Processing of Grinding Slurries Arising from NdFeB Magnet Production

Stephanie Kruse[1],*, Karoline Raulf[2], Anna Trentmann[1], Thomas Pretz[2], and Bernd Friedrich[1]

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Neodymium-iron-boron (NdFeB) grinding slurries are the residual output of grinding as shaping process step in the production process of sintered NdFeB magnets. The treatment of the grinding slurries with the aim of recovering rare earth elements may lead to a reduced demand in primary raw materials like the rare earth elements neodymium, praseodymium and dysprosium. In this study, the possibility of recycling the contained rare earth elements, which account for up to 30 wt % in the alloy, through a pyrometallurgical process was investigated. The necessity of a thermal conditioning prior to the pyrometallurgical process is described.

Keywords: Grinding slurries, NdFeB magnet, Rare earth elements, Recycling

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1 Motivation and Recycling Concept

Due to an increasing amount of rare earth elements (REE) needed for high tech applications combined with the supply risk of those elements from primary resources, strategies for recovering REE from scraps are essential for providing a stable supply. To this day, more than 95 % of REE needed in Europe is imported from China. Reasons for this are missing recycling concepts for the arising waste fractions, as well as primary resources that are economically exploitable.

Due to their high saturation magnetization and high magnetic polarization, neodymium-iron-boron magnets are among the strongest available permanent magnets. The high amount of REE contributes to the value of residues which occur as production waste during manufacturing. One of the most common residues in production is the grinding slurry or grinding sludge that arises within the grinding process of the sintered magnets. Up to 5 % of the magnetic alloy end up in this sink. In this investigation, the possibility of a pyrometallurgical treatment of such grinding slurries should be determined. Since REE have a high oxygen affinity, the main focus of the pyrometallurgical treatment is to separate REE from the iron by oxidizing and enriching them in a slag phase. Still, a thermal preconditioning of the grinding slurries is necessary as they contain remainders of liquid cooling agents. The cooling agent is an additive in the grinding process complying two crucial functions. First, it cools down the workpiece in the grinding point in order to avoid damaged spots on the workpiece surface evoked by high temperatures at the grinding point. Secondly, the cooling agent evacuates the grinding swarfs from the working area.

2 Background

This investigation aims at recovering REE from NdFeB magnet material in form of RE oxides. The thermal preconditioning assures the decomposition of the cooling agent and, therefore, avoids problems in process control within the pyrometallurgical process step. This step comprises the melting of the pyrolyzed grinding slurries and separation of REE as oxides from a cast iron phase. The oxygen needed for the oxidation is already contained in the raw material, as well as some amount of carbon.

2.1 NdFeB Magnets

The strong magnetical capacities of NdFeB magnets result from the main $Nd_2Fe_{14}B$ phase in the magnet structure. The matrix of the magnet consists of $Nd_2Fe_{14}B$ grains. Besides these grains, the structure contains phases of enriched neodymium, oxides and phases of enriched boron, which can be seen in Fig. 1.

During the sintering process the neodymium-rich phase and the boron-rich phase in between these grains are in equilibrium. During solidification both phases attach to the $Nd_2Fe_{14}B$ grain boundaries. The attachment is important for a decoupling of the grains as mutual interaction between single grains must be prevented. The distribution of the neo-

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[1] Stephanie Kruse (skruse@ime-aachen.de), Anna Trentmann, Prof. Dr. Dr. h.c. Bernd Friedrich, RWTH Aachen University, IME Process Metallurgy and Metal Recycling, Intzestraße 3, 52056 Aachen, Germany; [2] Karoline Raulf, Prof. Dr.-Ing. Thomas Pretz, RWTH Aachen University, I.A.R Department of Processing and Recycling, Wüllnerstraße 2, 52056 Aachen, Germany.

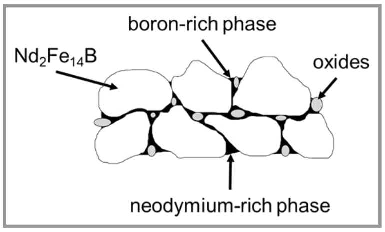

Figure 1. Structure of NdFeB magnets [5].

dymium-rich phase on the grain boundaries can be positively influenced by the addition of aluminum or gallium [2, 3].

Due to its lower price praseodymium is used as replacement for neodymium. As long as the proportion of Nd:Pr in the magnet does not pass the dimensions of 3:1, the magnetical characteristics are not influenced [3].

Pure NdFeB magnets have low Curie temperatures from 80 °C to 100 °C, which can be increased by additives. Heavy-rare earths like the elements terbium and dysprosium can significantly raise the temperature stability. The addition of 10 wt % of dysprosium can extend the temperature stability up to 200 °C. Terbium can affect the coercivity even stronger than dysprosium, but has a higher price and a lower availability [4]. The resistance towards corrosion can be influenced by the addition of cobalt or by coating the magnets with organic (resin) or metallic coatings (Al, Ni, Zn, Sn) [4].

The conventional production route for sintered NdFeB magnets consists of vacuum induction melting, followed by crushing and milling as well as pressing and sintering of the magnet material. Nd-Fe and Fe-B master alloys are used as input material. The process route is shown in Fig. 2.

The obtained ingot from melting of the alloys is crushed and grinded to < 5 µm, before the monocrystals can be aligned in a magnetic field. Subsequently, the pressing and sintering process takes place to adjust the favoured texture. During the next step, the material is grinded into its final shape. Within this process, the grinding slurries, which are the main issue of this paper, occur. Finally the material is magnetized [1, 2].

