In-house recycling of hard zinc and zinc ash by liquid metal centrifugation

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1. Abstract

Today’s share of zinc used for corrosion protection is about 32 %. This main area of application generates a need of 224,000 tons of zinc per year in Germany which is used in the continuous galvanizing and hot dip galvanizing industry. In the process of the latter two zinc containing residues are produced: zinc ash and hard zinc. The amount of produced zinc ash is 12.8 wt.-% and of hard zinc 11.5 wt.-% of the zinc input.

The aim of the presented work is the recovery of the metallic zinc from these two kinds of residues by using the filtering centrifugation technology. In this semi-continuous process solids can be separated from a liquid melt. The centrifuge consists of two rotating, hollow cones, also called rotor, in which the suspension of solid and liquid phases is sucked in. The liquid melt leaves the internal space through a small gap between the cones. The solids are forming a filter cake inside the rotor. Afterwards the cones are removed from the bath and accelerated to reduce the amount of remaining liquid zinc. The dry filter cake is finally removed from the rotor and collected besides the bath.

The melting centrifuge is adapted to the characteristics of the residues hard zinc and zinc ash. By water-modelling and small laboratory scale tests the dependencies of the different parameters of the material characteristics and the melting centrifuge are analysed.

The results of laboratory scale tests are resulting in a zinc recovery of 96 % by separating the intermetallic phases from hard zinc. The remaining liquid fraction in the new intermetallic concentrate won by the centrifuge equals 12 wt.-%. By this a reduction of 9 % of the zinc input at a Hot Dip Galvanising Company seems to be possible, accompanied with the reduction of the produced amount of hard zinc.

Keywords: zinc recycling, hot-dip galvanizing, filtering centrifuge, hard spelter, hard zinc, zinc bottom dross, zinc top dross, zinc ash
2. Background & Motivation

The biggest yearly German demand of the 700,000 tons of zinc is generated by the galvanising industry with a share of 32% or 224,000 tons per year [1]. The hot dip galvanising (HDG) process has a share of 50% in this sector of industry. By this process about 1,600,000 tons of steel are protected against corrosion in Germany [1]. Due to the fact that 1 ton of steel, representing a production energy of 2,500 kWh, can be protected over several decades of years by 30-70 kg of zinc, which is representing 125 - 300 kWh, the galvanising of steel parts becomes an economically very attractive process [2].

In the HDG process two kinds of residues are occurring. The so-called zinc top dross or zinc ash, in industry the latter term prevails, is formed on the surface of the HDG bath. While on the bottom of the galvanising kettle a layer of intermetallic phases is formed which is called hard zinc, zinc bottom dross or hard spelter.

The zinc ash consists mainly of metal-oxides, -sulphides and -chlorides, whereas the sulphides and chlorides are originated reaction-products by remainders of the pre-treatment steps. Due to its difference in density the zinc ash floats on the surface of the galvanising bath as a light brownish powder. From there it has to be removed continuously by the plant’s personnel before the galvanised goods are extracted from the bath. If this is not conducted properly the optical quality of the zinc coated goods is reduced by zinc ash adhering on the surface of the galvanised parts. This kind of HDG residues is containing 70 to 90 wt.-% of metallic zinc, as shown in table 1, due to physically bound zinc droplets. Because of its characteristic as a non-product stream the composition of the material varies which results in an average zinc metal content of 82 wt.-%.

Table 1: Chemical analysis of a sample of “Zinc Ash” from a HDG plant

<table>
<thead>
<tr>
<th>Element</th>
<th>Zn_{total}</th>
<th>Zn_{met.}</th>
<th>Pb</th>
<th>Cl</th>
<th>Fe</th>
<th>Al</th>
<th>Ni</th>
<th>Ca</th>
<th>balance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Share [wt.-%]</td>
<td>87.5</td>
<td>86.4</td>
<td>1.2</td>
<td>6.0</td>
<td>&lt;0.01</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>4.9</td>
</tr>
</tbody>
</table>

