable slag system. Metal losses in slag are too high as well as the RE content in the Ni/Co alloy(Table 5). Furthermore the high fluoride content in the slag causes difficulties in the subsequent leaching step due to chemical reaction with hydrogen. A fluoride containing flux is not suitable in the process.

6. Next Steps

The so far investigated slag systems have to be further optimised to fulfil the requirements of the project. Systematically further slag systems like Al_2O_3-CaO or boride-based systems will be investigated in order to reach higher distribution coefficients. Selective oxidation will be used to purify the Ni/Co-alloy. Based on this alloy a recycled Ni-MH-Battery alloy will be produced and tested. Project partners will extract RE from the slag and transform them into Mischmetal.

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