Development of High-performance Nd-Fe-B Magnetic-Filter for Separation of Metallic Impurities

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Abstract

Most mechanical processes such as grinding and milling accompany unexceptionally contamination impurities which have critical influences for follow-processes and product-properties. Especially metal impurities in ceramics are fatal. Therefore, many efforts are applied to reduce contamination by choosing ceramic milling-tools and - using magnetic-filters.

High-strength rare-earth magnets (Sm-Co, Nd-Fe-B) are used for magnetic-metal impurity-separation. This contamination is mostly Fe, Ni, or Co caused also from moving parts of milling facility and transfer systems, drives etc. A permanent magnet-based filter system is favoured because of easy adaptation, handling and maintenance.

In this study a new magnetic-filter was developed by advanced magnet-flux design. A multi-component magnetic core is built by several magnet-rings that initiate magnetic fields with high magnetic flow-densities in the intermediate Fe-rings. Most important is the performing and concentrating areas with highest field gradient inside 2 ring-gaps where the product passes without barrier at high throughput.

For testing, several products were contaminated with predetermined Fe-powder particles, filtered and investigated by chemical analysis, laser diffraction, scanning electron and optical microscopy.

The system has been rapidly introduced at ceramic industry in Europe. Applications and improvements at Ivoclar Vivadent AG, one of the world-wide largest dental-ceramic producers are reported.

1. Introduction, motivation

The present paper describes the design, testing and commercial application of a new permanent magnetic filter system. The inventor Zoz GmbH is originally a producer of conventional grinding equipment for crushing / particle size reduction of solid materials (ballmills) [1]. Parallel a specialization on High Kinetic Processing (MA, HEM, RM) [2] has been pursued where this at the same time describes the platform of the today's almost 50% share of the R&D-volume in the field of materials-design and development in the company. Since 1998, powders are produced by the above techniques of HKP-routes [2,3] and since early 2000 also PM-parts [4].

In general, we never designed or produced any kind of magnetic filter system before and this has certainly been of advantage, which will be understood later.

What we have been doing for decades, we used magnetic filters and adapted them into conventional sieving and mixing/milling-operations mostly supplied in ceramic industry [5].

Fig. 1: new magnetic filter system MF-DN100x110, complete and single components

However, we have been not very much satisfied with the properties of these units. Either it was pretty hard to clean them, the adaptation difficult, the throughput was to small, the magnetic-field-gradient was in particular due to a non magnetic-like design to low, the achieved magnetic-field-strength due to the at least today antiquated magnetic-materials was low, too - or all together. Next to this these products were not cheap at all.

Since today our main business-field is Powder-Metallurgy res. related to PM, the decision came more late than early. We decided to design a new magnetic filter (Fig. 1) with the aim to improve all the above.

Since the first two prototypes came up the system has been highly respected in corresponding industry in Europe.
One of the applicators, Ivoclar Vivadent AG in Schaan, Liechtenstein, who currently use a number of 12 of the new systems and is one of the world-wide largest dental-ceramic producers agreed to help us in further development and support this work significantly.
What is remarkable is the fact, that the time from idea to development and the first two prototypes was not longer than 6 months. Since that the new system is supplied all over Europe and this paper will explain why.

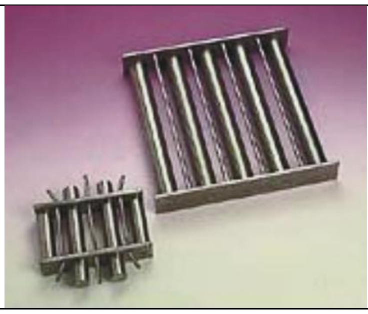
2. Common application of permanent magnetic filter systems