By the immersion of a steel part into the zinc bath the major phases, like the gamma-, delta- and zeta-phase, are formed. This refers to the Fe-Zn-phase-diagram shown in figure 1.
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Figure 1: Zinc rich corner of the Fe-Zn binary phase diagram (left) [3], Intermetallic phases, monoclinic $\zeta$–phase and hexagonal $\delta$-phase, and captured Zinc in metallographic section of a hard zinc specimen (right)

From the outermost $\zeta$–layer crystals are washed out; their origins are the intermetallic layers of the steel kettle walls and the steel parts. The formation of these intermetallic phases is supplemented by the reaction of zinc with remainders of iron salts which have not been removed completely in the pre-treatment steps. The iron-zinc-intermetallics, dominated by the $\zeta$–phase as FeZn$_{13}$, are remaining in the bath. There they are settling and finally concentrating on the bottom as the so-called hard zinc. From the bottom they have to be removed manually with a clamshell or a big blade. This process is costly, due to the disruption of the galvanizing process for approximately half a day and the needed support of two to four workers. If the hard zinc is not removed frequently every 5 to 10 production days, depending on the pre-treatment, on the composition of the steel and on the throughput, disturbances on the coated surface are appearing. These so called pits are not reducing the corrosion resistance but the optical and mechanical quality of the zinc coated goods. The typical composition of the removed hard zinc is presented in table 2, whereas the iron content represents 2.2 wt.-%. This concentration under-runs with 4.0 wt.-% the thermochemical zinc concentration of the intermetallic phase FeZn$_{13}$, which is at 6.2 wt.-%. This indicates an amount of 76 wt.-% of physically captured zinc. The optical analysis of the metallographic section of a hard zinc sample, shown in figure 1, underlines this statement due to the big areas of zinc between the needle like intermetallic phases.

Table 2: Chemical analysis of the hard zinc from a HDG plant

<table>
<thead>
<tr>
<th>Element</th>
<th>Zn</th>
<th>Fe</th>
<th>Pb</th>
<th>Sn</th>
<th>Ni</th>
<th>Al</th>
<th>Mg, Cu, Co, Mn, Cr, Si, Cd</th>
</tr>
</thead>
<tbody>
<tr>
<td>Share [wt.-%]</td>
<td>94.25</td>
<td>2.17</td>
<td>2.13</td>
<td>1.16</td>
<td>0.24</td>
<td>&lt; 0.1</td>
<td>$\Sigma &lt; 0.14$</td>
</tr>
</tbody>
</table>

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A typical mass flow for an HDG plant is shown in figure 2. For a throughput of 1,000 kg of steel parts the amount of the hard zinc and zinc ash weights 10.1 and 9.1 kg. The metallic zinc content in these streams weights 8.6 and 9.1 kg respectively. These values are obtained from [4] by using the mass balancing model of Wills [5]. A medium sized HDG company has a throughput of approximately 1,500 tons of steel per month.

Figure 2: Balanced mass flow of zinc in a HDG Process [4,5]

3. The Solid-Liquid Separation Technique

The physical separation of the solid from the liquid phase in a filter is a basic technique. Due to its simplicity it can be an economical attractive technique in the predominantly small to middle sized HDG companies.

3.1 Separation Principle

In the area of filtration processes it can be distinguished between depth filtration and cake filtration. The depth filtration of the melt is selected in many cases in the non-ferrous metallurgy if a high clarification of the melt is needed; for example in aluminium casting facilities. Due to the high solid content of the hard zinc and the zinc ash this technique is not economically attractive. The cake filtration plays an important role if a high throughput with low residual moisture of the cake is desired. The separation efficiency and the throughput of this technique can be increased by an accelerating force. With the help of a filter centrifuge these advantages can be transferred to the semi-continuous processing of the HDG residues.

The pilot scale filter centrifuge used at the IME is presented in figure 3. It consists of an axis which is equipped on the lower end with two hollow cones, also called rotor. On the opposite site an electric drive is installed with which the axis rotates. If the cones are immersed into a liquid metal bath, melt penetrates into the hollow space by the help of collecting holes in the upper or lower cone. Because of the rotation of the cones the melt is accelerated and forced to leave the cones at the gap of the contact area of the cones. If there are solids in the melt, which are heavier than the liquid phase, they are sedimenting immediately radially when entering the cones. Lighter particles are floating inside the cones. Due to the gap size of a few tenth millimetres the solid particles are kept
within the cones and are forming a growing filter cake during the loading time. As the filter cake obtains a maximum thickness the cones are removed from the melt. By accelerating the speed of the axis residual moisture can be removed from the filter cake; it flows back to the metal bath. After this ‘drying’ step, the cones are lifted further. The increasing distance of the cones initiates the automatic removal of the filter cake into a collection bin. With this last step the separation process restarts by the penetration of the melt with the two rotating shells.