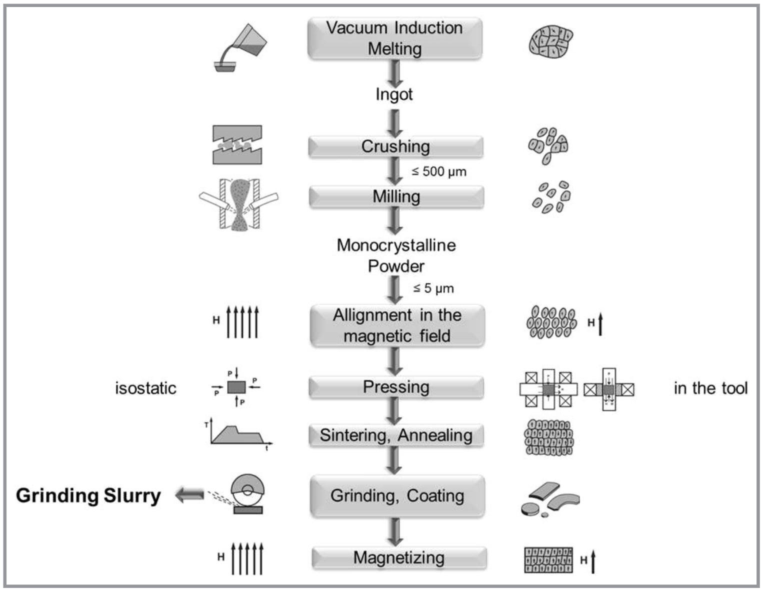

Figure 2. Flow sheet for the production of NdFeB sintered magnets [1, 2, 5].

2.2 Grinding Slurries

Grinding slurries are a mixture of metallic swarfs, abrasions of the grinding media and a relevant amount of cooling agent. As depicted in Fig. 3 an on-site solid-liquid separation is applied for the direct circulation of the applied cooling agent. Still, the amount of remaining cooling agent in the grinding slurries varies between < 1 % and > 20 %. The most current methods for solid-liquid separation are magnetic separation or filter systems.

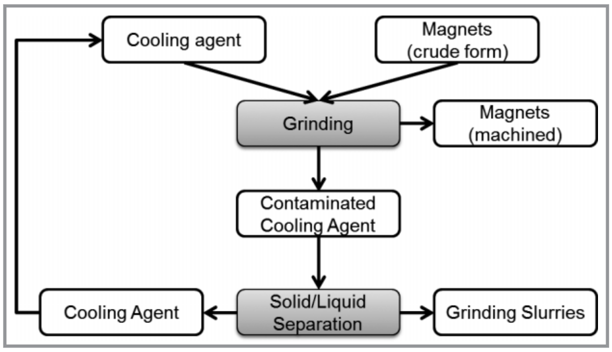

Figure 3. Grinding process: generation of grinding slurries and circulation of cooling agent.

Since the alloy is very hard, diamond abrasives are usually used for grinding. The amount of abrasions of the grinding media remaining in the grinding slurry is not considered to be significant for the material composition as the amount of carbon added by the cooling agent is far higher. Due to the contamination with cooling agent the produced slurries have a different and more inhomogeneous composition than the original magnet. An exemplary composition is given in Tab. 1 [6].

Other than in the machined magnet, the oxygen and carbon content is much higher. Carbon is introduced to the system mostly because of the organic cooling agent, whereas oxygen is also inserted from the ambient air since temperatures of up to 1000 °C [10] occur during grinding. Furthermore these amounts show significant variations depending on the composition of magnet and cooling agent as well as the physical treatment before pyrolysis. Due to the high variations in the composition and the deviation from the original magnet composition, the grinding slurries cannot be directly recycled inside of the production process.

Table 1. Exemplary composition of a grinding slurry according to Asabe [6].

Element	Contents [wt %]
Nd	19.5
Fe	Balance
B	0.8
Dy	2.4
Pr	4.9
Al	0.2
C	1.3
O	5.6

In Europe, there is no established industry for the recycling of NdFeB magnet grinding slurries. Most commonly, grinding slurries are stored and exported to China for recycling. If not, grinding slurries are either disposed as hazardous waste (due to the remainders of oily components of the grinding slurry) or tempered with other metal scraps in order to recycle the blend as aggregate in melting processes or in the concrete industries.

Tanaka et al. [7] propose the recycling process shown in Fig. 4 for NdFeB magnet grinding slurries. The grinding slurries are roasted in order to completely oxidize the grains after the physical pretreatment and then directly leached in order to separate iron and boron. Further, solvent extraction and reduction processes are applied for recovering the rare earth metals.

In order to significantly reduce the input to the hydrometallurgical processing route, in this investigation a pyrometallurgical enrichment of REE prior to the hydrometallurgical processes is executed [7].

2.3 Thermal Pretreatment

The main impurity hindering a direct remelting of the grinding slurries is the remaining amount of cooling agent. Due to volatile matters and the adverse influence of their vaporization within the pyrometallurgical process, a pretreatment is necessary in order to avoid problems with the process control. In addition, the metal swarfs as single particles in the grinding slurry tend to form agglomerates due to their particle size and shape. These agglomerates incorporate parts of the remaining cooling agent.

Minimizing the amount of organic and volatile substances contained in the cooling agent and destructing the existing agglomerates in order to separate the particles and gain access to the incorporated cooling agent are the main requirements for a preconditioning process prior to pyrometallurgical treatment. A thermal treatment via pyrolysis fits these requirements.

Pyrolysis is an endothermic process in which anaerobic conditions are used to decompose organic material. In consequence of the rise of temperature during the process caused by continuous external supply of energy, evaporation processes and thus drying of the material occur first. After that, degassing and chemical decomposition processes take place. They are divided into the phase of charring and the phase of gas formation [9].

