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Performance Improvements and Development Trends of the Industrially Sintered NdFeB Permanent Magnets

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Abstract – **The magnetic properties and the corrosion behaviour of the sintered NdFeB magnets are strongly influenced by the composition and the manufacturing processes. This paper reviews the industrial progress covering improvements in the properties of sintered NdFeB magnets, namely in the magnetic energy density $(B \cdot H)_{max}$, in the temperature stability and in the corrosion resistance, giving examples of magnet grades TERRAMAG®1) available in mass production**.

Keywords – HRE reduced NdFeB, TERRAMAG®*)

I. INTRODUCTION

Permanent magnets of NdFeB type have become indispensable electrotechnical components for our high-tech technologies. The sintered NdFeB permanent magnets achieve today the highest remanence and respectively the highest energy densities. Their main benefits, besides an improved magnetic performance, are the reduction of volumes and weights compared to other permanent magnet materials. The importance of sintered NdFeB permanent magnets in modern technologies can be deduced from the total world permanent magnet market, valued at about 9 billion US$ in 2015 [1].

The remarkable hard magnetic properties of the $Nd_2Fe_{14}B$ compound have been discovered in 1983 quite simultaneously by Sumitomo Special Metals (SSM), Japan and by General Motors (GM), USA, in a period of increased demand for strong and small permanent magnets. SSM patented and successfully started the magnet manufacturing using the powder metallurgical route, like for the production of SmCopermanent magnets, while GM patented and used the so-called melt-spinning process to produce "amorphous", isotropic NdFeB alloy ribbons and to work them in isotropic plastic bonded magnets. Later, Hitachi Metals Limited, emerged from SSMC more than 600 patents on sintered rare earth elements. Todaytheir portfolio includes different patents on the efficient, secure and commercial production of sintered rare earths permanent magnets.

The commercial mass production of NdFeB magnets started 1984 using the powder metallurgical route. It became the most common and most important technique used to manufacture anisotropic NdFeB magnets in big quantities and with a large variety of shapes. The abundant Chinese natural reserves of rare earths, the cheap labour costs and the powder metallurgical route, attracted several hundreds of Chinese investors, which started mass productions of sintered NdFeB magnets without legal licencebefore 1990. The results were and are an extremely hard Chinese-Chinese competition leading to price deteriorations, which in the most cases is "compensated" by cost savings in quality controls and by reduced R&D activities. The world market of NdFeB magnets is dominated today by the Chinese NdFeB manufacturers [2]. The Western users are on the one hand attracted by very advantageous Chinese price offers, on the other hand they risk getting poor magnetic properties and poor stability against corrosion, because despite the good price/performance ratio, the NdFeB magnetsalso have some drawbacks. One of them is their low Curie temperature of about 310°C, which results in higher negative temperature coefficients of the magnetic flux and of the coercive field strength [3]. Furthermore, a higher sensitivity to corrosion requires adjustments of the microstructure and special surface protections.

Based on the outstanding magnetic properties of NdFeB magnets, successful efforts have been made since 1997 by "BEC Gesellschaft fürProduktmanagementmbH" (BEC) and its Chinese partner in industrial R&D to overcome the instability handicaps and to improve the magnetic properties at room temperature as well as the stability with respect to opposite external fields and temperatures [3]. The industrial R&D activities of BEC on NdFeB magnets are focused on several main directions:

- Improvement of the corrosion and thermal stability, by replacing the Nd-rich phase by chemical stable intergranular intermetallic phases, using a new

[1]) TERRAMAG®is the trade name of corrosion and stable magnets developed and distributedexclusively by „BEC Gesellschaft fürProduktmanagementmbH", Germany and produced under licence of Hitachi Metals, Japan.

manufacturing method. Since 2002 the corrosion stable TERRAMAG® magnets of S series are commercially available in mass production [3]

- The development of corrosion stable NdFeB magnets with defined maximal operating temperatures without or with reduced content of Heavy Rare Earths (HRE) Dysprosium (Dy) and Terbium (Tb)

In this paper, the industrial progress covering the improvements in the corrosion and in the temperature stability of the NdFeB permanent magnets, new advanced technologies and new trends will be reviewed. Several examples of commercially available grades of NdFeB magnets developed by BEC are given to highlight successful R&D activities, such as the development of permanent magnets for high temperature applications without and with reduced content of HRE, the so called TERRAMAG® Z, TERRAMAG® R and TERRAMAG® L series, by using adapted processing routes. It updates the review published by BEC in 2005 [3].

