

Grain Boundary Diffusion

A technique to reduce heavy rare earth requirement in NdFeB permanent magnets

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Introduction

Rare earth magnets are the strongest known permanent magnets and NdFeB is stronger than the alternate rare earth magnets – SmCo and SmFeN. NdFeB magnets get their magnetic strength from iron at 65 weight percent of the composition and from neodymium, the optimal rare earth for magnetic strength. However, the ability of NdFeB to perform at temperatures above about 60°C is achieved by substituting one of the heavy rare earth elements (HREEs) for some of the neodymium (Nd). The two widely used substitute HREEs are dysprosium (Dy) and terbium (Tb). There are problems with the substitution. First the magnetic strength decreases with an increase in HREE content. Secondly, the heavy rare earth elements are less abundant and considerably more expensive than Nd. Therefore, methods have been sought to resolve these two issues with one of the more successful being grain boundary diffusion (GBD).

Background

NdFeB consists of three components:

1. **Rare Earth:** One or more rare earth elements can be used. Neodymium (Nd) is preferred, but praseodymium (Pr) is commonly supplied along with the Nd in a material known as NdPr (sometimes called didymium). The ratio of Nd to Pr in didymium is between 70 and 80% Nd to 20 to 30% Pr depending on the raw material ore. Newer, low-cost grade alternatives include cerium (Ce) and lanthanum (La) substitution for up to half of the Nd which results in reduction in magnetic strength, but with considerably lower material cost. Dysprosium (Dy) and terbium (Tb) are referred to as heavy rare earth elements (HREEs) and are substituted to improve resistance to demagnetization.
2. **Transition metal (TM):** mostly iron (Fe) and often with small additions of cobalt (Co). Iron is a very strong ferromagnetic element and provides the majority of NdFeB's magnetic strength. Cobalt increases the magnet's Curie temperature (T_c) improving high temperature performance. Cobalt is also added to the grain boundary region to improve corrosion resistance.
3. **Metalloid element — boron (B):** The presence of boron causes the elements to arrange into a tetragonal crystal structure. This provides for a strong magnetic field only along the length of the crystal, the C-axis. This is called magneto-crystalline anisotropy. In the tetragonal crystal the C-axis is often referred to as the "easy axis" of magnetization. Carbon (C) has been tried as a substitute for B but results in inferior magnetic properties.

The (atomic) formula for the magnet alloy is $\text{Nd}_2\text{Fe}_{14}\text{B}$ indicating the atomic ratio of the element types. A more complete atomic formula might be written as $(\text{Nd,Pr,Dy})_2(\text{Fe,Co})_{14}\text{B}$. In patents and many technical papers each element is often subscripted showing the relative atomic amount. Also, since both Nd and Pr are now commonly used together, we often refer to the combined raw material as NdPr.

There are many commercial permanent magnets and each has advantages and shortcomings. In Figure 1 we see the temperatures over which each material can be used. The chart is simplified and we should now explain some complications regarding NdFeB.

The NdFeB has a low temperature limit. It can be used down to -140°C , but below that temperature, the tetragonal crystal lattice deforms causing the uniaxial anisotropy to devolve to a cone of anisotropy resulting in significant loss in magnetic strength. This can be avoided by substituting Pr for $\geq 80\%$ of the Nd.

The NdFeB upper temperature limit is due to spontaneous demagnetization from crystal lattice vibration which becomes severe as the temperature approaches the Curie temperature. Above the Curie temperature ferromagnetism disappears and the material becomes paramagnetic. A second, and very practical limiting factor, is that magnetic strength, quantified as magnetic flux density, B_r , or energy product, $(BH)_{\text{max}}$, decreases as temperature rises. The rate of decrease informs us that by 230°C , B_r is 25% lower than at room temperature and $(BH)_{\text{max}}$ is about 56% of that at room temperature. A third factor limiting high temperature application is that the magnet's resistance to demagnetization decreases as temperature rises. This can be compensated for by substituting a HREE, such as Dy or Tb, for a portion of the Nd but with serious loss of magnetic flux density. (See Figure 2.)

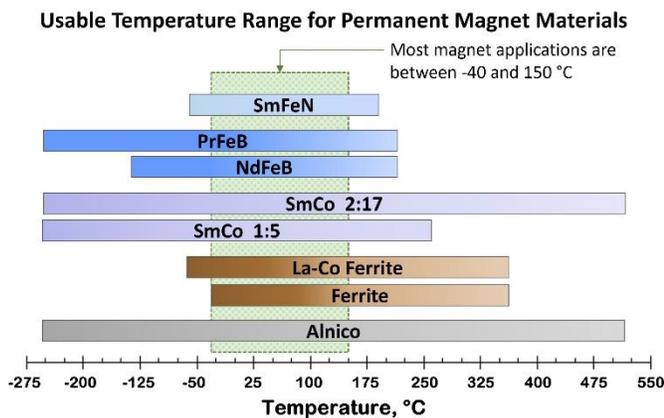


Figure 1. Usable temperature range for commercially available permanent magnets.^[12]

Resistance to demagnetization is quantified by the value of intrinsic coercivity (H_{ci}). A chart of the effects of adding Dy to the magnet composition is shown in Figure 2. The chart includes comparison lines for standard grades of NdFeB and for grades dependent upon optimized grain boundary diffusion (GBD). The naming convention of magnet grades often includes a suffix indicating minimum intrinsic coercivity and these are shown along the H_{ci} lines of the chart: M, H, SH, UH, etc. When comparing the two chart lines of H_{ci} versus Dy content, we see for any Dy content a higher B_r is achieved when GBD is used. By viewing the combination of the three lines, we see that for any value of H_{ci} a higher B_r is possible.