The formed products depend on the process temperature and the input material:
- coke,
- tars, oils, condensable hydrocarbons,
- water released by decomposition (the aqueous condensate) and
- non-condensable pyrolysis gases [9].

Together with the remaining inert material, the coke forms the solid residue. In the condensate tars, oils, condensable hydrocarbons and the water released by decomposi-

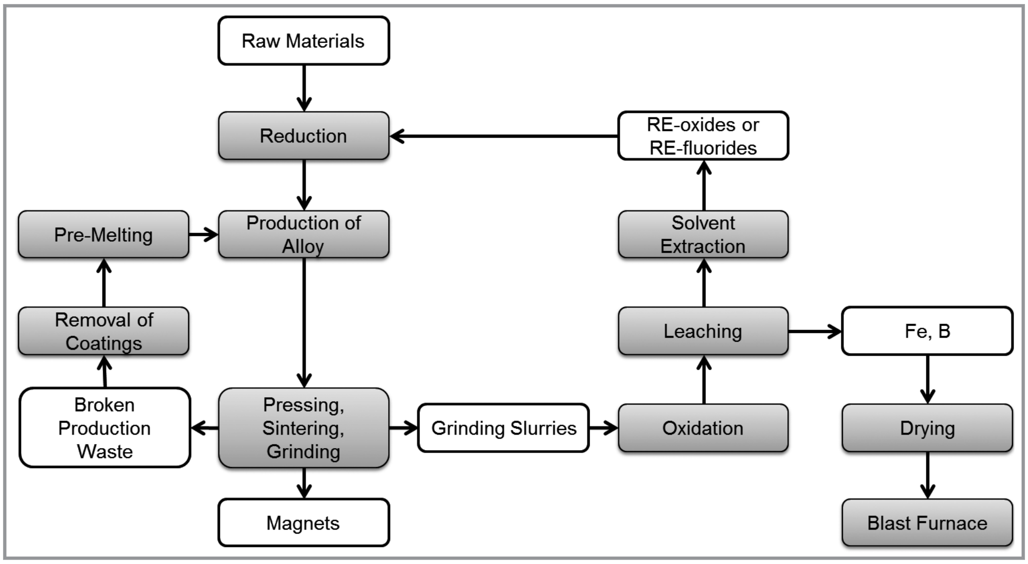

Figure 4. Recycling of production waste according to Tanaka [7].

tion remain. The remainder escapes as pyrolysis gas. The composition of the pyrolysis gas is specific depending on the composition of the input material and the interaction of all occurring processes, namely drying, evaporation, degassing and chemical fissions. Typical pyrolysis gases are, e.g., CO, CO_2, H_2S, H_2 and NH_3 [9].

The performance of pyrolysis processes depends highly on temperature and residence time. Additionally, the heating velocity of the input material is relevant as the specific heat transition coefficient and the surface of the material are crucial factors for the heat transfer [10]. The pyrolysis process aims to concentrate the metal swarfs in the solid residue while volatile components end up either in the liquid condensate or in the gas phase. Carbon remaining in the solid phase is been solidified.

2.4 Melting and Selective Oxidation

Selective oxidation is commonly used for separating elements with strongly differing oxygen affinities. Considering the activity of each component the elements with the lowest reaction enthalpy of the oxidation reaction are oxidized. Regarding the pure phases, REE are likely to build oxides since they have the highest oxygen affinity except for calcium. Thus, during melting the REE are separated as oxides from a metallic phase mostly consisting of iron. The phase diagram for the system Nd-Fe-O at 1077 °C is shown in Fig. 5 [11].

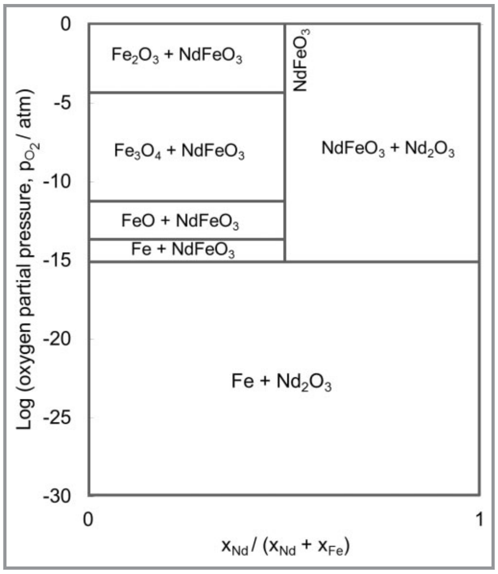

Figure 5. Phase diagrams of the systems Nd-Fe-O [11].

Irrespective of the neodymium content, two phases are present at low partial pressures of oxygen. Up to a mole fraction of 0.5 iron oxides and $NdFeO_3$ compounds are formed at higher oxygen partial pressures. From that content, Nd_2O_3 and $NdFeO_3$ are formed. The diagram indicates that the oxygen partial pressure has to be kept low to avoid the formation of iron oxides or mixed oxides. The authors, therefore, propose the usage of a carbon crucible and the addition of a carbon powder [11]. In the diagram the oxygen content is not taken into account concerning the composition [11, 12].

Most investigations aim at lowering the oxygen content in the material as far as possible in order to realize a circuitry of the magnet material [13]. This is why there is no investigation on the amount of oxygen needed for a selective oxidation of REE and a phase separation between two phases of which at least one is molten. Based on a content of 30 wt % REE in the grinding slurry an amount of approximately 4.5 wt % of oxygen would be necessary to completely oxidize the REE [13]. However, the experiments conducted in this paper indicate that melting and phase separation are facilitated by higher oxygen contents. The carbon in the grinding slurries accounts for the decreasing of the melting point as well as reducing iron oxides to iron in case of an excessive amount of oxygen in the material [11].