II. CORROSION OF NDFEB MAGNETS

A. *Corrosion of "common" NdFeB magnets*

The sintered NdFeB permanent magnets have a high sensitivity to corrosion. The low chemical stability relative to corrosion is based on the magnet microstructure consisting of the ferromagnetic matrix phase $RE_2Fe_{14}B$, called also Φ – phase, RE is for Rare Earths, with a very high saturation for RE = Nd and Pr and high magnetic anisotropy for RE = Dy and Tb [3-6]., The matrix phase is embedded in a ductile, intragranular distributed RE – rich phase, providing on one hand the magnetic decoupling of the Φ – grains, and, on the other hand, is responsible for high mechanical properties. In the microstructure of the "common" NdFeB magnets the RE – rich phase is containing up to 95 wt. % Nd and the rest in Fe. Its low melting point is important for the sintering process of the powder compacts, [3-6]. Further phases of the microstructure are the $Nd_{1.1}Fe_4B_4$ boride (the η-phase), which is not magnetic at room temperature, and impurities, for instance oxides, carbides and nitrides [3,6].

In dry atmosphere, the NdFeB magnets are showing higher corrosion stability than the $SmCo_5$ magnets [6]. But under conditions of higher temperature and the presence of humidity this Nd-rich phase is reacting as a rare earth metal and forms hydroxides, hydrides and hydrogen, leading to its decomposition [7]. In the microstructure of the "common" NdFeB magnets about 2 – 5 wt. % Nd-rich phase is "necessary" for sintering and mechanical resistance: at sintering temperature, the Nd-rich phase becomes "liquid", contributing so to achieve high material densities. This is reason, that very often NdFeB magnets produced in Fareast are containing a much higher fraction of Nd-rich phase, in single cases up to 10 wt. %, leading to increased corrosion behavior.

B. *Corrosion testing methods*

The users of permanent magnets are interested to estimate the magnet "lifetime" under the normal environmental operating conditions. Especially quality control engineers are using the so called "accelerated tests" to qualitatively estimate the corrosion behavior of the produced batches. Pioneers of corrosion tests simply used common kitchen pressure cookers for experiments. In the common language of the magnet manufacturers this test is also named **P**ressure **C**ooker **T**est or PCT. Under the very hard test conditions of 121°C at 100% relative humidity (RH), the corrosion is acceleratedenormously. The required equipment for PCT is cheap, it is a common autoclave, and the test is very easy to perform: the weight losses per surface unit due to corrosion of bar magnets stored for several days, usually for seven days, are determined [8,9]. Fig. 1 shows as example the measured specific weight losses after 7 days PCT for different commercially available magnets produced in Europe and in China. Depending on the composition and microstructure, i.e. on the magnet manufacturer, the determined specific weight losses vary between less than 2 mg/cm^2 for the TERAMAG® S grades and for other stable European grades, and 850 mg/cm^2 for any magnets produced by any Chinese competitors.

