Electroplating Dysprosium from IL-Based Solutions: A Promising Electrochemical Step to Produce Stronger High Performance Nd(Dy)-Fe-B Sintered Magnets

Gottfried Suppan,a,b Manfred Ruehrig,b Andreas Kanitz,b and Heiner J. Goresa,c∗

a Institute of Physical and Theoretical Chemistry, University of Regensburg, 93053 Regensburg, Germany
b Siemens AG, 91058 Erlangen, Germany
c Institute of Physical Chemistry, Westfälische Wilhelmsuniversität, 48149 Münster, Germany

An electrodeposition replacement to the commonly used physical vapor deposition methods used in enhancing the coercivity of Nd-Fe-B sintered magnets has been proposed. Dysprosium was galvanostatically electroplated on Nd-Fe-B sintered magnets that were previously electrochemically coated with copper. Electroplated dysprosium films were characterized by X-ray photoelectron spectroscopy, depth profiling, scanning electron micrographs, and energy dispersive X-ray spectroscopy. XPS analysis after argon ion sputtering indicates that minor impurities are only superficial as their atomic fractions in the sample nearly disappear with increasing etching time. The electrochemistry of synthesized dysprosium bis(trifluoromethylsulfonyl)imide dissolved in the air- and water-stable ionic liquid 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide was studied by cyclic voltammetry. Electroplating of metallic dysprosium was followed by a heat-treatment above the melting temperature of the Nd-rich phases of the sintered magnet base body whose magnetic characterization was performed in a hysteresis loop tracer for hard magnetic materials.

Due to it being the highest energy product of any known magnet, Nd-Fe-B magnets are widely used in high performance electric machines. The energy product \((BH)_{\text{max}}\), typically given in [kJoule/m³] is the figure of merit for the maximum magnetic energy a permanent magnet can provide in a real application like an electrical motor. \(B\) denotes the magnetic flux density [in Tesla] in the material and \(H\) is the value of the magnetic field strength [in A/m] acting on the magnet. At zero field the magnet has a remanent polarization \(B_{r}\), which gradually decreases with applied reverse fields, until the polarization changes direction (irreversibly) at negative field values of \(H_{c}\) (= coercive field). The coercivity \(H_{c}\) in Nd-Fe-B magnets decreases significantly with rising temperature and the operating temperature in motor applications can reach up to 200 °C.3 Magnet reversal or demagnetization of the permanent magnets has to be unconditionally avoided at any reverse field or thermal conditions during operation. The coercivity is the main material property limiting the application of high energy product Nd-Fe-B magnets. A method to overcome the risk of demagnetization at high temperatures is to further increase the coercivity of the material, so that \(H_{c}\) and \((BH)_{\text{max}}\) still show adequate values at operating conditions. Up to now, this has been achieved by alloying heavy rare earth elements (HREE) such as dysprosium or terbium as substitution of light rare earth elements in the magnetic grains (Nd₂Fe₁₄B-phase)5 on an industrial scale. HREE increase the magnetocrystalline anisotropy.3 Certain applications call for very high coercivity via substitution of Nd by Dy of up to about 10 wt%. However, substitution of LREEs by HREEs is not only very costly due to the scarcity of HREEs in comparison to Nd but causes a decrease in remanence \(B_{r}\) due to anti-ferromagnetic coupling as well.3

A more efficient method to increase \(H_{c}\), referred to as “grain boundary diffusion” (GBD) has been reported.3 In this process, HREEs are not alloyed with the other components but Nd-Fe-B magnets are only coated with HREEs that are then allowed to diffuse into the bulk material during a heat-treatment above the melting temperature of the intergranular Nd-rich phases. In comparison to traditional alloying, HREEs are distributed around the grain boundary phases, thus less amount of HREEs is needed. GBD-treated magnets also show enhanced coercivity without severe decrease in remanence.4 While the decrease in \(B_{r}\) for conventionally processed Nd-Fe-B-sintered magnets typically amounts to about 4% per wt%, added Dy as reported by Yu et Al.,7 GBD-processed magnets suffer a reduction of about 0.5% for the same addition.8

According to literature, magnets have been coated with Dy by physical vapor deposition (PVD sputtering).9,10 Sputtering techniques deliver high purity metallic films with very good adhesion resulting in excellent magnetic properties for the treated magnets, however this procedure is very costly due to the required high vacuum conditions and relatively low deposition rates making it cost ineffective as a large-scale industrial process. In addition to vacuum deposition methods, HREE sources for the grain boundary diffusion process (GBDP) have been prepared by coating the sintered magnets with pastes containing HREEs (mostly as fluorides or oxides).6,11 Rare earth metals are prone to oxidation and it is well known that both oxygen and carbon impurities in the magnet adversely affect the permanent magnetic properties.12 The introduction of Dy as DyF₃ or Dy₂O₃ has also proved not to be as efficient as that of pure metallic Dy.13 Therefore, it was expected that using high purity metallic films result in superior magnetic properties for the GBD-treated magnet.