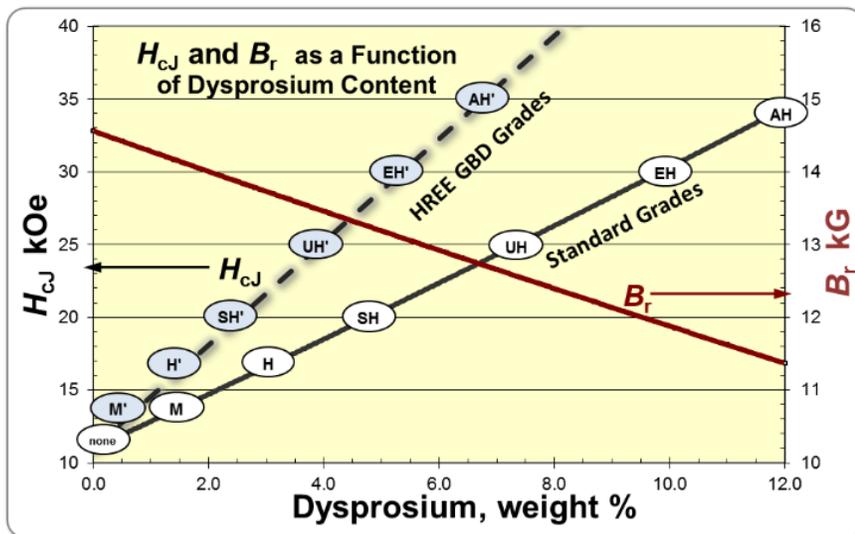


Figure 2. Residual induction (B_r) and intrinsic coercivity (H_{cJ}) as a function of dysprosium content for standard and for GBD grades.^[4]

Manufacturers usually show these GBD grades on charts along with conventional product such as in Figure 3 where grades with suffixes containing “G” are GBD products. They are indicated in color-coded boxes and appear above and to the right of standard grades. This chart was produced by Shin-Etsu. Other manufacturers offer similar grade charts.

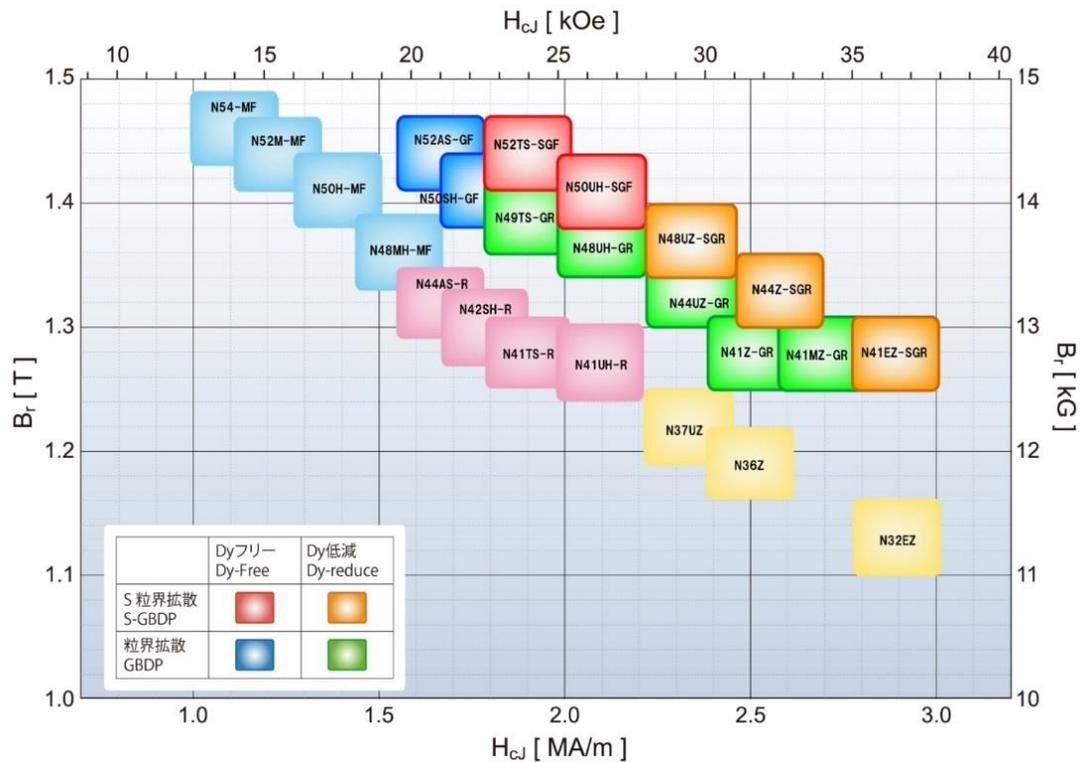


Figure 3. Chart of NdFeB magnet grades published by Shin-Etsu Magnetics. Color code indicates type of magnet. Downloaded 03-Nov-2022 from from: https://www.shinetsu-rare-earth-magnet.jp/e/products/chart_nd.html

Grain Boundary Diffusion

When we casually examine NdFeB magnets they appear to be solid “chunks” of metal. They are in fact a collection of particles (“grains”) held together by what we call a grain boundary. The metal in this boundary layer is often called the grain boundary phase and it is chemically and structurally different from the main NdFeB tetragonal crystal phase.

Grain boundary diffusion is the movement of elements from the surface of the magnet toward the center of the magnet. The magnet is heated to melt the grain boundary phase and permit metal atoms to move with relative ease through the liquid grain boundary phase. Thus, the surface with a high concentration of, for example Dy, will diffuse Dy through the liquid grain boundary phase to minimize the concentration gradient of Dy in the magnet. Refer to Figure 4. When Dy atoms are positioned adjacent to the grains of NdFeB the high temperature of processing causes some of the Dy to move into the NdFeB grain while some Nd atoms move into the grain boundary. This creates a core-shell structure with Dy in the outer surface of the NdFeB grains which increases the H_{cJ} of this grain. Since the Dy-enhanced layer surrounds the grain, the effective H_{cJ} of the entire grain is increased. Properties of the whole magnet are an average of the H_{cJ} of local regions, so the bulk magnet’s average H_{cJ} is also increased. In GBD treated magnets, Tb is often used as the HREE instead of Dy since Tb is more effective than Dy and with GBD less total HREE is required.