Most of technical commercial products depend on components where at least some of the materials are fabricated by mechanical processing techniques. Here the control of impurities/contamination is always important since mechanical processing without impurities is generally impossible. There is only variation in quality and quantity of the contamination which means that here the suitable tools for the control must be found.
E.g. in case of ceramic materials, it is very important to limit contamination of metals to an acceptable limit. Often it is not possible, to acceptably control contamination just by choosing all product touching parts from suitable ceramic material since the process is either to complicated or the application of ceramics is to expensive e.g. for transfer systems or not available res. both, e.g. drives etc.
Since Fe-alloys as the most common metal belongs to the ferromagnetic materials (Fe, Co, Ni and Lanthanides like Gd, Er, Dy..), a good chance to control and clean ceramic materials are magnetic filters to separate corresponding impurity fractions. Here various kinds of systems are applied where in general a compromise between maximum acceptable contamination ratio and needed throughput (and cost of course) must be accepted res. found.
Since adaptation, handling and maintenance/cleaning play an important role, filter systems based on permanent magnets are frequently used as they are easy to handle at low cost and small unit size.

3. State of the art of permanent magnetic filter systems

The state of the art of permanent magnetic filter systems is more or less described by placing a magnet into a mass-flow and trying to use modern magnetic materials. The principle is probably 100 years old [6] and has not been changed in recent decades. In industry, 2 main application routes can be noticed:

The first is placing a strong magnet into the same container where some suspension or slurry is currently stored with the hope that magnetizable particles will be attracted and pulled to the surface of this kind of magnetic trap and remain there until a cleaning cycle of the same (Fig. 2).

		
Fig. 2: magnetic trap used in former times at a major boiler-producer	Fig. 3: typical magnet-grating built up by several magnet-bars	Fig. 4: magnet-bars in block-like arrangement

A somewhat more sophisticated technically route is to build up a barrier for a volume flow of product in order to force it passing close to the magnet surface. However, the main problem is defined by the word *barrier* already. In order to have an acceptable barrier, its design leads usually to a non-barrier.

Figure 3 shows a typical magnet-grating that is built up by several magnet-bars. The gaps between the bars are relatively large in order that the product can pass suitably. Only the wanted effect is not reached since this arrangement does not guarantee that the major fraction of the product passes extremely close to the magnet surface which does not lead to a significant separation of magnetizable particles and will be shown later (application at Gewürzmühle Nesse).

Figure 4 shows a device with a block-like arrangement of magnet bars where the distance between them is much shorter and can therefore only be used for liquid products with low viscosity. The separation-effect versus throughput is regarded to be higher.

The device shown in Figure 5 is designed very similar, where the bar-block is assembled in a kind of pump-casing. Surprisingly, the magnet-bars are fixed inside a complicated non-magnetic metal-chamber obviously for a better option to clean the unit.

Fig. 5: magnet-bar in block-like arrangement in a kind of pump-casing

Fig. 6: magnet-bar in block-like arrangement with corrugated iron skin

Fig. 7: arrangement of several horseshoe magnets on the outside circle of a flat cylinder

Surprisingly because in this design the magnets have additionally a free play of several millimetres to the chamber where this (at air-induction) and for the thickness of the chamber wall leads to a tremendous loss of the magnetic flux density available on the surface of the chamber compared to the surface of the magnet bars. Comparisons to these kind of solutions will be given by the application of a major bath tub producer later in this paper.

Figure 6 shows again a block-like design of magnet bars but this time with a kind of curious skin of corrugated iron obviously in order to enlarge the surface where a magnetic field is induced, which is surprising again since the magnetic core has in average a number of millimetres free play to the curved metal skin. Next to this it seems that this device must be very hard to clean.

Finally figure 7 shows a simple arrangement of several horseshoe magnets on the outside circle of a flat cylinder. The particular shown unit is intended to be fitted on top of a funnel and is expected to fulfil more less an alibi-function for the user.

Thus it looks like that the devices mentioned above have not seen any significant improvements in recent time, except using more modern magnetic materials. We believe that this is due because magnetism is a rather difficult subject and therefore we give a brief survey of related correlations which is content of the next chapter.

4. Magnetic materials in brief

Generally we decide between diamagnetic, para- and ferromagnetic materials [7-11]. The name ferro- refers to iron and relates to history since the first magnetic materials have been noticed (magnetite) res. identified by its attraction to other naturally occurring iron based materials, probably at first by Greeks and Chinese thousand years ago [12].