3 Material and Methods

3.1 Characterization of the Grinding Slurry

The sample material used for the investigation originates from a grinding process realized in the laboratory in order to be able to reproduce and relate to all machining parameters as grinding media, cooling agent and cutting speed. Since diamond tools are state of the art to machine rare earth material, the grinding was performed with an epoxy-resin-bonded diamond grinding wheel (grain size: 100/120 mesh, grain concentration: 3.3 ct cm^{-3}). The cooling agent is based on mineral oil and boric acid and water-miscible (Mobil™ Mobilcut 230). It was applied at a concentration of 5 % and with a throughput of 30 – 50 L per minute and wheel. The grinding slurries were obtained as solid remainder of a solid-liquid separation of the contaminated cooling agent by vacuum filtration. The magnets used for the investigation were different batches of scrap NdFeB magnets.

The generated grinding slurries were analyzed regarding their chemical composition. Beside the elemental analysis (method ICP-OES), the moisture content (analysis in dependence on DIN 51718) and the volatile content (according to DIN 51720) in the solid fraction were determined as summarized in Tab. 2. The percentages in Tab. 2 specify the maximum and minimum levels of sum parameters and elements defined by the analyses. The variation depicts the inhomogeneity of the material as well as possible deviation due to the composition of the scrap magnets.

Moisture content and the content of volatile in the solid phase allow estimating the amount of cooling agent remaining in the grinding slurry. As the cooling agent is a mixture of oils, acids and additives the exact amount could not be defined. The composition according to the product specification sheet is shown in Tab. 3.

Table 2. Variation of the composition of grinding slurry after solid-liquid separation.

	Contents
Nd [wt %]	14.8 – 19.0
Fe [wt %]	Balance
B [wt %]	0.9 – 1.2
Dy [wt %]	5.7 – 6.1
Pr [wt %]	0.1 – 4.4
Al [wt %]	0.1 – 0.7
C [wt %]	1.5 – 1.8
O [wt %]	3.0 – 4.1
Sum parameter	
– Moisture content [%]	25.1 – 33.1
– Volatile content in solids [%]	0.7 – 4.1

Table 3. Composition of cooling agent (Mobilcut 230).

Material	Contents [wt %]
Mineral oil	Balance
Boron acid	10 – 20
Ethanol	1 – 5
Monoethanolamine oleate	1 – 5
Rapeseed oil	1 – 5
Others	< 10

3.2 Setup and Trials

3.2.1 Pyrolysis

The pyrolysis experiments for the thermal disintegration of the remaining cooling agent and the destruction of existing agglomerates were carried out in a pilot-plant scale batch pyrolysis reactor. Within framework tests different pyrolysis temperatures as well as different retention times were investigated. The temperatures were varied between 200 °C and 650 °C in iterative steps of 50 °C. The retention time was reduced in iterative steps of 15 min from 60 min down to 15 min.

For each test a batch of 0.5 kg grinding slurries is filled into the batch reactor. Afterwards, the reactor is closed and placed in the pyrolysis furnace. The batch reactor is connected to the condensate tanks via exhaust pipes for the generated pyrolysis gases. Since the pyrolysis takes place under anaerobic conditions, the batch reactor is purged with argon for five minutes before heating and during the process. The reactor pressure is adjusted to a value of about 10 mbar below atmospheric pressure to prevent gas leakage out of the reactor and simultaneously minimize inflow of ambient air. In order to measure the effective temperature in the reactor, it is equipped with three thermocouples, which allow the monitoring of the temperature in the middle of the reactor and on its inner and outer wall (Fig. 6).

The gases generated within the process are passed through the condensate tank where a portion of the gases is condensed due to the water cooling. It remains in the condensate tanks as liquid phase (condensate). The remaining, non-condensable gases pass further to a burner where the gases are burned with a flare before they enter an extractor hood.

After reaching the pyrolysis temperature and holding it for the intended retention time, the furnace is turned off and the solid remainder is completely cooled down. Subsequently, the pyrolyzed material and the resulting condensate can be removed and recorded.

3.2.2 Melting

The melting trials for achieving a phase separation between REE and iron of the pyrolyzed material were carried out in a vacuum induction furnace. A graphite crucible was used to provide carbon in case that the contained carbon is not sufficient to reduce iron oxides and to reduce the melting point of the material by forming cast iron. Besides, by using a graphite crucible, interactions with the formed oxide

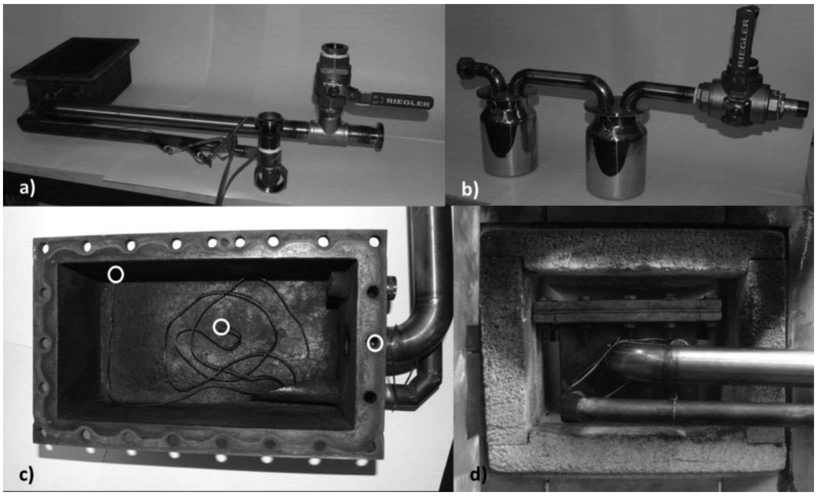

Figure 6. Equipment and setup for pyrolysis experiment: a) locked pyrolysis batch reactor, b) condensate bins, c) open pyrolysis reactor with measuring points for temperature, d) pyrolysis reactor in the furnace.

phase can be avoided mostly. The crucible is placed into a larger graphite crucible serving as susceptor within the inductor. The setup can be seen in Fig. 7.