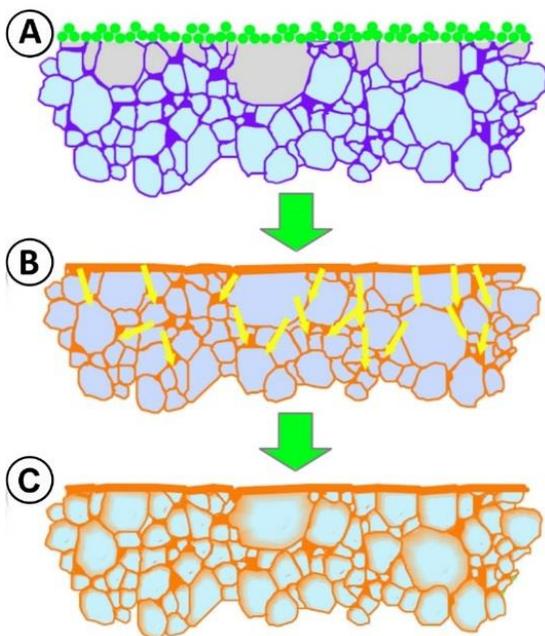


Figure 4. The grain boundary diffusion process described by Shin-Etsu.^[5,16] **A:** Coating powders, consisting of Heavy Rare Earth (HRE) compounds, are deposited on the magnet surface. **B:** The HRE elements diffuse into the magnet through the liquid phase around the grain boundaries. **C:** HRE elements react with the main 2-14-1 phase, creating a reaction layer at the interface.

The resulting distribution of dysprosium (or terbium) in the treated magnet has been compared with conventionally produced magnets. Refer to Figure 2 and to Table 1.

Table 1. Reduction in Dy content possible from use of GBD process. [18]

| Dy Content | |
|----------------------|---------------|
| Conventional Magnets | Low Dy Series |
| ~ 2 % | → 0 % |
| 4 ~ 5 % | → 1.5 ~ 2.5 % |
| 7 ~ 8 % | → 3.5 ~ 4.5 % |
| 8 ~ 9 % | → 5.0 ~ 6.0 % |

The GBD process results in different percentages of the HREE dependent on location within the magnets. The depth of diffusion is limited, from a practical standpoint of time and temperature, to between 3 and 4 millimeters from the surface of the finished magnet. In figure 5 we see the change in H_{cJ} as a function of distance from the surfaces of a 6 mm thick magnet. The base magnet composition yields an H_{cJ} of 1790 kA/m. At each magnet face the H_{cJ} is increased to 2270 kA/m and at a depth of 3 mm from each face, the H_{cJ} is increased to 2010 kA/m.

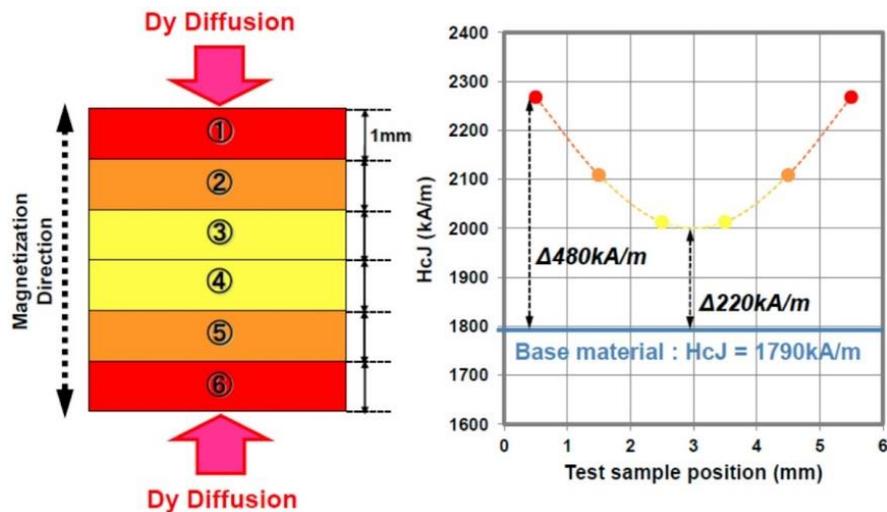
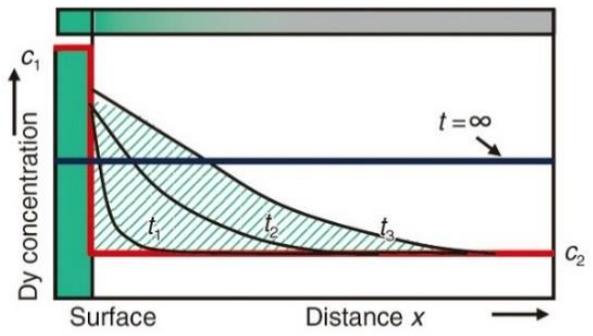


Figure 5. Coercivity as a result of diffusion from two faces of a magnet. [14]

Coercivity as a function of diffusion depth

Diffusion is defined as the net movement of a material (atoms, molecules, etc.) from an area of greater concentration to an area of lesser concentration. The result of diffusion over a limited time span is a gradient of composition. In grain boundary diffusion (GBD) the surface of the magnet remains high in concentration of the heavy rare earth element (HREE) while regions below the magnet surface increase in concentration. The rate of diffusion depends upon many factors including the thickness of the grain boundary, degree of liquidity (is it totally liquefied or partially solid), concentration gradient of the HREE between adjacent regions in the magnet, contamination within the grain boundary, size of the diffusing atom, etc. Figure 6 presents the mathematical model for the diffusion process. [3] There are practical limits on treatment temperature (too high promotes additional grain growth) and time (long times are costly in energy consumption, production capacity, and grain growth). [14]



$$C = c_1 + \frac{(c_2 - c_1)}{2} \left[1 + \operatorname{erf} \left(\frac{x}{2\sqrt{D_0 \cdot t \cdot \exp(-E/k_B T)}} \right) \right]$$

- C: Dy concentration
- c_1 : Surface concentration
- c_2 : Base concentration
- x: Distance
- E : Activation energy
- D_0 : Vibration factor
- k_B : Boltzmann's constant
- t: Time
- T: Temperature

Figure 6. Basic diffusion model.^[3]

The coercivity distribution in a magnet after diffusion of the heavy rare earth element (Dy or Tb) is shown in Figure 7. The concentration of the HREE decreases exponentially from the magnet surface to locations below the surface reaching a maximum (practical) depth of about 4 mm. To achieve a greater depth of penetration requires excessive thermal processing time and temperature.