Every known material in nature shows diamagnetic behaviour which is caused in atomic diamagnetism and is not related to a permanent magnetic moment. In the atomic model of Bohr, negative loaded electrons orbit around the positive nucleons in the center where the electrons additionally rotate around the own axis, the spin. Any orbital moved electrical load describes an orbital current and is known to cause a magnetic dipole.

However, in any atom, the orbit magnetic dipoles from orbit and spin are opposite directed in twos which means in case of saturated electron-shells, the magnetic moments compensate each other. To explain the reason, why these materials interact with an external magnetic field, the quantum theory is needed [13-16]. Figure 8 gives a scheme of the electro-movement and the resulting magnetic moments.

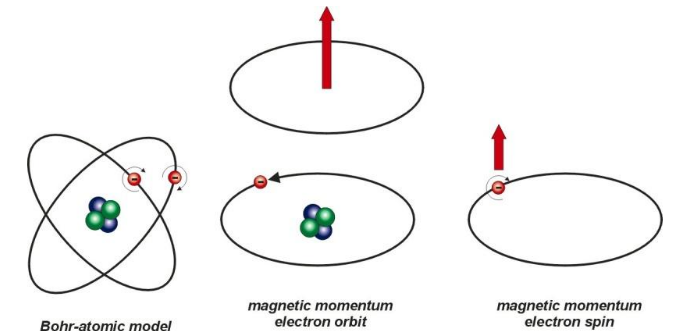


Fig. 8: schematic of electron orbit and spin with magnetic moment

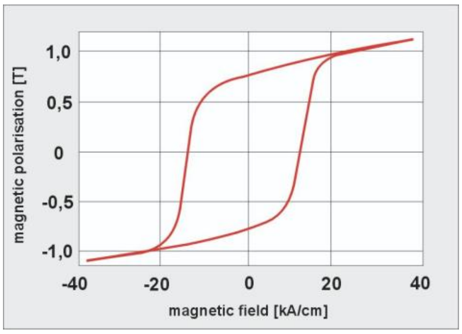


Fig. 9: a typical hysteresis of ferromagnetic material (produced by Simoloyer CM01)

In case of non-saturated electron-shells, the left and right turn oriented currents as well as negative and positive spins do not fully compensate each other which means the individual atoms behave like magnetic dipoles. This effect is very soft and in strong interaction with the thermodynamic. The higher the temperature, the lower the paramagnetic behaviour. A full alignment of all magnetic moments is achieved at no temperature [7-11].

This does happen in case of ferromagnetic materials [7-11, 17]. Here the interaction of neighboured atoms leads to an alignment of spins on neighboured lattice places even without the presence of an external magnetic field. This occurs only in crystalline structure and leads to regions of numerous but each aligned atomic dipoles. These domains are not aligned among each other but will do so under the presence of an external magnetic field. If this magnetic field is strong enough, all domains can be aligned and the material is magnetized at saturation. After removing the external field, the domains will dominantly remain aligned resulting in a permanent magnet. This relation between initial magnetization and remaining (remanent) magnetization at varying field strength describes the typical hysteresis for ferroelectrica [18-21]. Figure 9 shows a typical hysteresis of Nd-Fe-B magnetic material produced by HKP with the Simoloyer [22].

A characteristic value is, the magnetic field (strength) (H) is important and is expressed in the unit of Oersted:

magnetic field (strength) $$\mathrm{H} = \left[\frac{A}{m}\right] \qquad 1\,\mathrm{Oe} = \frac{1000}{4\pi} \cdot \frac{A}{m} \tag{1}$$

The magnetic flux density or magnetic induction (B) is expressed in units of Gauss (G) and Tesla (T):

magnetic flux density (magn. induction) $$\mathrm{B} = \left[\frac{Vs}{m^2}\right] = \mathrm{Tesla}\ [\mathrm{T}] \qquad 1\,\mathrm{Tesla} = 10000\,\mathrm{Gauss}\,[\mathrm{G}] \tag{2}$$

The magnetic flux density of the earth on the surface refers to about 1×10^{-4} T which refers to 1 G.