A thermocouple is set between the crucibles to track the approximate temperature development. Due to the low bulk density of the input material, only a relatively small amount of material (approx. 80 g) can be inserted.

At the beginning of the experiment, the furnace is repeatedly evacuated and then flooded with argon to 750 mbar before starting the heating process. First evaporation phenomena are observed in the low temperature range of 400 – 500 °C. This is believed to be a residue from pyrolysis. However, the smoke generation is comparatively low. The melting point of the grinding slurries cannot be uniquely determined due to the inhomogeneity of the material and, therefore, must be held optically through a sight glass. Melting is preceded by a sintering process, which results in a significant shrinkage of the grinding slurry. After melting a bubbling is observed, which can be explained by escaping oxygen or CO/CO_2. With fading of the bubbling a phase segregation is visible. Here, the slag phase is formed above the molten metal phase and solidifies. The end of the experiment is characterized by a lack of agitation in the melt.

4 Results and Discussion

4.1 Pyrolysis

A retention time of 15 min and a pyrolysis temperature of 600 °C were experimentally identified as optimal process parameters in order to minimize the amount of cooling agent. Microscopical analyses of the pyrolysis input and output prove the disintegration of existing agglomerates, which is shown in Fig. 8.

At a temperature of 600 °C the carbon remaining in the solid remainder of the pyrolysis is transformed into a solidified coke. The comparison of the chemical composure of input and output material shows that a part of the carbon previously found in the liquid cooling agent was transferred into the solid phase (cf. Tab. 4).

In combination with the mass balance of the pyrolysis process (cf. Fig. 9) the composition of the solid pyrolysis output indicates the successful process conduction and the minimization of the remaining cooling agent. The moisture content as well as the volatiles in the solid phase are reduced to an amount below the detection limit. The mass of condensate nearly equals to the moisture amount in the input material, only a minimal amount of solid components ends up in the condensate as fly ashes.

The yield for the recyclable fraction of metallic swarfs exceeds 99 %. For further pyrometallurgical processing, the percentages of carbon and oxygen in the solid remainder are relevant. The evaluation of the experiments leads to the conclusion that storage and the amount of remaining cooling agent or moisture influence the percentages of oxygen and carbon in the solid pyrolysis product which is further treated for recycling.

4.2 Melting

The melting trials clearly indicated that the material properties influence the melting and phase separation since the melting point varies strongly without changing process

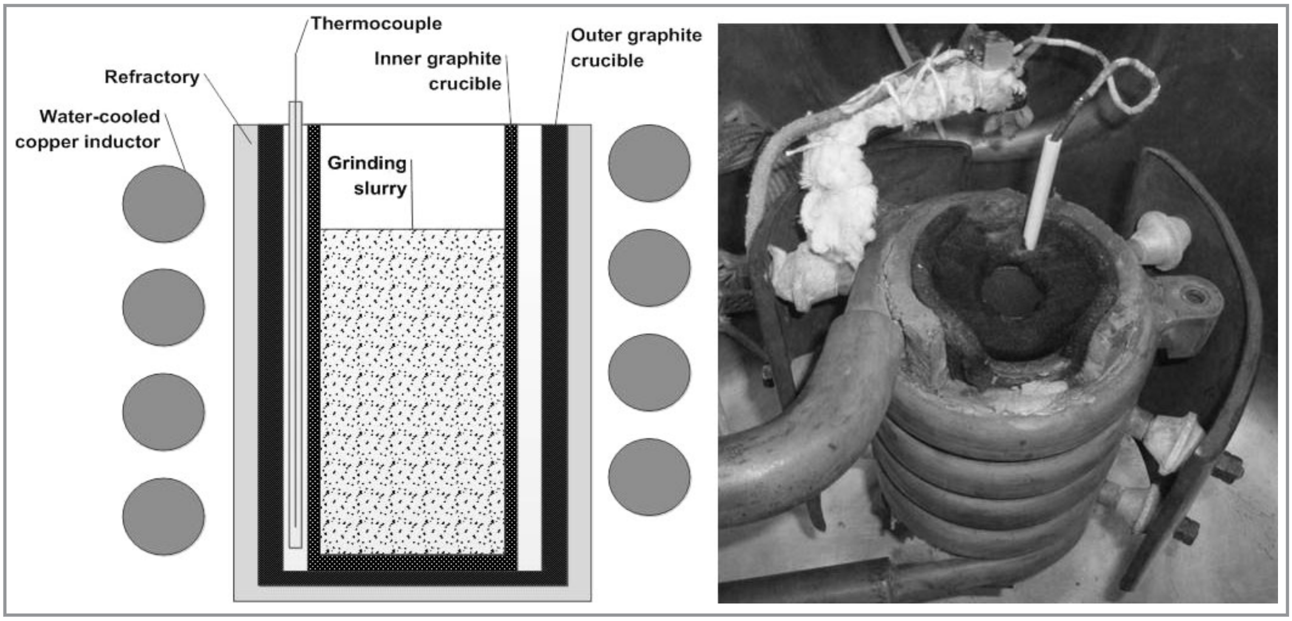

Figure 7. Setup of the melting trials: a) schematic, b) lab-scale testing.