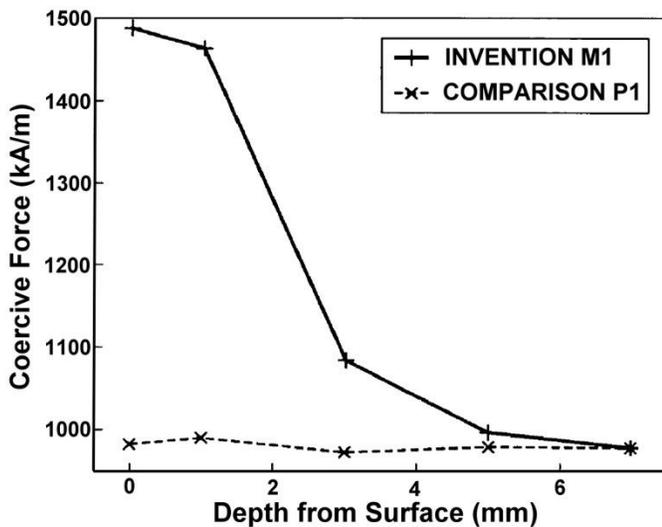


Figure 7. Coercivity as a function of depth from magnet surface.^[22]

After diffusion of the HREE into the grain boundary, an exchange occurs wherein some HREE enters the NdFeB grain and a corresponding amount of Nd exits into the grain boundary. The result is a “core-shell” structure which can be seen in Figure 8. The establishment of the shell requires adequately high temperature to permit the elemental exchange. The increase in processing temperature shows the increasing shell thickness as heavier yellow coloration around the NdFeB grains.

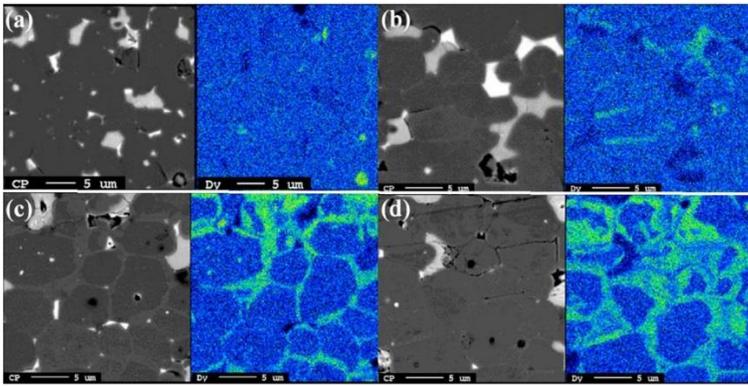


Figure 8. Scanning electron microscopy and Dy-mapping images of the DyF₃ dip-coated magnets at a depth of 20 microns from the surface as a function of the first post-sintering annealing temperature. Annealed at: (a) 700°C, (b) 800°C, (c) 900°C, and (d) 950°C. Blue indicates the main phase and yellow indicates the presence of HREE.^[7]

Maximum diffusion occurs to a depth of 3 to 4 mm limiting total magnet thickness to about 6.5 mm in the smallest dimension, generally in the direction of magnetic orientation. It is often the case that 4 sides or less of a rectangular magnet will be treated with GBD. That is apparent in the testing of sample magnets provided to GM and shown in Figure 9.^[6]

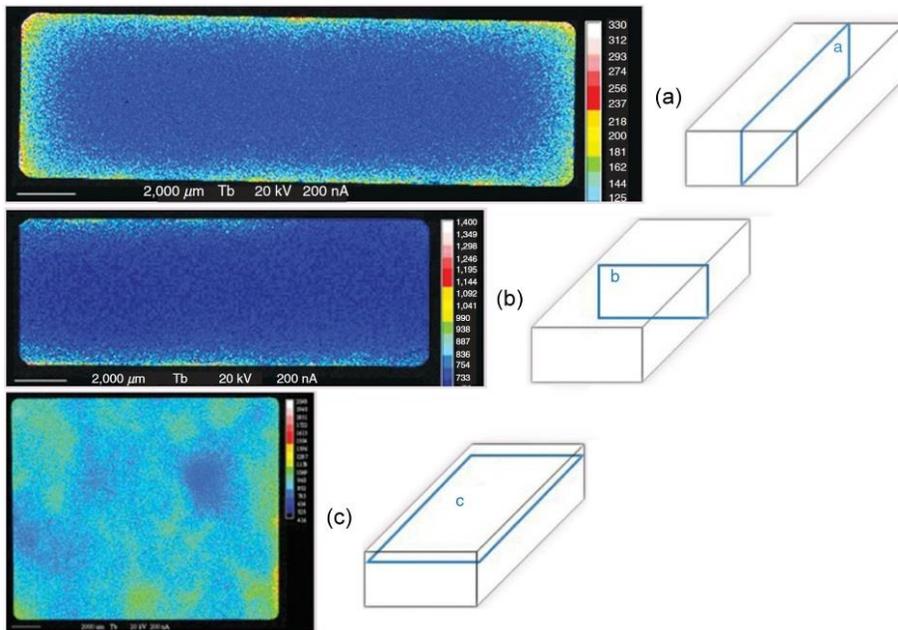


Figure 9. EPMA maps of different cross sections of a Tb-diffused magnet. (a) A Tb map of a plane at the center of the magnet perpendicular to the width. (b) A Tb map of a plane at the center of the magnet perpendicular to the length. (c) A Tb map of a cross section at approximately 0.5 mm from the large surface of the magnet.^[6]

The Electron Probe Microanalysis (EPMA) shows concentration of terbium as a yellow color in the images of Figure 9. From the EPMA map of (b) we see that the edges of the magnet have not been GBD processed. From the EPMA image of the face of the magnet it can be seen that diffusion of the Tb

is non-uniform. This is identified by the variation in Tb density indicated by variations in the yellow color density. Further, concentration of Tb is highest at the edges and corners where diffusion takes place in two or three dimensions.

Magnet properties are generally shown as either the hysteresis loop for regions of maximum HREE concentration or as an overall average for the magnet. The coercivity shown in Figure 5 is that of the region at the specified depth from the magnet surface. In Figure 10 hysteresis curves are presented from two regions: the corner of a magnet representing the highest concentration of HREE and from the very center of the magnet representing the minimum (or “base”) concentration of HREE.