Figure 3: The pilot scale filter centrifuge at the IME (left), cones for separation (right)

A typical sequence, lasting between one and five minutes, of these different process steps is shown in figure 4 as function of time and rotating speed. These process steps are shown for different materials in figure 5.

Figure 4: Typical process cycle of a filtering centrifuge
Figure 5: Sketches of the centrifuge process steps for material with a lower (left column), with the same (centre column) and with a higher density (right column) than the melt.
4. Optimisation in the water model

To optimize the separation efficiency of the filter centrifuge for the two kinds of residues, the zinc ash and the hard zinc, series of experiments at the IME are conducted. In the first step a water model has been used, shown in figure 6. The geometrical size of this model is according to the size of the laboratory melt unit. The operation of a water model is economically attractive in comparison to the laboratory scale test or even pilot scale test with a liquid melt. Because of that, large test series have been conducted to evaluate the dependency of the parameters:

- Loading speed (acceleration of impure melt),
- Loading time,
- Cone slope,
- Immersion level of the cones,
- Orientation of the collecting holes,
- Installation and orientation of a partition wall in the cones,
- Loading speed (acceleration of filter cake),
- Loading time,
- Drying time,
- Particle size and distribution.
- Separation of particles from the bath’s surface, from a mixed slurry and a solid bottom layer.

To evaluate the separation efficiency of settling solids, like in the case of hard zinc, spherical silica particles with a density of ~2.6 g/cm³ are used. To simulate the collection of the zinc ash, EPS (expanded polystyrene) particles have been selected with a density of ~0.04 g/cm³.

Figure 6: Sketch and picture of the filter centrifuge water model at the IME
4.1 Modelling of Zinc Ash Separation

4.1.1 Experimental Setup

To model the loading and removal of lighter material than the liquid phase, EPS (expanded polystyrene) particles are used. Due to their very low density of \(\sim 0.04 \text{ g/cm}^3\) an evaluation by weight of the conducted trials is not possible. To face this problem, the received filter cakes have been analysed by their volume in a measuring cylinder after their removal from the centrifuge cones. Table 3 shows the process parameters which are kept constant for all the trials and those varied throughout the experimental series.

Table 3: Selected parameters to simulate the separation of zinc ash by the filtering centrifuge

<table>
<thead>
<tr>
<th>Constant Parameters</th>
<th>Varying Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter of cones 100 mm</td>
<td>Cone slope 30°, 45°</td>
</tr>
<tr>
<td>Shape of collecting holes</td>
<td>Immersion level of cones 0, 10, 20, 40 mm</td>
</tr>
<tr>
<td>Gap between the cones</td>
<td>Acceleration 13, 64, 132, 251 m/s²</td>
</tr>
<tr>
<td>Direction of rotation</td>
<td>Partition walls inside Used or no</td>
</tr>
<tr>
<td>Holes in the upper cone</td>
<td>Partition wall position 45°, 90°, 135° from holes</td>
</tr>
<tr>
<td>Temperature of water 20°C</td>
<td></td>
</tr>
<tr>
<td>Experimental Method</td>
<td></td>
</tr>
</tbody>
</table>

The obtained filter cake volume of one process cycle is selected as the main important value to compare the effects of the varying parameters regarding the collection efficiency. All varying parameters were analysed fully factorial. This means, that all combinations have been analysed. By conducting the trials in a non-organised order, outer effects on the results are minimised. Each trial is conducted three times. The calculated standard deviation of the three results represents the accuracy. If this value exceeds 10 % of the average value, 2 more trials are implemented, to erase the maximum and minimum value of the received five results. By this method, the representativeness of the used values increases strongly.