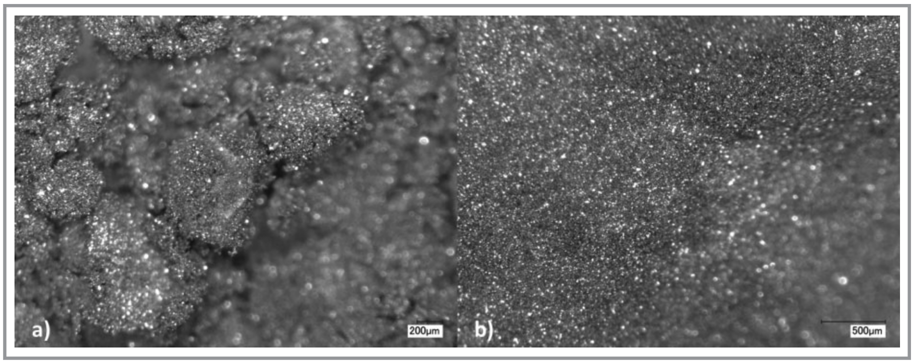

Figure 8. Microscopical analysis of the input (a) and output (b) material of the pyrolysis process.

parameters. Therefore, no general process temperature can be given. Because of the inhomogeneity of the material the influence of the composition cannot be determined exactly, but the analyses show a relation to the amount of oxygen contained in the pyrolysis product. Materials having an oxygen content of less than 7 wt % could hardly be molten down as can be seen in Fig. 10b, while material containing more oxygen gives a clear phase separation (cf. Fig. 10a). The blueish color of the formed slag phase indicates a high amount of RE oxides.

For considering the influence of the oxygen content XRD analyzes of the starting materials and of the pyrolysis products have been carried out. The determined phases and its shares are listed in Tab. 5 as normalized values. In addition to these structures further characteristic radiations are included in the emission spectra, which could not be evaluated. Consequently, subject to the proportions of the structures a certain error occurs. The rare earth elements may also not be distinguished in the emission spectrum from each other since their characteristic radiation is nearly identical. In addition, a determination for amounts < 5 % is inaccurate. Material M1 is a grinding sludge, which is treated immediately after the grinding process. The oxygen content in this case is about 3 wt %. Material M2 on the other hand has been stored after grinding under water so that the oxygen content of about 7 wt % is determined here. The material M1$_{\text{pyrolyzed}}$ cannot be molten under the formation of an SE-rich oxide phase and a metal phase, while the melting and phase separation run smoothly at M2$_{\text{pyrolyzed}}$.

Table 4. Composition of input and output material of the pyrolysis process.

	Sum parameter		Element [wt %]							
	Moisture content [%]	Volatile content in solids [%]	Nd	Fe	B	Dy	Pr	Al	C	O
Input to pyrolysis	25.1 – 33.1	0.7 – 4.1	14.8 – 19.0	Balance	0.9 – 1.2	5.7 – 6.1	0.1 – 4.4	0.2 – 0.7	1.5 – 1.8	3.0 – 4.1
Solid output of pyrolysis	0	< 0,1	14.9 – 18.4	Balance	0.9 – 1.1	5.7 – 9.2	0.1 – 4.7	0.1 – 0.7	0.2 – 2.5	4.3 – 9.4

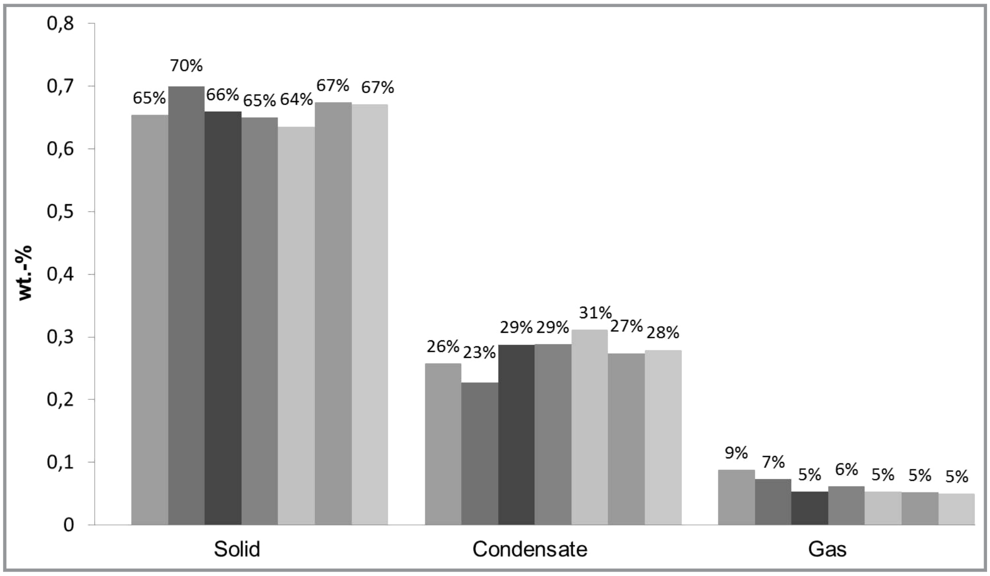

Figure 9. Mass balance of pyrolysis processes of NdFeB grinding slurries.