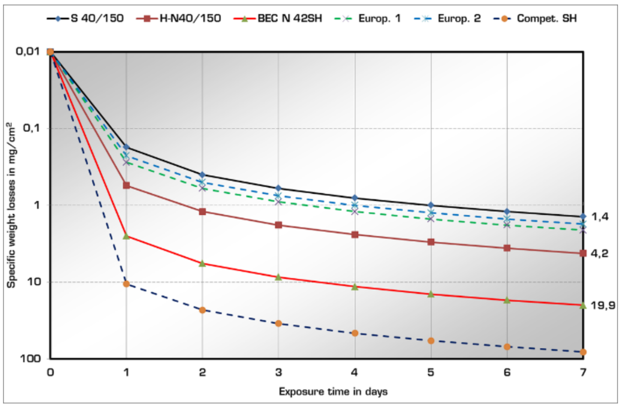

Fig. 1: Examples of measured specific weight losses under PCT conditions (121°C, 100% RH) measured on different commercial available permanent magnets. After seven days PCT the measured specific weight losses of the TERRAMAG® S magnet amount to less than 2 mg/cm^2, compared with those of "common" SH magnet produced by the competition. The red line shows an example of an extreme instable Chinese magnet produced in 2016: after 7 days PCT the magnet is practically dissolved

On request of the automotive industry, the **H**ighly **A**ccelerated **S**tress **T**est (HAST) has been introduced for several years. The particularity of this test is the controlled relative humidity. The typical parameters of this test are 130 – 134°C at 80 – 95% relative humidity. According to any opinions these conditions are more reflecting the natural use conditions of the permanent magnets [10]. On the other hand, the equipment costs are very high and during the exposure time of at least 10 days, it is not recommended to stop the test for sample analysis.

Generally, the samples characterized by specific weight losses lower than about 2 - 5 mg/cm^2 after seven days PCT are declared “corrosion stable” [11]. This very rough characterization is leading very often to confusions and to exaggerations and the Chinese manufacturers are indicating arbitrary data related to the specific weight losses, which do not represent any qualitative specification of the permanent magnet materials.

C. *PCT test and equivalency model of BEC*

The PCT parameters for NdFeB magnets have been not officially approved by an international standard, yet. Depending on the used equipment the pressure, the temperature and the relative humidity can vary in ranges of 2-3 bar, creating 120 – 130 °C and 80 - 100% humidity, respectively [11]. For examples, the test parameters used in BEC Laboratory are 2.5 bar pressure resulting in 121°C and 100% relative humidity, the test period is 7 days. To correlate the effects of a PCT exposure time τ_{PCT} with an “equivalent real time” τ_R in which the magnet will suffer similar corrosion effects under its real operating conditions of temperature T and relative humidity RH, we introduced an “acceleration factor AF [8]

$$AF = \frac{\tau_R}{\tau_{HAST}} \qquad (1)$$

A quantitative estimation of the factor AF is possible starting with the Arrhenius equation.The relationship between the rate a reaction proceeds and its temperature, and by experimental measurements of the activation energies of heat and humidity [8]. Using AF BEC was in the position to evaluate the effect equivalency between the specific weight losses under PCT and under real operating conditions.

Table 1 shows examples of the equivalent magnet” lifetimes” correlated to an exposure of 1 day under PCT conditions used by BEC.The specific weight losses measured after 7 days PCT in Fig. 1 are roughly varying between 1 and 1000 mg/cm^2. Based on the typical density of NdFeB magnets of 7.6 g/cm^3, a specific weigh loss of 1 mg/cm^2 will cause a calculated surface corrosion of 1.3 µm thickness. According to the BEC equivalency model of Table 1 the same corroded surface thickness is expected after 12.5 years under operating conditions 60°C and 80% RH.

Table 2 shows as example the estimated corroded volume fraction per year of a magnet block with the dimensions 40 x 20 x 3mm depending on the specific weight losses of the used material measured under and on the operating climatic conditions.