4.1.2 Experimental Results

In the figures 7 and 8 the average volume of the filter cake is expressed as a function of the immersion level of the centrifuge cones and the loading time.
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Figure 7: Maximum loading with 45° cones and 60 seconds loading time: 6 ml at the surface of the bath at 156 rpm (left) and 44 ml at the surface of the bath at 342 rpm (right)

As the left diagram of figure 7 shows, it is possible to collect the maximum amount of 6 ml floating material after 60 seconds at a rotation speed of 156 rpm, respectively 13.3 m/s². This is possible, if the cones of the filtering centrifuge are adjusted at the surface of the bath. The material which is collected at a lower immersion level is floating in the hollow space at the moment the cones are immersed into the bath. The right diagram of figure 7 is revealing an increase to 44 ml of separated material for a raising rotational speed of 342 rpm, or 64 m/s². It is possible to collect this amount of material at the liquid’s surface. The effect of the further increase of the acceleration is shown in the figure 8.

Figure 8: Maximum loading with 45° cones and 60 seconds loading time: 52 ml 10 mm below the bath surface at 491 rpm (left) and 70 ml 20 mm below the bath surface at 677 rpm (right)

The acceleration of the cones to 491 and to 677 rpm implies the separation of 52 ml and 70 ml. The results are revealing that the highest collection efficiency moves from 0 to 10 and to 20 mm below
the liquid surface with an increasing rotating speed. The results of the 45° cones with the highest loading volume are compared in table 4 with the highest collection efficiency of the 45° cones with partition walls at different orientations and 30° cones. The obtained values show the highest amount of separated material for the cone slope of 45° with 70 ml at the highest speed in comparison to 46 - 55 ml with partition walls. The use of partition walls reduces the collection efficiency. The amount of 31 ml collected by the 30° cones at the optimum conditions shows the effect of the lower volume to the collecting efficiency.

Table 4: Maximum loading of floating material after 60 seconds loading time for the selected parameters

<table>
<thead>
<tr>
<th>Cone Slope, Orientation of Partition Walls</th>
<th>Maximum Material Loading per Cycle, Standard Deviation [ml]</th>
<th>Rotating Speed [rpm]</th>
<th>Acceleration [m/s²]</th>
<th>Immersion level of the Cones [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>45° none</td>
<td>6</td>
<td>156</td>
<td>13.3</td>
<td>0</td>
</tr>
<tr>
<td>45° none</td>
<td>44</td>
<td>342</td>
<td>64.0</td>
<td>0</td>
</tr>
<tr>
<td>45° none</td>
<td>52</td>
<td>491</td>
<td>132</td>
<td>10</td>
</tr>
<tr>
<td>45° none</td>
<td>70</td>
<td>677</td>
<td>251</td>
<td>20</td>
</tr>
<tr>
<td>45° 45°</td>
<td>55</td>
<td>491</td>
<td>132</td>
<td>10</td>
</tr>
<tr>
<td>45° 90°</td>
<td>54</td>
<td>491</td>
<td>132</td>
<td>10</td>
</tr>
<tr>
<td>45° 135°</td>
<td>46</td>
<td>491</td>
<td>132</td>
<td>10</td>
</tr>
<tr>
<td>30° none</td>
<td>31</td>
<td>677</td>
<td>251</td>
<td>20</td>
</tr>
</tbody>
</table>

4.1.3 Discussion

The described results are showing a movement of the process window with the highest collecting efficiency by increasing the acceleration. At higher rotating speeds the loading zone moves below the surface of the bath. This phenomenon is related to the formation of a vortex between the surface of the bath and the immersed cones at speeds exceeding 342 rpm. This vortex, on which surface floating particles are transported to the collecting holes, causes two effects:

- At low immersion levels the vortex has a higher diameter as the distance of the collecting holes and any material is flooding into the cones,
- At the correct immersion level the vortex diameter is coinciding with the distance of the collecting holes of the upper centrifuge cone and a high amount of material is collected.
4.2 Modelling of Hard Zinc Separation

To simulate the separation of particles heavier than the liquid phase, spherical silica particles have been added into the water basin, shown in figure 6. In earlier experiments cones with the holes in the lower side have been selected to analyse the loading phenomena [6]. As the rotation of the cones starts a vortex develops underneath the cones and sucks particles from the bottom layer of the bath into the cones. The results won by this setup are showing

- a high collection efficiency for a particle size exceeding 50 µm,
- a strong increase of the collection efficiency with an increasing particle size,
- the reduction of moisture to < 5 wt.-% is obtained by the highest acceleration of the filter cake at 350 g or 2,500 rpm,
- the influence of the cone design to residual moisture is small and only a tendency for a smaller slope with a reduction of the liquid fraction can be determined,
- a reduction of the loading time by using partition walls in an orientation of 90° to the collecting holes [6].