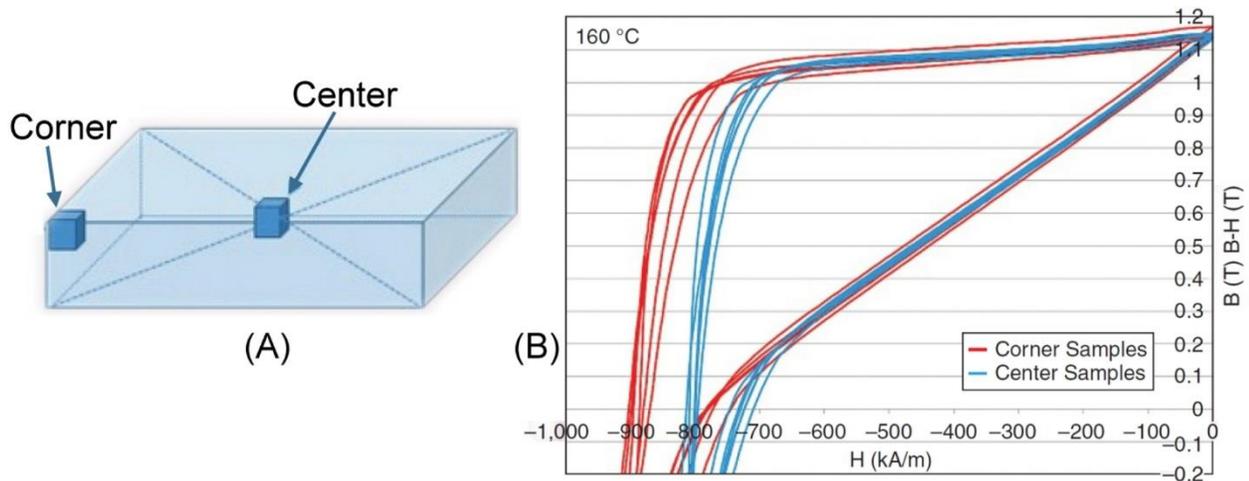


Figure 10. (A) Schematic of magnet locations of samples for VSM measurements. (B) Second quadrant BH curves for corner and center 2-mm cubic samples machined from magnets with Tb GBD. Edited from reference.^[6]

This effect can be shown schematically as in Figure 11 with H_{cJ} as a function of depth into the magnet and near edges and corners.

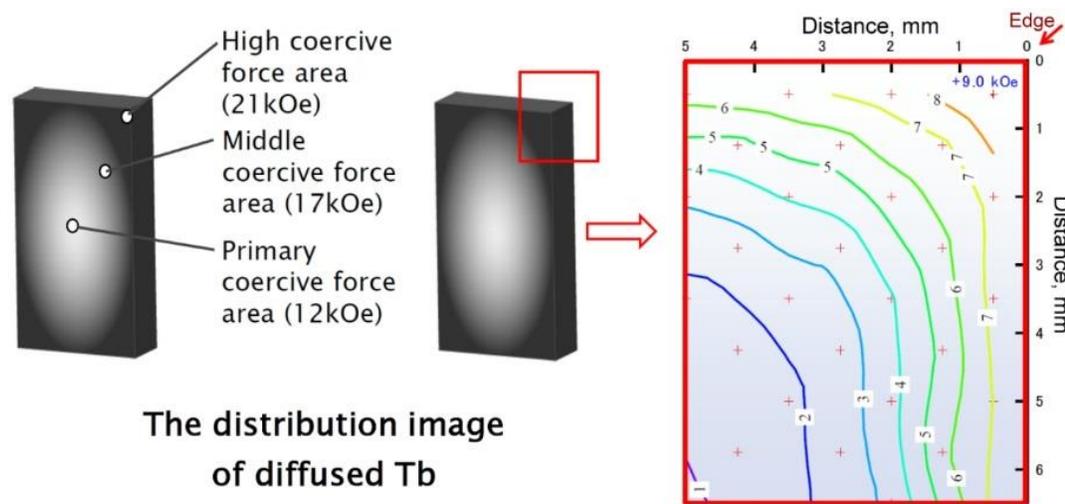


Figure 11. Variation of intrinsic coercivity as a function of location within a rectangular magnet.^[5,16]

This should immediately alert one to the possibility of selectively treating a magnet to insert HREE where the greatest resistance to de-magnetization is required. This might be called “selective area grain boundary diffusion” as illustrated in Figure 12.

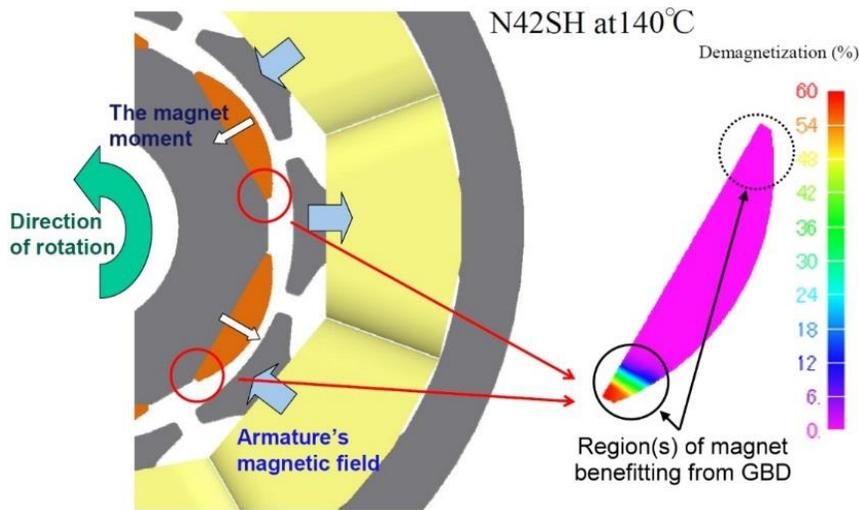


Figure 12. Selective area GBD. Circled region of the magnet benefits from GBD to enhance coercivity. Both edges of the magnet might be treated to eliminate need to identify the enhanced edge for handling and assembly.^[5]

Figure 12 shows that the treated area can be considerably less than the surface of the whole magnet, perhaps less than 20% of the total magnet surface. Treating opposite edges can eliminate the chance of incorrectly positioning magnets.

Magnet size limitations

Due to the limited distance over which diffusion occurs, magnet coercivity is only increased within this region and is only maximized very close to the magnet surface. Therefore, a maximum for the thinnest dimension of the magnet is about 6.5 mm (0.165”). Treating larger magnets is possible but with lesser effect on overall coercivity. When a magnet is measured either by VSM or hysteresigraph, the output properties are the average of the entire magnet. Further, the demagnetization curves are slightly distorted by the presence of regions of different intrinsic coercivity. For example, Figure 13 shows a deviation from a very square intrinsic curve (Before GBD curve) to one with a pronounced slope after the knee of the curve (After GBD curve).