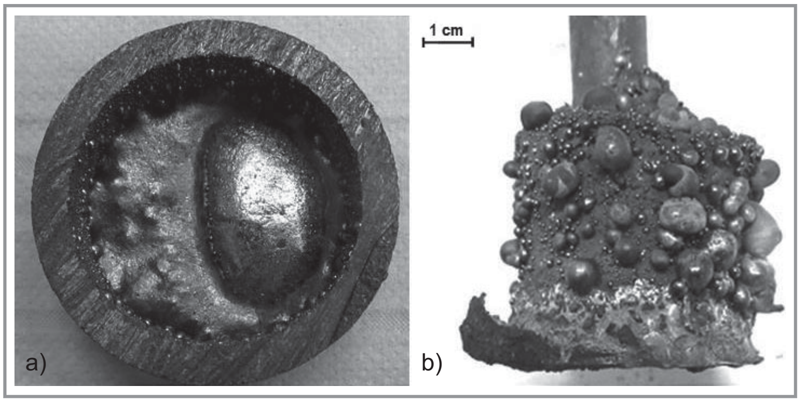

Figure 10. Separated phases after melting (a) and agglomeration with melt droplets (b).

According to XRD analysis the starting material consists entirely of the magnet alloy phase $Nd_2Fe_{14}B$. Other phases cannot be detected in significant amounts. By pyrolysis decomposition of most of $Nd_2Fe_{14}B$ occurs with formation of α-Fe (α-ferrite, α-iron), RE carbides, RE oxides and $NdFeO_3$. In this case, considerable differences in the structural composition of the pyrolysis products are identified. The decomposition of $Nd_2Fe_{14}B$ is less distinctive in M1, so conclusively > 30 wt % of this phase are still detectable. The proportion of produced α-Fe is accordingly low, whereas it accounts for > 80 wt % in $M2_{pyrolyzed}$.

Another conspicuity is the relatively high fraction of $NdFeO_3$ in $M2_{pyrolyzed}$. While the contents of RE oxides are similar, which probably is due to the oxidation of the neodymium-rich phase, at M1 a stronger formation of carbides can be noticed. Thus, the explanation for a successful melting is to be found in the incomplete decomposition of the magnet phase as well as in the formation of RE carbides or a lack of $NdFeO_3$. Due to a lack of thermodynamical data on the magnet alloy and the mixed alloy, modeling of the melting process and the selective oxidation is not possible. Concerning the XRD values a decomposition of $Nd_2Fe_{14}B$ takes place with a simultaneous formation of α-Fe and $NdFeO_3$. Due to a lack of oxygen, the present carbon tends to build carbides with the REE. Because of the small amount of boron in the composition the formed phases cannot be detected or quantified. It is likely that boron can be found in the oxides phase as stated by [11].

The following results of the melting trials only refer to successfully molten material since no analyses could be conducted on the other samples. Considering the mass balance, it appears that upon successful phase separation, the slag phase accounts for about 30 % and the metal phase for 60 – 65 % of the input. There is a difference of 5 – 10 % in form of various losses. This is shown in Fig. 11.

Concerning the yields of elements, Fig. 12 shows the amount of selected elements in the targeted phases based on the content in the pyrolysis product. The oxide phase has been analyzed via XRF while for the metal phase an ICP-OES has been carried out. For REE the relevant elements are Nd, Dy and Pr, which can be found mostly in the oxide phase. Besides, the yield of iron in the metal phase is given in the diagram (cf. Fig. 12).

Even though the samples had different compositions and were treated differently during pyrolysis, the yields after melting all tend to be similar. Regarding iron, 90 % of the amount contained in the educt can be found in the metal phase for all trials except V4. The amount of REE in the oxide phase shows a stronger variation, but still most of the REE are converted to the oxide phase. This is supported by the analysis of the amounts in the opposite phases which is below the detection limit for most of the samples regarding REE and below 1 wt % regarding iron. Losses can be explained by the evident bubbling at the beginning of the melting phase. This leads to splashing and the incomplete recovery of both phases from the crucible, which has a higher relevance due to the small scale of the experiments.

5 Conclusion and Outlook

In summary, it can be said that melting of grinding slurries, which have been pyrolyzed under certain conditions, is suitable to achieve a phase separation between a REE-rich oxide phase and a cast iron. Here, a major reduction of the contents in the opposite phases can be obtained. The proposed processes can be summarized as shown in Fig. 13.

The grinding slurry is subjected to a solid-liquid separation directly after grinding in order to recover the cooling agent. Since this is a state-of-the-art process that is commonly carried out, the process is not part of the investigation but has to be shown for the sake of completeness. The following storage is needed to

Table 5. Textural composition of the grinding slurry with and without preoxidation via water storage (normalized values).

Phases [wt %]	M1 (3 wt % O_2)	$M1_{pyrolyzed}$	M2 (7 wt % O_2)	$M2_{pyrolyzed}$
$Nd_2Fe_{14}B$	100.00	31.36	100.00	0.73
α-Fe	–	57.80	–	82.36
RE carbide	–	5.24	–	1.57
RE oxide	–	2.97	–	2.31
$NdFeO_3$	–	2.61	–	13.02