4.2.1 Experimental Setup

To evaluate the effect of a fully mixed suspension of the solid-liquid-phases a new setup has been selected. By the use of a stirrer the difference of the mixed phases and a bottom layer is analysed. For these experiments the parameters are chosen as presented in table 5.

Table 5: Selected parameters to simulate the separation of hard zinc by the filtering centrifuge

<table>
<thead>
<tr>
<th>Constant Parameters</th>
<th>Varying Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slope of cones 45°</td>
<td>Bottom Layer Distance</td>
</tr>
<tr>
<td>Particles collected &lt; 50 µm</td>
<td>Acceleration 64, 132, 251 m/s²</td>
</tr>
<tr>
<td>Collecting Time 100 seconds</td>
<td>Collecting Holes Top or Bottom Cone</td>
</tr>
<tr>
<td>Gap between the cones</td>
<td>Stirred Bath Yes or no</td>
</tr>
<tr>
<td>Direction of rotation</td>
<td></td>
</tr>
<tr>
<td>Shape of collecting cones</td>
<td></td>
</tr>
<tr>
<td>Temperature of water 20°C</td>
<td></td>
</tr>
<tr>
<td>Experimental Method</td>
<td></td>
</tr>
</tbody>
</table>

Extended loading times are needed for the separation of solids with a particle size < 50 µm [6]. Due to that fact the particle size < 50µm is selected for the experiments. By this experimental setup the differences in collection efficiency are most strikingly.
The particles separated by the centrifuge for each process cycle are dried and weighted. The weight of the dry filter cake is chosen as the main determining value to compare the collection efficiency of the varying parameters. The statistical procedure to analyse the won data is according to the procedure chosen for the simulation of zinc ash separation.

4.2.2 Experimental Results

The development of the collection efficiency is expressed in figure 9 as a function of the rotational speed. The low amount of collected material from a bottom layer is expressed by the two bottom lines in the figure. Only a small amount of material can be collected at all centrifugal speeds. If the centrifuge collects the material from a stirred slurry at turbulent conditions, the amount of collected material raises at the lowest speed from zero grams to 2.0 respectively 2.4. At the highest speed of 677 rpm more than 7 grams of material can be separated at the same loading time. The amount of collected material is higher, if the cones are immersed further into the bath. This is shown by comparing the amount of collected particles at 491 rpm, which leads to 2.8 and 3.9 grams of filter cake, and at 677 rpm, which leads to 7.2 and 7.6 grams.

![Figure 9: Increased separation of material affected by a stirred slurry compared with a bottom layer of solids](image-url)
4.2.3 Discussion

The modelling of the centrifugal separation shows the dependency of the collection efficiency on the grain size of separated particles, on the loading speed and the immersion level of the cones. As the particle size exceeds 50 µm the loading time can be reduced rapidly. A shorter loading time can be reached as well at turbulent bath conditions and at higher loading speeds.

4.3 Summary of Water Model Experiments

By assessing the experimental results of the water model the following statements can be formulated:

- Separation of lighter and heavier solid particles should be possible by the filtering centrifuge of the IME,
- Floating material needs to be collected with collecting holes in the upper cone
- Speed and immersion level of the cones is influencing strongly the collection efficiency of floating particles which implies an exact control of the parameters if a transfer to a liquid melt is executed,
- Heavier material from a solid bottom layer has to be collected with collecting holes in the lower cone,
- Particles exceeding a diameter of 50 µm are separated in an acceptable loading time,
- The loading time to separate heavier solids than the liquid can be reduced significantly in a stirred bath or by an increased rotational speed,
- The highest removal of liquid from the filter cake is obtained by the highest acceleration