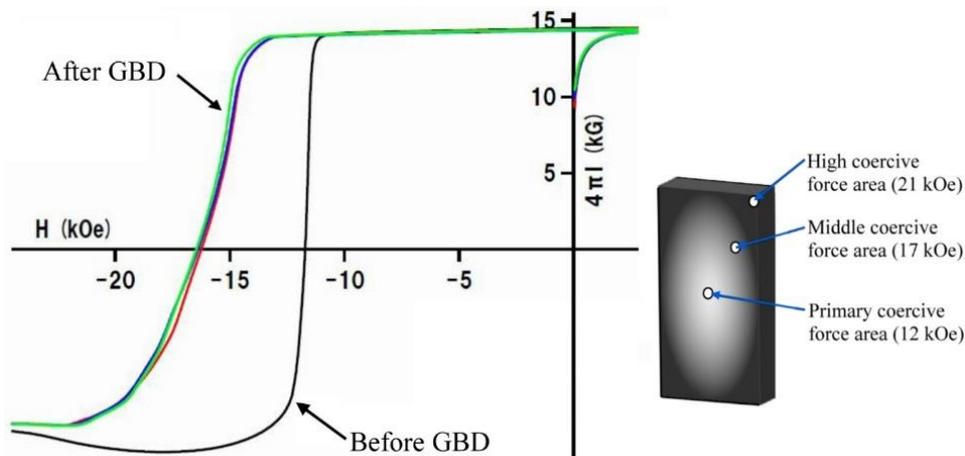


Figure 13. Hysteresis curves and coercive field strength distribution in GBD processed magnets.^[6]

HREE content of the starting magnet can be adjusted so that there is adequate intrinsic coercivity for normal demagnetizing field exposure and the GBD enhances coercivity in regions experiencing greater de-magnetizing fields. For example, a Dy content of about 4 weight percent results in an intrinsic coercivity of 1600 kA/m (20 kOe). Performing GBD at edges and corners can produce an edge coercivity greater than 2400 kA/m (30 kOe) while the bulk of the magnet remains at 1600 kA/m.

Thin GBD processed magnets may be stacked to build a magnet of greater thickness though the production cost for this may be prohibitive. Referring to Figure 14, using the layered structure to build up total magnet thickness both reduces eddy currents and takes advantage of the GBD process throughout the thickness of the magnet.

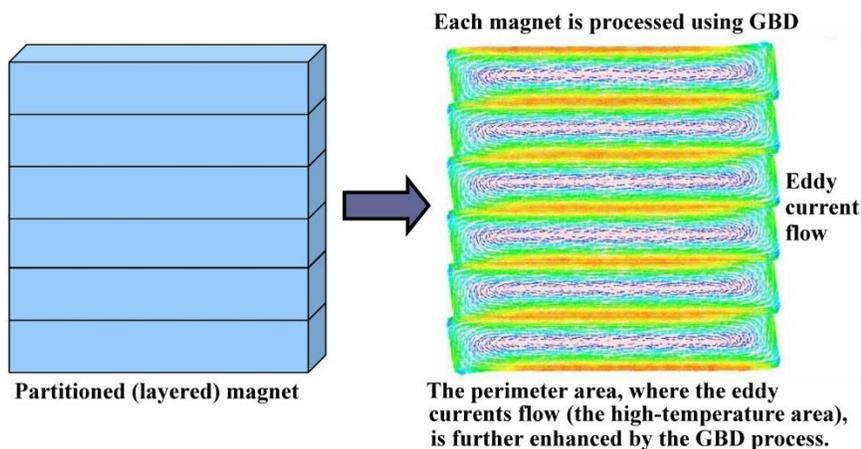


Figure 14. Using both partitioning and grain boundary diffusion processing, the heat resistance of NdFeB magnets can be greatly improved.^[5]

Dysprosium versus Terbium

Dysprosium has been the preferred HREE for several decades due to its greater abundance than terbium and considerably lower cost. However, terbium is more effective at increasing H_{cj} than dysprosium and has a lesser effect on reducing B_r . (Refer to Table 2.) H_A is the symbol for the

anisotropy field which is a measure of the potential resistance to de-magnetization. We see that H_A of Dy is more than twice that of Nd and Tb is 1.5x that of Dy. J_s is saturation polarization, the maximum intrinsic magnetic flux density which is closely related to B_r . Both Dy and Tb result in a significant reduction of J_s , though Tb is almost identical to Dy. Therefore, for maximum effect where the GBD process is utilized, terbium is often the preferred HREE.

Table 2. Selected Magnetic Properties of Terbium and Dysprosium.^[8]

| Compound | H_A MA/m | J_s T |
|---------------------------------------|---------------|-------------|
| Nd₂Fe₁₄B | 5.3 | 1.61 |
| Dy₂Fe₁₄B | 11.9 | 0.72 |
| Tb₂Fe₁₄B | 17.5 | 0.70 |

The difference in magnetic performance from using Tb instead of Dy is shown in Figure 15 from Hirota et al.^[2] The dotted demagnetization curve shows the reduction in B_r from conventional production methods which require high HREE content throughout the bulk magnet to achieve high H_{cJ} . The solid line curve with H_{cJ} of 1050 kA/m is for a magnet produced with no (or very little) HREE. In the other curves, we see that DyF₃ is more effective at increasing H_{cJ} than Dy₂O₃ and that TbF₃ is the most effective. In all GBD cases, the B_r remains close to that of the original magnet. The intrinsic curves of both the original magnet and that made by the binary alloy method are more “square” at the knee of the curves. That is, the value of H_k/H_{cJ} is closer to 1 and the slope past the knee of the demagnetization curves is closer to vertical.