Table 1: Estimated magnet “lifetimes” according to [9] under different real application conditions of temperature and humidity, related to the PCT conditions used by BEC Laboratory

T [°C]	**RH [%]**	**1 day PCT is equivalent to**
50	80	4,16 years
60	80	1,8 years
75	80	191 days
85	85	50 days

Table 2: Estimated corroded volume fraction according to [9] of a magnet 40 x 20 x 3mm in dependence of the typical material PCT specific weight losses and on the operating climatic conditions

Specific material weight losses [mg/cm^2]	**Volume in % corroded per year under the following climatic operation conditions**			
	50°C / 80% r.h.	**60°C / 80% r.h.**	**75°C / 60% r.h.**	**85°C / 85% r.h.**
2	0,01	0,02	0,06	0,22
5	0,02	0,04	0,15	0,55
20	0,07	0,17	0,59	2,21
80	0,29	0,68	2,39	0,37
150	0,55	1,27	4,55	18,86

D. *NdFeB magnets with improved corrosion stability*

The corrosion behaviors as well as the magnetic properties of the sintered NdFeB permanent magnets are strongly influenced by the composition of the intragranular phase. Due to its role during sintering and due its binder role between the grains of the Φ-phase theNd-riche phase cannot be eliminated, but wecan replace it [3,6,7,11] by other Nd-rich intermetallic, low melting stable phases, such as Nd_3Co, NdCu, $Nd_5(Ga,Cu)_3$ or $Nd_6Fe_{13}M$, with M = Ga, Cu or Al [6-8]. Investigations demonstrated that such magnets are improving the corrosion stability in aggressive media [9].

BEC focused its R&D activities on the use of additives containing RE, Co, Cu, Al and Ga in adequate stoichiometric ratios, leading to intermetallic phases with low melting points [3]. These activities were resulting in the development of TERRAMAG® magnets of the S- and H-N-series, classes of modern NdFeB magnets with improved corrosion stability, for applications up to 230°C [14]. The typical specific weight losses after 7 days PCT are lower than 2 mg/cm^2 for the TERRAMAG®magnets of the S-series and lower than 5 mg/cm^2 for the TERRAMAG®magnets of the H-N-series [14], s Fig. 1.

The magnets of TERRAMAG® S-series were the first corrosion stable NdFeB magnets produced in China and they are still belonging to the NdFeB magnets with the highest corrosion stability existing on the world market.

Significant progress has been made to reduce the

HRE Dy and Tb contents. New technologies such as alloy preparation by strip casting, power milling by hydrogen decrepitation and using new target mills, automatic compacting presses, multi chamber continuous sintering furnaces and other more allowed to increase the coercive force [s. the next chapter of this paper] by other ways than the increase of the magnetic anisotropy by Dy and Tb.

BEC successfully enlarged the corrosion and temperature stable TERRAMAG® family by developing Dy and Tb free or with reduced Dy and / or Tb contents magnets. Their improved corrosion stability - their typical specific weight losses after 7 days PCT are lower than 5 mg/cm^2- has been achieved similar as by the TERRAMAG® of S-series, by adjusting the microstructure.

Despite important progress with respect to the chemical stability, the NdFeB magnets are sensitive to corrosion: in contact with humidity they begin to rust like iron parts. Therefore, for applications where humidity is present coating is recommended. Different metallicand organic coatings have been developed to protect the magnet surface in aggressive atmospheres [14]. For transport and storage temporary surface passivation methods are recommended. Passivated NdFeB magnets are usually stable at temperatures lower than about 30°C and relative humidity up to 60%.