For corrosion prevention in harsh environments Nd-Fe-B permanent magnets are usually coated by protective layers. Electrochemical coatings with metallic layers from Al, Mn, Zn or Ni respectively and alloys thereof have been reported.14–16 For example, the deposition of nanocrystalline aluminum in non-chloroaluminate ionic liquids was reported by El Abedin et al.17 The electrodeposition of rare earth metals in ILs such as Y, Gd and Yb was reported by Glukhov et al.18 An extensive review of rare earth metal deposition from ionic liquids was given by Cui et al.19 A general overview of electrodeposition of non-metallic metals, including Li, Na, Mg, Al, Ti, Zn, Cr, Cu and alloys thereof as well as several semiconducting materials such as Si, Ge and GaAs, InSb, ZrTe, CdTe from ionic liquids is reported in a book entitled “Electrodeposition from ionic liquids”.20 Borisenko et al. have reached a coating thickness of tantalum of up to 700 nm from the ionic liquid 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide (BMP TFSI).21

In this study, the authors want to present an alternative method to introduce heavy rare earths into Nd-Fe-B sintered magnets by electroplating, a technique competitive to established procedures based on costly and slow physical deposition techniques. This process is based on an air- and water-stable, hydrophobic ionic liquid due to the favorable properties of these electrolytes over their organic counterparts, as high vapor pressures, flammability and toxicity of...
Experimental

As a starting material for the magnet, commercially available sintered magnetic materials (Vacodym VD 722 HR, Vacuum schmelze GmbH & Co KG, Hanau, Germany) were used. These magnets have an intrinsic Dy content of 0.5 wt%. The sample magnets had dimensions of 0.3 x 0.3 x 1.0 cm. The magnets were thermally demagnetized and remained uncoated (no corrosion protection layer) after sintering. BMP TFSI was purchased from Sigma Aldrich and dried at 100°C at 10⁻³ bar for 17 h to reach a water content of 5 ppm as determined by Karl Fischer titration (Titroline KF trace, Schott Instruments GmbH). Dysprosium bis(trifluoromethylsulfonyl)imide (Dy(TFSI)₂) was synthesized as described in Ref. 25 but Dy₂(CO₃)₃ was used as starting material instead of Dy₂O₃. This approach resulted in a faster reaction time, in accordance with Ref. 26, as well as a in a higher yield (96.3 wt%) than via the oxide route (89 wt%). Bis(trifluoromethane)sulfonimide (Sigma Aldrich) was dissolved with a slight excess of Dy₂(CO₃)₃ (5 mol%) in MilliQ quality water. The solution was filtered to remove remaining carbonate. Water was removed in a rotary evaporator and the salt was subsequently dried at 200°C and a pressure of 10⁻¹ bar for 41 h to remove remaining water to a remaining content of below 50 ppm as monitored by Karl Fischer titration. Elemental analysis of the product was carried out on a Elementar Vario-EL (Elementar Analyse Systeme GmbH) (calc: C: 7.19; N: 4.19; S: 19.18; found: C: 7.6; N: 4.23; S: 19.2); Pure Dy and Cu sheets (99.99%) were purchased from Sigma Aldrich. After drying, all substances were stored in an argon-filled glove box (MBraun, Darmstadt, Germany) controlled by Nova 1.6 software. All prepa-

Results

Electrodeposition of Cu.— To enhance the adhesion of the deposited Dy metal on the magnetic samples, a Cu layer was deposited first as Dy could be deposited on Cu substrates as a dense, adhesive layer of metallic luster. Small additions of Cu should not be detrimental to the magnetic properties of the Nd-Fe-B samples. The bath used for Cu deposition consisted of 0.2 mol kg⁻¹ copper trifluoromethanesulfonate in 1-butyl-3-methylimidazolium trifluoromethanesulfonate (BMIM OTf). Successful deposition of Cu from air and water stable ionic liquids has been reported in the literature. Stainless steel (BMIM OTf) saturation with copper trifluoromethanesulfonate. The bath used for Cu deposition consisted of 0.2 mol kg⁻¹ copper trifluoromethanesulfonate in 1-butyl-3-methylimidazolium trifluoromethanesulfonate (BMIM OTf). Successful deposition of Cu from air and water stable ionic liquids has been reported in the literature.25