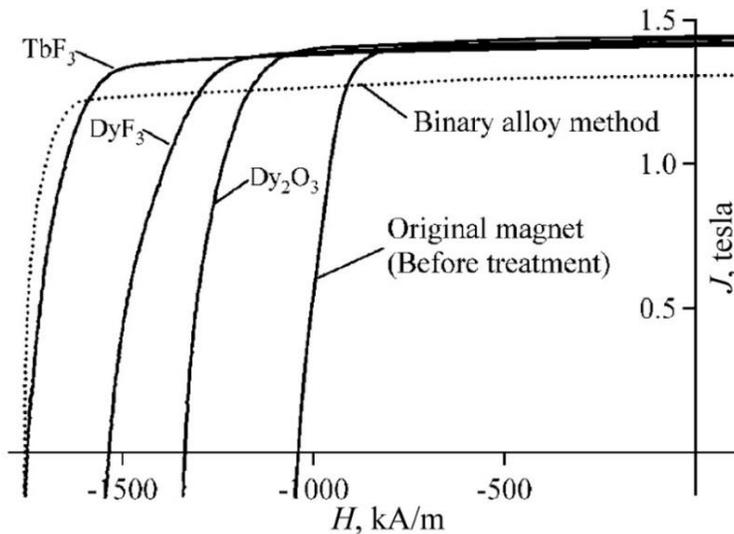


Figure 15. Demagnetization curves of the original magnet and magnets after the GBD process with Dy₂O₃, DyF₃ and TbF₃, and a magnet produced by the conventional binary (dual) alloy method.^[2]

The effectiveness of producing high intrinsic coercivity via GBD is compared to conventional magnet processing in Figure 16. The figure shows the weight percent of DyF₃ or TbF₃ required to produce the indicated H_{ci}. Very importantly the values of H_{ci} are for a magnet of thickness of 2 mm.

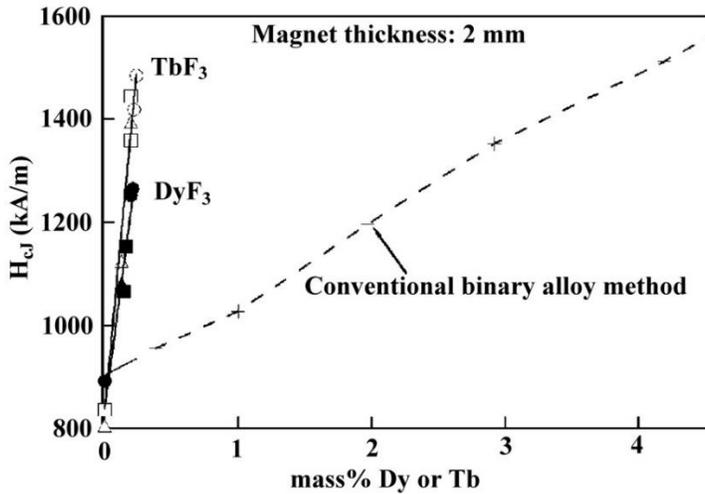


Figure 16. Coercivity plotted against Dy or Tb contents in various magnets.^[2]

Performing Grain Boundary Diffusion

Figure 17 presents a typical process flow chart for making NdFeB magnets. Standard magnets are manufactured without the three steps labeled as “Grain Boundary Diffusion Process.” GBD magnets would, of course, include these three steps. Of importance is that the GBD steps occur after the magnets are machined to size and shape. The Grinding shown at the end of the GBD process is for cleaning residual diffusion materials from the surface of the magnet. Surface treatment includes any of the normal coating processes.

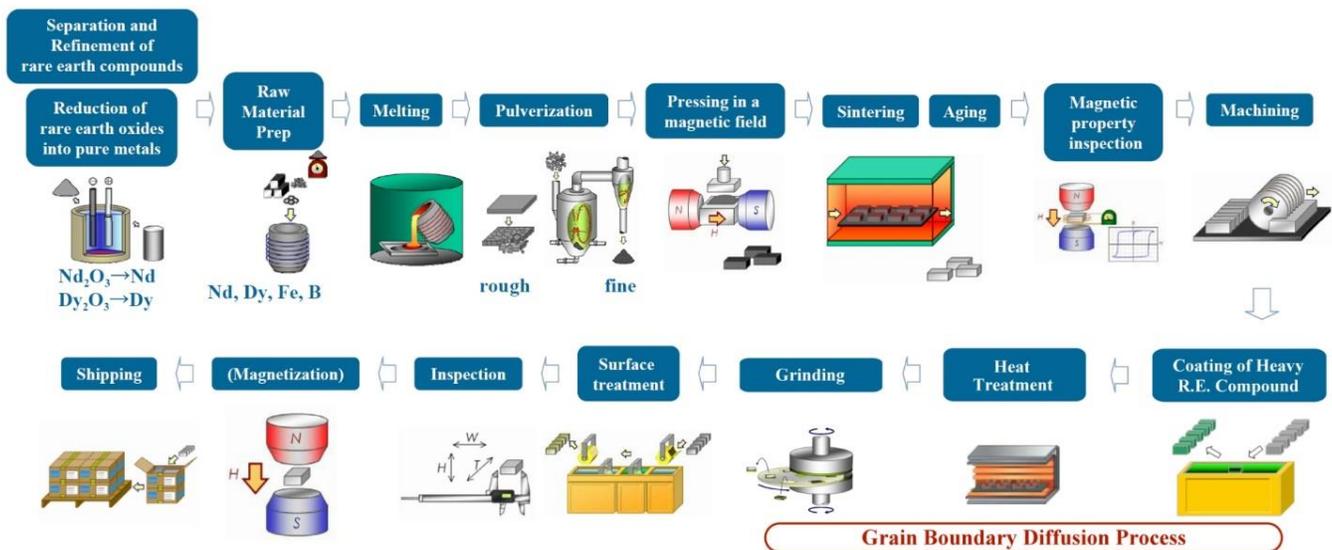


Figure 17. Manufacturing process for NdFeB magnets including the grain boundary diffusion (GBD) process. Graphic depictions from Shin-Etsu edited for clarity.^[5,16]

Measurement of Magnetic Properties

Measurement of magnetic properties presents a challenge for both the magnet producer and the user. It is often impractical to mechanically section magnets to test and verify performance. Search coil, Helmholtz coil, and gauss probe tests can be useful for determining total flux density of the magnet and field around the magnet but these methods do not produce information about intrinsic coercivity. One is left measuring the entire magnet in a VSM or hysteresigraph and that produces an averaging of properties for magnetic flux density and a biased demagnetization curve shape. (Refer to Figure 13.) The value of H_{cJ} may approximately indicate performance of the magnet. But H_{cJ} values and demagnetization curve shape will change as a function of magnet thickness. Thicker magnets will have a smaller magnet volume exhibiting enhanced H_{cJ} as presented in Figure 18.