III. TEMPERATURE STABILITY OF NDFEB MAGNETS

A. *Temperature stability of the "common" NdFeB magnets*

According to the ISO / IEC recommendations a magnet grade is said "thermal stable" at anoperating temperature, if the irreversible losses for a working point $B/\mu_0H \sim -1$ are lower than 3 %. Physically this condition is generally achieved if the coercive force H_{cJ}> 420 kA/m at operating temperature, Fig. 2. Ternary NdFeB permanent magnets achieve coercive forces of about 800 kA/mat room temperature (RT), which allow applications only up to maximal 60°C. For applications at higher temperature, sufficient "reserves" of coercive force H_{cJ} at room temperature are necessary. The coercive force can be improved by different ways by adjusting the stoichiometry and so the microstructure. During about 35 years of existence in thousands of investigations, different elements have been added to $Nd_2Fe_{14}B$- alloys to improve the permanent magnetic properties. The added elements can be classified in three types [15]. In the past, the most common way was the replacement of Nd by the HRE Dy or Tb, both are increasing the anisotropy field strength and hence so the intrinsic coercive force H_{cJ} at room temperature [16]. In addition, a partial substitution of Fe by Co increases the Curie temperature and thus improves the temperature dependence of the remanent polarization, but too high Co-concentrations are deleterious to the coercive force [13]. The second type consisted in elements like Al, Ga, Cu, Sn, Bi which leaded to replacing the intragranular Nd-rich phase by other low melting intragranular phases [17- 20]. The third type were refractory metals like Nb, V, Mo, Ti, which form precipitate in the matrix phase and cause domain-wall pinning. [21,22]

Today the magnet manufacturers are offering a plethora of nuances, called also grades of NdFeB permanent magnet materials with adjusted composition able to assure the coercive force necessary for operating temperatures up to 230°C. As indicator for the maximal operating temperature, the Chinese Norm became most accepted. According to it, the maximal operating temperature of a NdFeB material is rather "estimated" by the value of the coercive force H_{cJ}at RT. The characters N, M, H etc. are indicating the maximal operating temperature of cylinders having the ratio height / diameter = 0.7. For example, according to this norm the N-materials, characterized by H_{cJ}> 955 kA/m at RT can be used up to 80°C, the H-materials with H_{cJ}> 1353 kA/m at RT, up to 120°C and the EH-Materials, H_{cJ}> 2387 kA/m at RT, up to 200°C. The working line of the reference cylinder is $B/\mu_0H \sim -1.47$ and the Chinese norm allows irreversible losses up to 5% at the maximal specified operating temperature [23]. In order to fulfil the stability condition a minimal coercive force of only H_{cJ}> 340 kA/m is required.

But a minimal coercive force at the maximal operating temperature is specified nowhere [23]. The thermal stability of an application is synonymous with avoiding irreversible losses at the operating temperature. For this purpose, the magnetic properties and their temperature dependence must be considered. According to BEC "philosophy" the minimal coercive force H_{cJ} at the maximal operating temperature is of major importance. Therefore, BEC is specifying the minimal coercive force H_{cJ} at the maximal operating temperature for all TERRAMAG® permanent materials. Fig. 2 is showing schematically the minimal H_{cJ} for the TERRAMAG® of S-series (the black curve - according to the IEC recommendations), of H-N series (the blue curve – the "physical" H_{cJ}limit to avoid irreversible losses) and according to the requirement of the Chinese norm (the red curve).

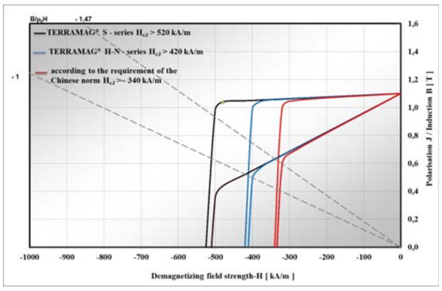

Fig. 2: Required minimal H_{cJ} at the maximal operation

temperature of the TERRAMAG® S- and H-N series, compared with the requirement of the Chinese norm

Fig. 3 is showing the minimal magnetic properties remanence and coercive force at 150° of TERRAMAG® grades S- and H-N and comparatively of the "common" N grades according to the Chinese Norm.