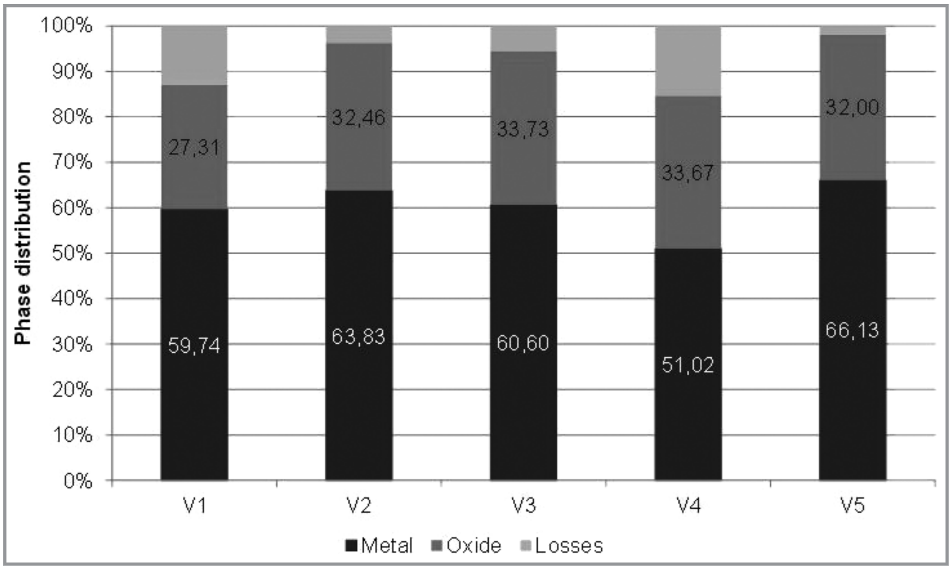

Figure 11. Distribution of produced phases.

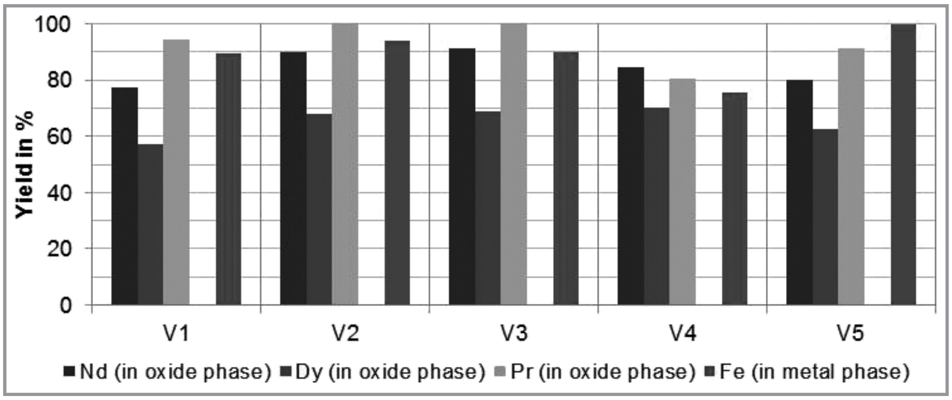

Figure 12. Yields of Nd, Pr, Dy and Fe in the targeted phase regarding to the contents in the input material.

increase the oxygen content. The proposed pyrolysis is carried out at 600 °C with a holding time of 15 min to achieve best results in the subsequent meting process. The pyrolyzed material should have an oxygen content of at least 7 wt % and also has to contain carbon (approx. 2 wt %) although the exact limit value has to be determined. For the melting the use of a carbon crucible is proposed since a lack of carbon in the pyrolysis product can be compensated. The process temperature is not determined exactly because of a variation of the melting point within each trial. The temperature is set to 1350 – 1550 °C with a holding time of 15 min after melting. The phase separation is usually visible.

The RE oxide phase can be further treated within state-of-the-art hydrometallurgical processes such as leaching, precipitation and solvent extraction if needed. This has to be taken into account since high purities are required for RE compounds. The quantity of input material in this case is reduced to 30 % compared to a sole hydrometallurgical treatment. In order to generate RE metals as saleable products an additional reduction process could be the final process step. Both processes are indicated in dotted lines since they are not part of this investigation and have not been conducted up to now. An assessment of the saleability of the concentrates after each processing step is necessary. In order to assess the economic feasibility of the recycling process, the energy efficiency of the proposed processes must be considered. The investigated recycling process must, therefore, be compared to the pure hydrometallurgical treatment (leaching, precipitation, solvent extraction) of grinding slurries. In addition, a subsequent hydrometallurgical treatment must be taken into account as saleable concentrates with high purities are required.

Abbreviations

REE	rare earth elements
ICP-OES	inductively coupled plasma optical emission spectrometry
XRF	X-ray fluorescence
XRD	X-ray diffraction

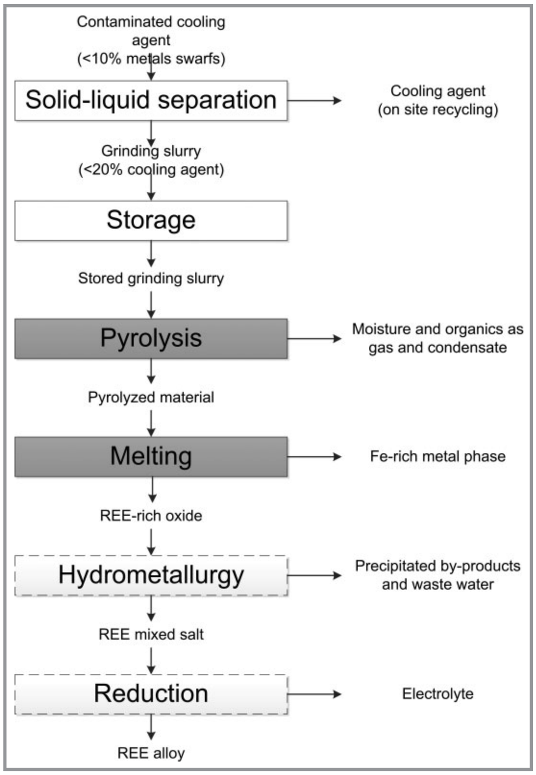

Figure 13. Flow sheet of the recycling concept.

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