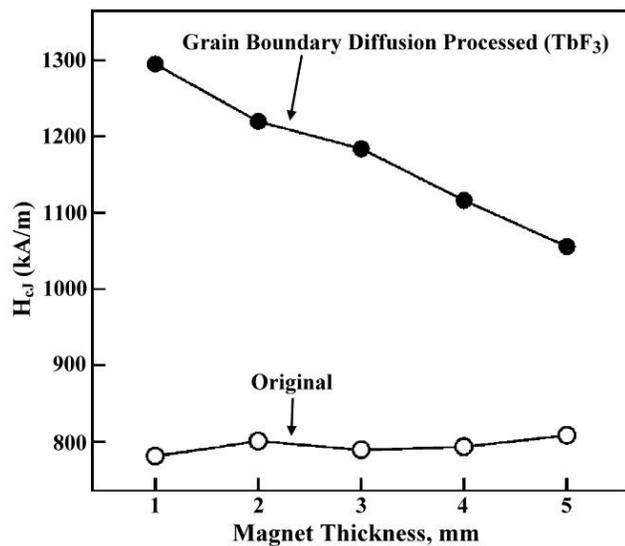


Figure 18. Coercivity as a function of magnet thickness, diffusion from both sides.^[2]

The problem becomes more complicated when selected area GBD is utilized. It may be necessary to construct test fixtures that reflect enhanced H_{cJ} at locations within the magnet for the adequate satisfaction of design requirements.^[16]

Other Issues

We've mentioned that either Dy or Tb may be used and that the compound containing the HREE might be an oxide, a fluoride or other. There are multiple techniques for applying the HREE either as an element or a compound such as electrophoretic coating, physical vapor deposition (PVD), application by spray painting, printing, etc. Some of these techniques have been patented and others are performed as proprietary (company secret).

An issue related to effectiveness of diffusion is the grain boundary thickness. The grain boundary composition and thickness have been causes of magnet corrosion through the galvanic couple between the grain boundary phase and the NdFeB main phase. Further, the rare earth-rich grain boundary phase has been the subject of hydrogen absorption, volume expansion and magnet decrepitation. The magnet industry has attempted to improve resistance to corrosion by minimizing the grain boundary

thickness and by adjusting its composition. The grain boundary phase also influences the temperature required for liquid phase sintering. The grain boundary composition is a complicated topic in its own right and will not be covered here.

The potentially thicker grain boundary present to facilitate diffusion has the potential to increase magnet corrosion.^[19] Figure 19 provides the weight loss due to corrosion of GBD processed magnets compared to magnets without GBD.

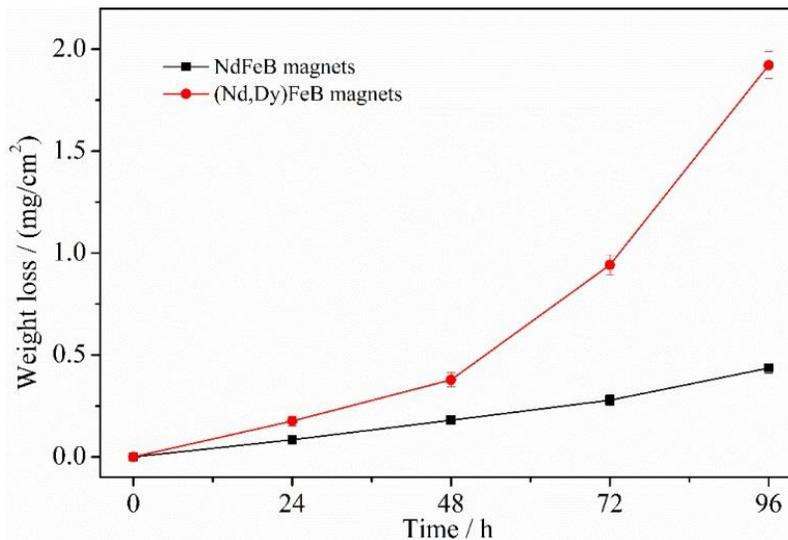


Figure 19. Weight loss of pristine Nd-Fe-B magnets and diffusion-treated (Nd,Dy)-Fe-B magnets for 24, 48, 72 and 96 hours.^[19]

The conclusion of reference 19 states: Weight loss from autoclave testing and “potentiodynamic polarization curves confirm that the corrosion resistance of (Nd,Dy)-Fe-B magnets is lower than that of pristine Nd-Fe-B magnets due to the large amount of rare earth and the wide grain boundaries, which provide a fast corrosive channel for external corrosive media.”^[19]

There are recently issued patents related to affecting the HREE introduction into the bulk magnet in such a manner as to minimize the amount required relative to the binary alloy method. HREEs are introduced in the starting powders and unlike with GBD the HREEs are located uniformly throughout the bulk magnet. This process is being called grain boundary engineering (GBE) with initial patents assigned to Noveon, a Texas company formerly known as Urban Mining. There is a potential for GBE to also minimize corrosion potential for NdFeB magnets. Because the bulk magnets are uniform in composition and structure throughout, demagnetization curves accurately represent the complete magnet and all sub-parts thereof. Importantly, compositional uniformity permits magnets of any size to be manufactured — they are not limited to 6.5 mm thickness as in GBD.

The techniques of GBD and GBE provide reduced HREE while increasing H_{cJ} . Alternative strategies for HREE reduction include NdFeB grain size reduction. Finer grains increase H_{cJ} until the size approaches the super-paramagnetic limit.^[20,21] However, smaller grain size increases material reactivity which requires very careful handling and protection from exposure to air and moisture until the powders are densified in sintering.

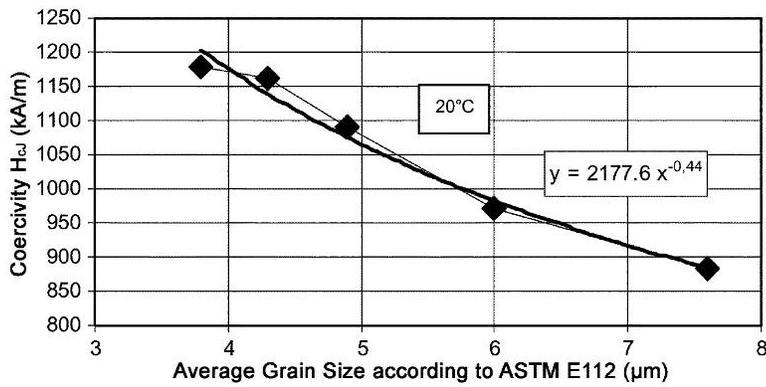


Figure 20. Dependence of coercivity on average grain size.^[21]

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