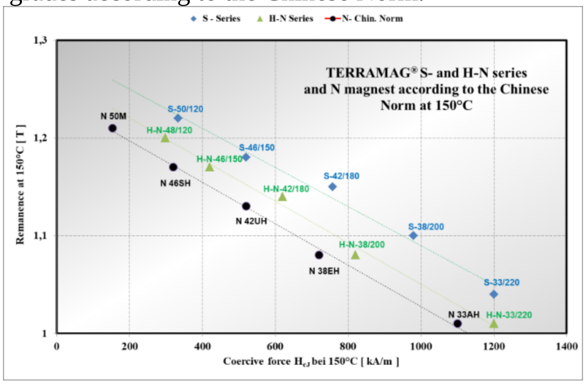

Fig. 3: Minimal magnetic properties remanence Br and coercive force H_{cJ}at 150°C of TERRAMAG® grades of S- and H-N series compared with the minimal properties of the "common" grades specified by the Chinese Norm

B. *Activities to reduce the HRE content in sintered NdFeB magnets*

To reduce the cost created by the very expensive HRE Dy and Tb and to support the growth tendency of the NdFeB magnet market, many producers started activities to develop NdFeB magnets HRE – free or with HRE – reduced contents. BEC and its Chinese partner have made significant progress in increasing the coercive force by other way than using the usual contents of Dy and / or Tb and developed the TERRAMAG® of Z-series, Z like ZERO Dy and Tb, and the of the R-series, R like REDUCED Dy and Tb contents. The development has been successful due to new manufacturing processes [24] and by adjusting the microstructure, for example by reducing the impurity levels, especially that of oxygen (low oxygen technology), by improving the fine powder properties, namely the size uniformity and the particle size distribution, by improving the magnetic alignment during powder compaction, by optimizing the sintering process, which resulted in the reduction and in a better uniformity of the microstructure grain size and by adjusting the composition of the intra granular phases. The development activities to reduce the HRE contents are not finished, a large supplementary potential exists and will be exploited soon.

Fig. 4 presents the TERRAMAG® Z- and R grades for applications up to 230°Cavailablein mass production. The specified H_{cJ} at the maximal operating temperature are H_{cJ}> 420 kA/m or H_{cJ}> 520 kA/m depending of the customer requirements.

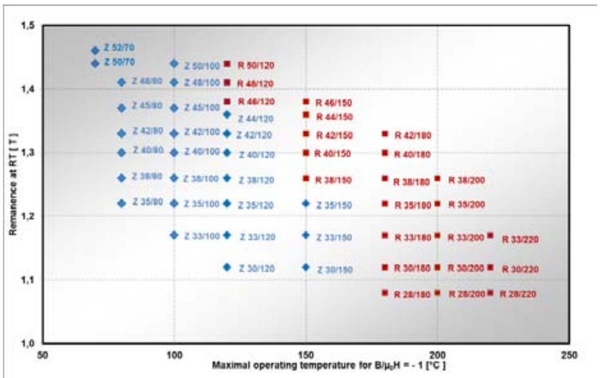

Fig. 4: TERRAMAG® grades of Z- and R- series available today in mass production

C. *NdFeB permanent magnets grades with very high energy density*

The HRE reduction and the new used manufacturing technologies offer a great potential to enhance the remanence and so the energy density of the NdFeB magnets. The remanence of an anisotropic Nd-Fe-B magnet is given by:

$$\mathbf{B_r \approx J_s * f_\phi * f_o * f_\rho} \qquad (2)$$

with:

$\mathbf{J_s}$= the saturation polarization of the ϕ-phase,

$\mathbf{f_\phi}$= the Volume fraction of the ϕ–phase in the microstructure,

$\mathbf{f_o}$ = the alignment degree of the ϕ-grains,

$\mathbf{f_\rho}$ = the relative density.

The saturation polarization J_s of the $Nd_2Fe_{14}B$ compound is 1.61 T, those of the compound $Dy_2Fe_{14}B$ is only 0.7 T [15]. The saturation polarisation of a $(Nd,Dy)_2Fe_{14}B$ compounds is resulting according to the fraction of Nd and Dy mixture. By reducing Dy, the value of $J_s$increases. The "low oxygen technology" effects the increase of the volume fraction $\mathbf{f_\phi}$of theΦ-phase in the microstructure, whereas finer powders and the optimization of the compacting technology effect the increase of the alignment degree $\mathbf{f_o}$of theΦ – phase.