In case of a magnetic filter application, where clearly because of the needed throughput not all material can be forced to directly touch the magnet-surface, it is of major importance, to accelerate particles passing close to the magnet surface to the same. For the force F on a charged particle q moving with velocity v, the vector equation (3) is valid:

$$\mathrm{F} = qv \cdot B \tag{3}$$

Commonly used to determine the quality of magnetic materials is:

$BH_{maximum}$ energy product $$\mathrm{BH}_{\max} = H \cdot B\ [\mathrm{MGOe}] \tag{4}$$

Since in case of a magnetic filter, the volume-specification over the distance from the magnetic surface to the to be separated particle is unknown res. consists of variable product in variable concentration in air or suspension the permeability, which describes the inversed resistance of a medium against the magnetic flux lines is important too.

absolute permeability: $$\mathrm{B} = \mu_0 \cdot H \qquad \mu_0 = 4\pi \cdot 10^{-7} \left[\frac{Vs}{Am}\right] \tag{5}$$

examples: vacuum $$1\frac{Vs}{Am} \tag{6}$$

steel, approx. $$10\frac{Vs}{Am} \tag{7}$$

The permeability at air-induction μ_{Luft} which must be taken into account for magnetic filter operation of dry powders can be determined to be close to 1 (vacuum).

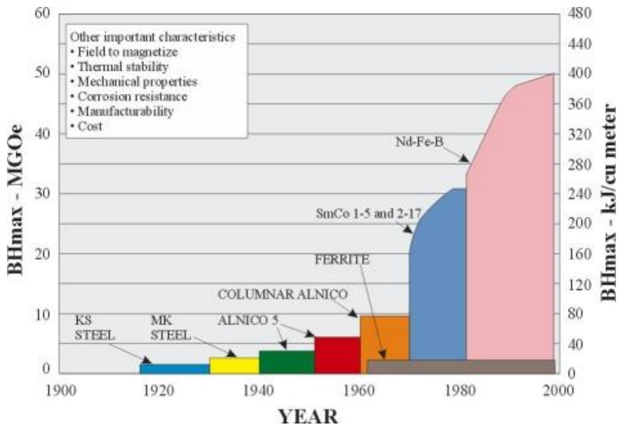


Fig. 10: Development of Permanent Magnets in the 1900's

Figure 10 shows a survey of commercially used permanent magnetic materials in historical chronology. The summary here should start with the alnico magnets (Al-Ni-Co) having been developed around the 1940’s and are still in frequent use today [23]. In the 1960’s, hard ferrite or ceramic magnets were developed. These magnets are made of strontium hexaferrite where the base material is strontium carbonate and iron oxide which is both, very well available at relatively very low cost. These materials represent probably about ¾ of the world magnet consumption and are used e.g. in motors, speakers, etc.[24]. Since around 1970, sintered samarium cobalt magnets are available and do supply very high magnetic properties at excellent corrosion resistance and thermal stability. The listed Sm2Co17 composite e.g supplies a maximum energy product of over 30 MGOe and can be used up to 350°C (Curie temperature 700-800°C).

The permanent magnet material with the highest available maximum energy product are the Nd-Fe-B magnets (usually Nd2Fe14B) which are available since around mid 1980’s where today, refined materials can lead almost up to 50 MGOe. A disadvantage is the relatively lower Curie temperature at 310°C that can be shifted by adding e.g. 5 % Co to about 370°C which refers to a working temperature of 150-200°C in maximum. Also the corrosion resistance is low, so these magnets usually must be protected by coatings or caging [23].

5. Advanced magnetic like design

In case of a magnetic filter application, high magnetic properties of the material are of major importance. Taking into account, that the insofar best commercially available materials (Nd-Fe-B) are limited to a maximum energy product of about 50 MGOe where the theoretical maximum value is at about 64 MGOe [23], a promising improvement of magnetic filter systems must use these best materials but improve the application which can here mean the design of the magnetic field and the design of the technical / mechanical unit where the product is send through.