5. Evaluation in Laboratory Scale

The results received from the water model experiments are validated by trials in a laboratory centrifuge for hot melts, the setup of the equipment is shown in figure 10.
The centrifuge consists of an installation with a pneumatic drive to immerse the cones installed on an axis into the liquid melt. The melt is heated by a resistance furnace adjusted by an Eurotherm 2704 controller using two thermocouples, one in the melt, one at the heating elements. With an optimised control algorithm for the processing of zinc a temperature accuracy of 0.5 K is obtained. For the experiments an amount of ~10 kg zinc with ~10 kg of hard zinc is heated up to 450°C. By this setup the concentration of intermetallic phases in the melt amounts ~12 wt.-% due to the fact that the concentration in the hard zinc equals 24.5 wt.-%. The axis with the two cones is accelerated by an electric drive, which is capable to adjust the rotational speed. To ensure a constant concentration of intermetallics the removed filter cake from each trial is added back to the bath after weighing. As the chemical composition or a cross section of the filter cake is analysed a profile of 3 mm thickness is won. By accelerating the filter cake after the loading time to a maximum of ~2,000 rpm for 30 seconds residual liquid zinc is removed. In all the trials a non-organised order of changing the parameters is selected to minimise the influence of effects from the outer experimental frame, for example personal influences. In the hot trials the melt is not stirred, like in the water model, to evaluate the mixing effect of the centrifuge rotor itself.

5.1 Separation of Intermetallic Phases from Hard zinc

In the first series the effect of the cone distance to the bottom of the crucible is evaluated. For this trial 30° cones are selected with the collecting holes in the bottom cone. The loading time varies between 30 and 90 seconds, the distance of the cones to the bottom of the crucible varies from 50 to 100 mm. In figure 11, the weight of the filter cake is expressed as a function of the loading time and the depth of the immersion.
The results correspond to the statements of the water model; at a loading time of 90 seconds the filter cake weight increases from 113 to 188 grams by immersing the cones 50 mm further to the bottom.

By the same experimental setup the influence of the rotating speed and the collecting holes is analysed at a constant loading time of 30 seconds. As figure 12 shows, a rising acceleration is leading to a higher amount of separated solids. The 10 grams filter cake separated at 150 rpm is increasing to 190 grams at 450 rpm. The turbulent conditions produced by immersion of the cones to the bottom of the crucible results in an similar course of the different setup of the collecting holes in the upper, lower or in both cones. As an example the rotating speed at ~ 290 rpm is selected, where 74 grams (upper cone, 290 rpm), 72 grams (lower cone, 280 rpm) or 87 grams (both, 284 rpm) are separated at the different cone-setsups. The repeatability of the trials is expressed in figure 13 as the width of the obtained filter cake weights for three trials.
Figure 12: Dependency of the filter cake amount to the rotating speed and the orientation of the collecting holes at a constant loading time of 30 seconds

Figure 13: Repeatability of the results presented by the width of the filter cake weight
In order to evaluate the capability of the filter centrifuge to remove smaller concentrations of intermetallic phases continuously from a zinc bath the filter cakes are not fed back after the process cycle to the bath as in the trials before. Starting from a 20 kg zinc melt with 2.8 kg of hard zinc, representing 3 wt.-% of solid phases in the melt, the stepwise removed filter cakes are weighed. Then samples are taken and analysed by ICP and by a light-optical microscope. The results are shown in figure 14. The weight of the separated filter cake drops within the first three trials from 310, to 197 and to 96 grams. The iron content of these cakes ranges between 5.5 and 5.2 wt.-%, which corresponds to a residual liquid content of 12 to 16 wt.-%. By continuing the separation process the amount of intermetallic phases in the bath is reduced which results in lower weights of the filter cakes and a diminishing recovery. An asymptotical gradient for the weights of the filter cake is reached with 18.2 grams at the 6th process cycle. After the 10th process cycle the iron concentration drops to 3.7 wt.-%. Nevertheless the centrifuge is capable to remove crystals even at very low concentrations.