The theoretical value of the maximum energy density was calculated to be 512 kJ/m^3 (~ 64 MGOe). Since 2002, the year of the discovery of NdFeB magnets, the energy density of industrial produced NdFeB magnets have been considerably ameliorate, its energy density jumped from 280 kJ/m^3 to 425 kJ/m^3 [14].

D. *The grain boundary (GBD) process*

In the 90s several papers reported that low melting additions containing Dy can locally create very big grains sizes up to 100 µm having non-uniform Dy distribution[18,25]: the grain boundaries are very rich in Dy, while the grain core are rich in Nd, and no Dy was detected. But despite the distribution uniformity and the anomalous grain sizes, the magnet showed high remanence and a considerable coercive force.

Later investigations have shown that the coercive force of sintered magnets can be dramatically improved by the diffusion along the grain boundary of Dy and Tb applied on the magnet surface [25]. This process to enhance the coercive force of the NdFeB permanent magnets is called **G**rain **B**oundary **D**iffusion (GBD) process. GBD became a very important manufacturing route to produce high coercive NdFeB magnets by saving HRE. The applied material containing the HRE can be mixtures of oxides and fluorides or a secondary low melting alloy, the so called the two alloys method, added to the fine powders [26,27].

Intensive R&D activities of BEC and of its Chinese partner resulted in the development of the TERRAMAG® magnets of the L-series, L like LEAN, produced by using the GBD process for magnets with a maximal thickness of 6 mm.

As an example, Fig. 5 gives the typical demagnetization curves of TERRAMAG® S-42/180 and R-S-42/180 grades, developed for operating temperature up to 180°C. At180° the specified H_{cJ}> 520 kA/m. The Dy content in the initial S product was about 8 wt. %. Due to technological progress, the Dy content could be reduced up to approximately 5 wt. % by conserving the temperature dependence of the magnetic properties. Further Dy content reductions are aimed using the GBD process for magnets with thickness up to 6 mm.

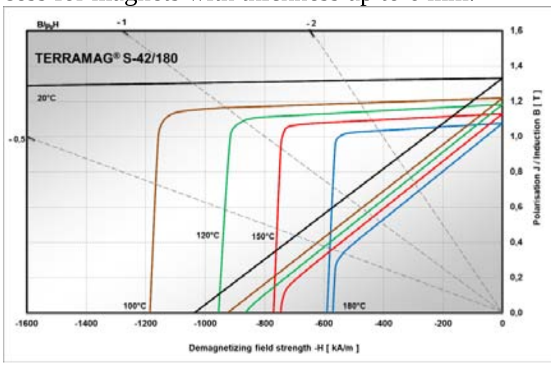

Fig. 5: Typical demagnetization curves J(H) and B(H) of the grades TERRAMAG® S 42/180 and R-S-42/180. TERRAMAG® S-42/180 is available in mass production since several years, it contains approximately 8 wt. % Dy. The grade R-S-42/180 has been recently developed and it contains less than 5 wt.% Dy.

IV. CONCLUSIONS

In the last 10 years, spectacular increase of the magnetic properties of sintered NdFeB magnets can be reported, today the diversity of the corrosion and temperature stable TERRAMAG®magnets developed by BEC and its Chinese partnerenable users to select appropriate magnetic materials for all important application fields.

For motor applications, which require higher temperatures, TERRAMAG®magnets for operating temperatures up to 230°C are available in mass production.Their maximal operating temperature for loading line B/μ_0H is definite by the specified minimal coercive force H_{cJ}.

By adjusting the alloy composition and the processing technology TERRAMAG®magnets with energy densities up to 425 kJ/m^{3}have been developed and are also available by serial production.

The content of the HRE has been significantly reduced and can so satisfy most of the requirements posed by the industrial applications.

The future development activities will be especially focused on the development of HRE – free and HRE – reduced TERRAMAG® magnets.

The very promising GBD technology will be pursued with a high priority.

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