One area where significant improvements can be expected, is the design of the gaps where the product passes since here the magnetic field is loosing almost all of its performance due to the low permeability of air res. of the non-magnetizable base-product. This particular relation between distance and magnetic property is obviously not at all obeyed in case of most of the before described conventional systems, even the effect can easily be imagined if one try to take a piece of steel, bring it close to a permanent magnet and see what happens very close to it and see what happens just a few millimeters away from the magnet surface [25]. However, this approach is limited res. in direct interaction with the available throughput of any kind of gap or distance since a filter that would block the mass flow of product would not be suitable. Also and this simply under economically view, it is not imaginable that all (solid) particles of the product can directly touch the surface of the magnet which might be described as a flat container or pipeline lined with a huge number of magnets. A device like this would be extremely huge on the one hand and even more expensive on the other hand.

If this is accepted, the main target must be to accelerate the magnetizable particles towards the magnet surface and collect res. fix them thereon [26, 27].

If it is further taken into account, that for the acceleration of a magnetizable particle in the magnetic field, the magnetic field gradient, which is described by the variation of the magnetic flux density over the distance to the magnetic source is a determining factor, then the product of field gradient and flux density must be of major importance for a magnetic filter system [7].

Since the magnetic flux density can be imagined like the density of magnetic flux lines, it was the main idea, to increase the density of these flux lines by arranging thin anti-polar ring-magnets around iron cores each and press the single rings together and by this condense the magnetic field in the same way. Since this would happen right at the surface of magnets and ring-cores initially, a significant increase of the field gradient could also be expected.

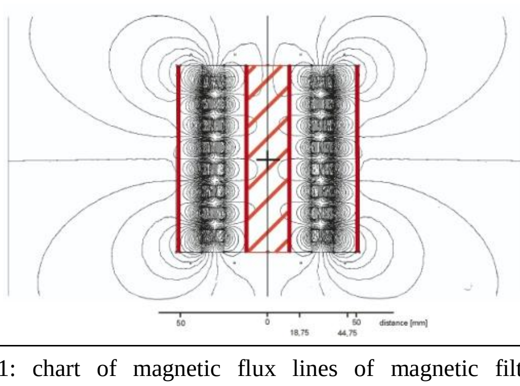


Fig. 11: chart of magnetic flux lines of magnetic filter MF-DN100x110

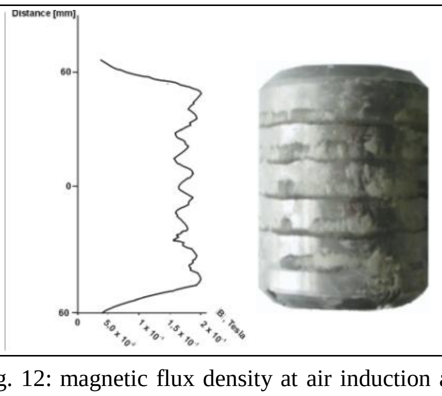


Fig. 12: magnetic flux density at air induction at res. of magnetic unit MK-078105

Figure 11 shows the chart of magnetic flux lines of the developed magnetic unit MK-078105 with a number of 10 anti-polar arranged ring-pairs that initiate in the intermediate Fe-rings magnetic fields with high magnetic flow-densities (*figure 12, left*) where the barriers inside and outside represent the inner and the outer circle limit built up by an inner tube called center unit as well as by the outer tube defined as standard pipe DN100 (see figure 1).
The chart in figure 12 exhibits the magnetic flux density determined on the gap-middle-axis of each circle-gap where here it can be seen what was meant with the statement of extremely reduced magnetic properties at air induction just some millimeters (here 4.25 mm) away from the magnet surface where it should be considered, that the here introduced design is strongly expected to achieve a significantly higher level than any other design.
Additionally, by this technical solution, concentrating areas with highest field gradient inside two determined ring-gaps where the product can pass without any barrier at high throughput are performed. Since the entire magnetic filter system contains only a low number (3) of single units, only assembled by one single screw, it can easily be disassembled, cleaned and adapted to pipe-lines carrying multi-phase flows and slurries.
For a better removal of separated particles from the magnetic unit, the peaks of the same are non-magnetic which means the waste can be pushed up- or downwards to these peaks and easier removed from there.
The main dimensions of the unit are given in table 1:

Inner diameter of the outer tube	99 mm
Outer diameter of the magnetic unit	78 mm
Inner diameter of the magnetic unit	48 mm
Outer diameter of the center unit	27 mm
Free gateway of the outer circle-gap	2897 mm²
Free gateway of the inner circle-gap	1215 mm²
Nominal gateway	NW 71.6 mm dia

Table 1, main dimensions of magnetic filter MF-DN100x110

6. Simple in-house testing

The far easiest and most suitable way of testing a magnetic filter system is a comparatively quantitative test in real condition which means the new filter system should be applied for products/materials where common systems had already been applied.
With the two prototypes we produced, we did follow exactly this procedure and send these units to some huge industrial applicators. A quantitative statement is however not possible since we have never seen the prototypes again, they were bought away on the other day. Since this is a non defined but very satisfying result, we produced another 10, sent them out and the same thing happened. Then production had been increase to 25 and currently the production batch is 50.
In between we thought that we need to do some testing at least in-house where it has really not been easy to reserve at least one of the systems for our own purpose.
We decided for some very simple testing, where we did choose 3 different type of media (water, sand and enamel-slurry), loaded each with a determined number of Fe-powder, partly mixed it and then separated again by means of the new filter system.

Fig. 13: experimental set up for Fe-powder and water, simulation of full water flow, water drained

Figure 13 shows the experimental set up for Fe-powder in water. Since this composition can be hardly mixed, we placed 2g Fe-powder on the inner border of the funnel in a standard Zoz-sieve SW50 where the magnetic filter was adapted to the end of the funnel. In order to simulate a full water flow, we used a large plastic container to guide as well as another one to supply more water that can pass by gravity at once. The view on the right shows at the same time the lower end of the funnel where the magnetic filter is assembled.

For the composition Fe-powder in sand, we followed the same procedure, just that we mixed sand and Fe-powder in a ratio of 1250:1 by weight% in a Zoz-Rollermill RM1 which referred again to 2g of Fe-powder. The procedure is shown in figures 14.

Fig. 14: mixing of Fe-powder in sand in a Zoz-Rollermill RM1, simulation of full powder flow, sand drained

For the composition Fe-powder in enamel-slurry, we followed the same procedure as in case of Fe-powder and water and contaminated the slurry with 2 g of Fe-powder by placing on the inner border of the funnel. The procedure is shown in figures 15.

Fig. 15: experimental set up for Fe-powder and enamel slurry, simulation of full slurry flow, enamel slurry drained

In case of the water flow, where we calculated and measured flow parameters as 18l/s and 4.4m/s we separated 1.5g, in case of the sand flow 0.83l/s and 0.2m/s at separated 1.5g and in case of the enamel-slurry flow 0.9l/s and 0.2m/s, at approximately separated 2g.
As expected these simple results show that the quantitative separation depends strongly on the flow-velocity which is at fixed magnet unit lengths a number for the remaining time of the multiphase flow in the magnetic field and of the viscosity of the same which refers again to the flow velocity.
The remaining time can be influenced by extending the length of the magnetic unit which has in fact been simulated in case of the water flow by adapting not one, but two magnetic filter units. Figure 16 shows these two magnetic units.

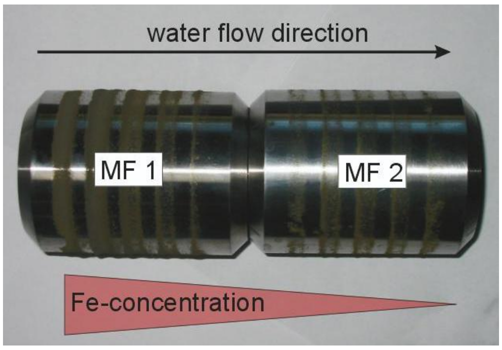


Fig. 16: the 2 magnetic units as dismantled after the test of Fe-powder and water

7 Applications

7.1 Application report and testing at Ivoclar Vivadent AG

As stated before, the far easiest and most suitable way of testing a magnetic filter system is a comparatively quantitative test in real condition which is reported here.
Ivoclar Vivadent as one of the major dental ceramic producers in the world must and did take care for clean and in particular Fe-free dental ceramics. Insofar this is a perfect condition for a *crash-test* for the new developed system.