Cyclic voltammetry on Au substrate.— The cyclovoltammetric behavior of the Cu species was investigated at 25°C with a scan speed of 5 mV s⁻¹. An Au-sputtered quartz crystal was used as the substrate, see Fig. 1. The potential was swept in the negative direction from the OCP to ~2.5 V vs Ag/Ag⁺ to ~1.4 V to OCP. Two redox processes were observed. The first redox couple a₁/c₁ can be attributed to the stripping (a₁) and deposition (c₁) of Cu (0), while the second a₂/c₂ is associated with the oxidation of Cu(I) to Cu(II) at a₂ and its reduction from Cu(II) to Cu(I) at c₂ starting at 0 V, respectively. The absence of a nucleation loop at a₂/c₂ indicates a facilitation of the electrodeposition of Cu on Au. Preliminary experiments showed a similar behavior for other Cu sources such as Cu(TFSI)₂ and other ionic liquids such as BMP TFSI (data not shown).

SEM images and composition analysis on Nd-Fe-B-samples.— Deposition time of 15 min at 100°C at ~1.0 V vs Ag/Ag⁺ produced coated Nd-Fe-B-samples with metallic copper coatings of about 100 nm thickness determined by the increase in sample weight and passed current of ~0.62 C, which resulted in a current efficiency of 88%. The layer thickness was calculated by the weight difference between the uncoated and the coated samples using a high precision microbalance with a resolution of 10 µg before and after deposition, respectively. SEM micrographs of the copper deposits are shown in Fig. 2a. The surface morphology was granular and the particle diameter was approximately 4 µm. Composition analysis of the deposits was determined by EDX (Fig. 2b) which shows pure copper and just minor N, O and F impurities.

Electrodeposition of Dy.— The deposition of dysprosium from phosphonium-based ionic liquids has been reported in the literature.25 We used a bath of 0.2 mol kg⁻¹ Dy(TFSI)₃ in 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide for electroplating Dy metal.

![Figure 1. Cyclic voltammogram of 0.2 mol kg⁻¹ Cu(OTf)₂ in BMIMOTf with a scan rate of 5 mV s⁻¹ at 25°C on Au substrate.](image-url)
Cyclic voltammetry of Dys on Au substrate.—Cyclic voltammograms of 0.147 M Dy(TFSI)$_3$ in BMPTFSI recorded at 25°C at various scan rates on a gold substrate against Ag/Ag$^+$ are shown in Fig 3a. A shift of the cathodic peak potentials to the cathodic side is seen for increasing scan speeds as well as an increase of the cathodic peak currents. Increasing the temperature results in the shift of deposition potentials to more positive values, as well as the increasing current, as shown in Fig. 3b. This is ascribed both to an increase in mobility of the electroactive species toward the electrode surface as well as to a reduced nucleation overvoltage at elevated temperatures. The cathodic peak appearing around –1.25 V in the voltammograms is ascribed to the reduction of Dy(III) to Dy(0), as no other reductive processes could be observed. The reduction of Dy in this system is irreversible, shown by the absence of an anodic peak corresponding to the oxidation of Dy(0). Irreversible behavior of the Dy plating process was also shown by Lodermeyer et al. using a solution of dysprosium triflate and dimethylpyrrolidinium triflate dissolved in dimethylformamide (DMF). The anodic peak at +0.75 V corresponds partly to the breakdown of the TFSI-anion as well as to the oxidation of the Au-electrode. This behavior of BMPTFSI has been described by Endres for ultra-pure BMPTFSI an Au (111). As shown in Fig. 4, the cathodic current density increases linearly as a function of the square root of the scan rate indicating a diffusion controlled process for the reduction of Dy. Fig. 5 shows the shift of cathodic peak potential $E_{pc}$ as a function of the scan rate $v$ obtained from the cyclic voltammograms in Fig. 3a. The peak potential plotted against the logarithm of the scan rate log(v) shows a linear behavior, indicating that the reduction process is irreversible.