Figure 14: Continuous removal of Fe-Zn intermetallic phases from a zinc melt

An example of the cross section of a filter cake is shown in figure 15. The left picture shows the inner side - towards the rotor axis - of the filter cake. The intermetallic FeZn13-Phases are represented by the white, longish crystals. Remaining zinc captured between the smallest intermetallic phases can be identified by the grey colour. The right picture represents the middle area of the filter cake with big pores of air as black spaces. The air has replaced the residual liquid in the drying step. Zinc is captured between the fine intermetallic phases due to the phenomena of pendular saturation or saturation by capillary rise. The pendular saturation is built at particle-particle contact points and...
is determined by capillary forces or the surface tension. If the centrifugal forces are higher than these effects, remaining liquid can be removed. The capillary rise is determined by the balance of the liquid’s weight in the liquid column under the centrifugal force and the force of the air-liquid-solid interface [7].

Figure 15: Cross Section of the recovered filter cake from hard zinc, left picture: inner side, right picture: middle of filter cake

5.2 Discussion of the Experiments in Laboratory Scale

To separate the intermetallic $\zeta$- and $\delta$-phase from hard zinc the filter centrifuge of the IME shows the capability to produce a filter cake with an iron concentration of 5.5 wt.-%. Remaining zinc between small crystals results in an amount of 12 wt.-% liquid phase as the maximum iron concentration is 6.2 wt.-% of the prevailing FeZn$_{13}$-phase.

6. General Assessment and Conclusions

The presented results of the water model are showing the capability to separate heavier and lighter solid particles with the filter centrifuge from liquid media. The chosen materials are representing the heavier intermetallic phases (settling effect) or the lighter zinc ash (floating effect). The efficiency to collect floating material is very sensitive to the rotating speed and the immersion depth of the rotor into the bath. The increased internal volume of the 45° cones results in a higher amount of separated material, 70 ml, in comparison to the 30° cones with 31 ml. The use of partition walls inside the cones does not influence the collecting efficiency. The applicability of the centrifuge for the treatment of zinc ash in a ‘hot’ experiment has to be proven in the near future.

The water model filter cake showed a residual moisture of < 5 wt.-%. The filter cake of intermetallic phases separated from a liquid zinc bath contains 12 wt.-% of residual liquid and 88 wt.-% of the prevailing $\zeta$-phase. The higher amount of the residual liquid phase in the melt experiments compared with the water model is related to capillary residual zinc between intermetallic phases < 50 µm. The amount of this residual moisture is affected by the surface tension, capillary, interfacial
and centrifugal forces. It can be removed at very high accelerating forces, but not at the chosen acceleration of ~ 2,000 rpm.

The water model shows that intermetallic separation from hard zinc is possible if the particle diameter exceeds 50 µm. The metallographic analysis of the separated filter cakes received from laboratory experiments reveals also the separation of intermetallic phases of less than 50 µm. This is due to the well known phenomenon of the cake filtration, which allows the separation of a finer material in the cake than on the surface of the filter media itself.

A clear dependency of the loading time from the immersion depth and the rotating speed is detected by the water model, verified by the results of processing the liquid melt. At a constant loading time of 90 seconds the weight of the filter cake rises by 166 % from 113 to 188 grams if the cones are immersed 50 mm closer towards the bottom. The increase of the speed from 150 rpm to 450 rpm results in an increase of the filter cake weight from 10 to 190 g. The water model shows further a better separation of solids from a bottom layer, if the collecting holes are in the lower cone. The results gained by the experiments of the hot melt are showing similar results if cones are in the upper, lower or in both shells with 74 grams (290 rpm), 72 grams (280 rpm) and 87 (284 rpm). This difference to the water model results can be due to the turbulent conditions in the liquid bath.

If an industrial scale centrifuge is implemented at a HDG company to treat the hard zinc residues the amount of zinc input can be reduced by 9%, as shown in figure 16. This reduction can be reached if the separation efficiency corresponds to the obtained results in laboratory scale. The determination of the input reduction due to the recycling of zinc ash, the upscaling of the process for ash and hard zinc as well as the economical analysis of the implementation will be the future tasks of the presented project.

Figure 16: Reduced zinc input by the implementation of the centrifugal technique for hard zinc processing in a hot dip galvanising company
7. Acknowledgements

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8. References