Figure 17 shows 3 from 6 sieving-operations for ceramic base and ready products where each 2 of the magnetic filters MF-DN100x110 are used.

Fig. 17: 3 / 6 sieving operations at Ivoclar Vivadent with ea. 2 adapted magnetic filters MF-DN100x110

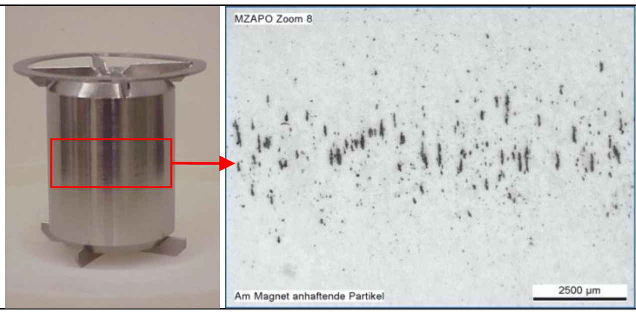


Fig. 18: separated waste on the outer surface of the magnetic unit and enlarged

Figure 18 shows an example of separated waste on the outer surface of the disassembled magnetic unit (left) and enlarged by optical microscopy after removal on a bond-strip.
Figure 19 shows a consolidated ceramic sheet under optical microscopy in conventional processing route (left) and including additional application of the new magnetic filter system (right). On the contrary to the left sample, the one on the right hand side does not exhibit any inclusions res. black oxide spots, which is the best expectable result for the new device.
In order to try to determine the source of the separated magnetizable particles, we collected 1.3g of waste after sieving 400kg of powder with an apparent density of 1.2kg/dm³ at a sieving rate of 50kg/h, applied optical, scanning electron microscopy (SEM) and X-ray diffraction (EDX).
Figure 19 shows an optical microscopy, an SEM-micrograph as well as the EDX-analysis referring to the marked spot on the SEM-image, identifying extremely high Fe-content of 91 at% which allows the conclusion that an Fe-particle has been separated, preserving the product.

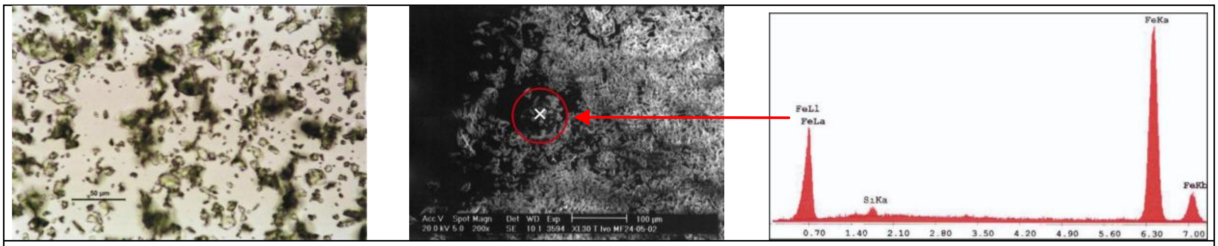


Fig. 19: optical microscopy, SEM and EDX Analysis of separated particles, very high Fe-content

7.2 Application report and testing at major enamel applicators

In case of the following reports of two major enamel applicators, the magnetic filter MF-DN100x110 has each been assembled in a pressure capsule D-MF1011* as seen in Fig. 20. The product is then pumped through the filter. In case of the first example, about 7 tons of enamel slurry with a density of 1.7-1.75kg/dm³ have been pumped through a pipeline system within 1h where the new magnetic filter is adapted. 69.7g of waste have been separated and partly characterized.

Fig. 20: pressure capsule D-MF1011*with assembled magnetic filter in a pump-line at a Zoz-screen-cart

Fig. 21 shows an optical microscopy, an SEM-micrograph as well as the EDX-analysis referring to the marked spot on the SEM-image, identifying high Fe-content of 41 at% which may allow the conclusion that an Fe-particle has been separated.