The relationship between the half peak potential $E_{p/2}$ and the peak potential $E_p$ for an irreversible electrochemical process, see Ref. 31 is:

$$|E_p - E_{p/2}| = 1.857 \frac{RT}{\alpha n F}$$

Where $\alpha$ is the charge transfer coefficient, $n_\alpha$ is the number of exchanged electrons, $R$ is the gas constant, $F$ the Faraday constant and $T$ the absolute temperature.
the absolute temperature. From Eq. 1 the value for \((\alpha \times n_2)\) was calculated to be 0.76 at 25 
°C.

Eq. 2 shows the relationship between the current density and the scan rate for an irreversible system:

\[ J_c = 0.4958n_2F^{1/2}c_0D_0^{1/2}v^{1/2}(\alpha n_2/RT)^{1/2} \]  

[2]

where \(D_0\) denotes the diffusion coefficient, \(c_0\) the concentration of the electrochemically active species and other constants have their previously defined meaning. With the previously obtained value for \((\alpha \times n_2)\) as determined from Eq. 1, \(D_0\) can be determined from Eq. 2 and from the slope of the plot of \(J_c\) against \(v^{1/2}\). The resulting value for \(D_0\) was calculated to be 0.76 at 25°C.

While the products for the systems based on the TFSI-anion follow the Walden-rule stating that diffusion coefficients \(D\) and molar conductivities \(\Lambda\) of different liquids can be compared with the help of the well-known Stokes-Walden-rule stating that diffusion coefficients \(D\) and molar conductivities \(\Lambda\) in two different electrolytes or to a selected electrolyte at two different temperatures.

This equation is also often written as:

\[ D\eta = \text{const} \]  

[4]

Electrolyte compositions, viscosities, diffusion coefficients and Walden-products for all systems discussed above are shown in Table I. While the products for the systems based on the TFSI-anion follow the rule, the product for the tetrafluoroborate- electrolyte is two orders of magnitude lower than predicted by Eq. 4, indicating a higher association of Dy\(^{3+}\) by the BF\(_4^-\) and Cl\(^-\)-anions compared to the large, weekly coordinating TFSI- anion in phosphonium- and pyrrolidinium-based systems.

**Electrodeposition of Dy on Cu-coated Nd-Fe-B-samples.**—The temperature was set at 150°C for deposition times in the range of 1 h and 11 h to produce samples of varying Dy content \(\omega_{dy}\), calculated by Equation 5:

\[ \omega_{dy} = (\omega_s + m)/(s + m) \]  

[5]

where \(m\) represents the deposited mass of Dy and \(\omega_s\) and \(s\) are the initial dysprosium mass fraction in the sample and the sample weight, respectively. The layer thickness was calculated by assuming even distribution of the plated material and complete coating of a sample surface of 2 cm\(^2\).

Deposition was conducted in galvanostatic mode with a fixed current density of –0.5 mA cm\(^{-2}\). Previous attempts to reduce the deposition times by increasing the current density up to –2 mA cm\(^{-2}\) resulted in poorly adhesive, black deposits. At higher current densities, the electrolyte bath changed from a pale yellow to a dark yellow/brown color, as well. This change was accelerated with increasing Dy concentration. A possible explanation is higher residual moisture, the effect of residual moisture on the stability of ionic liquids has been described in the literature. All Dy-coated samples were cleaned from any ionic liquid residue by dry acetone under argon atmosphere. SEM micrographs of the as-deposited Dy on Au-quartz crystal are shown in Fig. 6a). In comparison to electroplated copper, the surface morphology of the electroplated Dy is slightly porous. The deposition of Dy was confirmed by EDX and XPS as shown in Fig. 6b) and Fig. 6c). EDX spectrum of the sample shows S, C, O and F besides Dy. XPS analysis after argon ion sputtering indicates that impurities

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**Table I. Electrolyte compositions, diffusion coefficients, viscosities and Walden-products for three different dysprosium electrolytes.**

| Electrolyte | Ionic liquid  | Diffusion coefficient \([\text{cm}^2 \text{s}^{-1}]\) | Viscosity \([\text{mPa s}]\) | Walden-product \([\text{cm}^2 \text{mPa}]\) |
|-------------|---------------|--------------------------------|-----------------------------|---------------------------------|
| 0.147 M Dy(TFSI)\(_3\) | BMP TFSI | \(2.01 \times 10^{-8}\) | 85\(^{33}\) | \(1.7 \times 10^{-6}\) |
| 0.075 M Dy(TFSI)\(_3\) | P\(_{2225}\) TFSI | \(2.0 \times 10^{-8}\) | 83\(^{33}\) | \(1.76 \times 10^{-6}\) |
| 0.1 M DyCl\(_3\) | EMIM BF\(_4\) | \(4.87 \times 10^{-10}\) | 219\(^{32}\) | \(1.06 \times 10^{-8}\) |
| 0.1 M DyCl\(_3\) | EMIM BF\(_4\) | \(7.76 \times 10^{-9}\) | \(\times 10^{-9}\) | \(\times 10^{-9}\) |