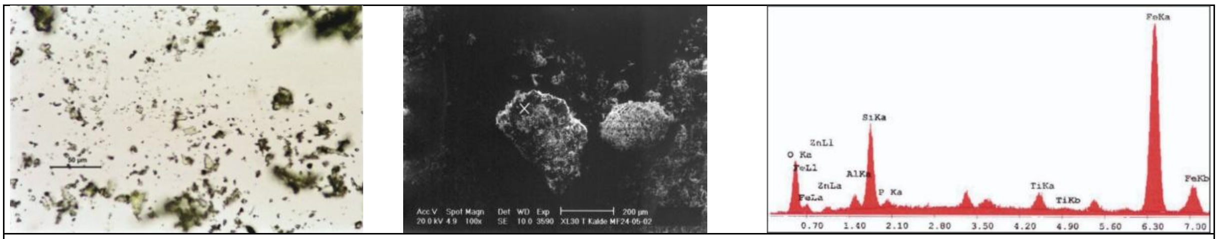


Fig. 21: optical microscopy, SEM and EDX Analysis of separated particles, high Fe-content

In case of the second example, 1.5 tons of enamel slurry with a density of 1.4-1.75kg/dm³ have been pumped through a pipeline system where the new magnetic filter is adapted after a sieving rate of the Zoz-screen-cart of 1200kg/h. The separated waste has been cleaned where a number of 7.85g remained.

Figure 22 shows an optical microscopy, a SEM-micrograph as well as the EDX-analysis referring to the 2 marked spots x_4 and x_5 on the SEM-image, identifying high Fe-content of 74 at% res. high Ni-content of 80 % where the fist can be expected to be a contaminating Fe-particle and the second caused by some remain in the tested base-slurry that contents Co- and Ni-rich Oxides for bonding of the slurry.

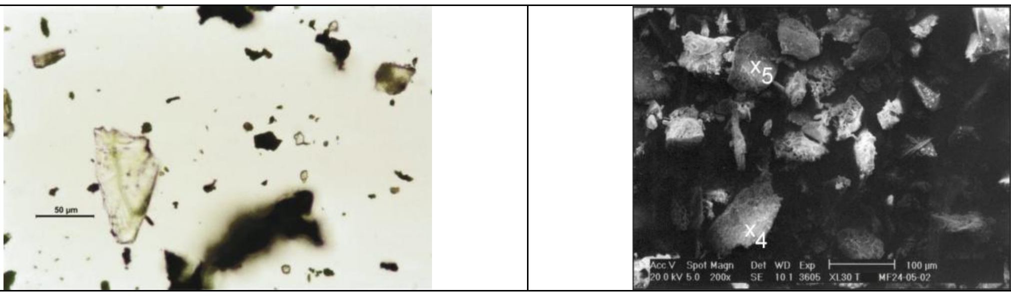


Fig. 22: optical microscopy, SEM and EDX Analysis of separated particles, high Fe-res. Ni-content

7.3 Application at Gewürzmühle Nesse GmbH

The application report concludes with an example for the application of the same magnetic unit MK-078105 (2 each) but assembled in a tube-capsule RF-DN140x250 at Gewürzmühle Nesse GmbH, a major spices, herbs and condiments producer in Germany.
Fig. 23 exhibits the assembly of the unit in the plant (left) as well as separated waste on the surface of the magnetic units (right). Here 100 kg of pepper have been filtered in 15 min where 3 g of waste have been separated.

Fig. 23: tube capsule RF-DN140x250 at Nesse and disassembled magnetic units

8. Conclusions

A new magnetic-filter has been development by advanced magnetic-flux design using modern permanent magnetic material.
A multi-component magnetic core is built up by several magnet-rings that initiate in the intermediate Fe-rings magnetic fields with high magnetic flow-densities. Most important is a technical solution of performing and concentrating areas with highest field gradient inside 2 ring-gaps where the product can pass without any barrier at high throughput.
Due to it's far superiority compared to conventional systems and due to the possibility of quick and easy comparatively quantitative testing has been rapidly introduced in industry.

Commercial application examples at major producers in dental ceramic, enamel and spices have been given.
The immediate and significant success here is certainly caused by deficiencies elsewhere, in particular in applying magnetic like design.

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