\(^{a}\)Diffusion coefficient predicted by the Walden-rule.
apart from oxygen are only superficial as their atomic fractions in the sample nearly disappear with increasing etching time, as shown in Table II. The shift of the Dy 4d5 peak from 152.3 eV from metallic Dy to around 157 eV and the constant concentration of oxygen in the XPS depth profile indicate dysprosium oxide/hydroxide formation which is ascribed to short exposure to air during transfer into the XPS-chamber.

**Table II. Composition analysis of electroplated coating by XPS after plating and after etching.**

| Elemental peak | Atomic concentration [%] |
|---------------|-------------------------|
|               | C1s | N1s | O1s | F1s | S2p | Dy4d5 | Au4f |
| As deposited  | 14.6| 2.2 | 59.3| 9.9 | 4.1 | 6.8   | 2.1  |
| After Ar-etching | 0.0 | 1.0 | 59.0| 2.9 | 0.1 | 8.4   | 11.0 |

**Figure 6.** a) SEM micrograph b) EDX analysis of electrodeposited Dy and c) XPS Analysis of electrodeposited Dy on Au-sputtered quartz crystal. Shift of the Dy 4d5 peak with increasing Ar⁺-etching time (top to bottom) from a 0.2 mol kg⁻¹ solution of Dy (TFSI)₃ in BMP TFSI under Ar-atmosphere at −0.5 mA cm⁻² for 2 h at 150°C.

Annealing and ageing process.— After the coating procedure, the samples were cleaned and stored inside a glove box under Ar-atmosphere. Prior to the annealing and ageing process all samples were wrapped in molybdenum foil to decrease volatilization of Dy and transferred into a quartz tube. All manipulations were made under Ar-atmosphere. After transfer, the quartz tube was evacuated (1 × 10⁻³ Pa) and the vacuum was not broken until the ageing procedure was completed. The samples were annealed at 900°C for 6 h with subsequent cooling to room temperature. A finishing ageing procedure at 400°C was applied for 2 h.

Magnetic measurements.— Fig. 7 shows the demagnetization curves of all samples. While the untreated sample and the samples with 0.55 and 0.62 wt% show good squareness, the samples with 0.72 as well as 0.91 wt% show a knee in the intrinsic curve. A square shaped demagnetization curve is important to minimize irreversible demagnetization caused by operation conditions such as reverse fields. This behavior can be explained by the limited diffusion of Dy into the base body, which results in a decreasing concentration of Ndₓ₋ₓDyₓFe₁₄B₁ with increasing depth and a non-uniform value for Hₓ throughout
Table III. Deposition times, mass of deposited Dy, coating thickness, current efficiencies, total Dy content and the change in coercivity for different samples.

| Deposition Time [s] | Deposited Dy [mg] | Coating thickness [μm] | Current Efficiency [%] | Total Dy content [wt%] | Coercivity [kA m^{-1}] | Coercivity Increase [%] |
|---------------------|-------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
| 4500                | 1.01              | 0.29                   | 79.9                   | 0.55                   | 1236                   | 10.3                   |
| 12000               | 2.47              | 0.80                   | 73.3                   | 0.62                   | 1273                   | 13.1                   |
| 21600               | 4.55              | 1.48                   | 75.1                   | 0.72                   | 1307                   | 16.4                   |
| 39600               | 8.7               | 2.5                    | 78.3                   | 0.91                   | 1350                   | 20.4                   |

After final heat-treatment an increase in coercivity with increasing total amount of Dy is found similar to samples based on PVD coatings indicating a maximum achievable gain of about 600 kA m\(^{-1}\) for the magnet geometries used in this investigation. This limitation can be explained by the limited diffusion length of the Dy into the material, thus creating a high anisotropy Nd\(_2\)\(_x\)Dy\(_x\)Fe\(_{14}\)B\(_1\) shell wrapping the grains leading to suppression of reverse domain nucleation processes. With increasing diffusion depth, the concentration of Dy diminishes creating less such grains, thus the coercivity in the bulk remains unchanged. To explore limits further, thicker electrodeposited layers would be necessary. Deposition of thicker layers requires further process optimization.

**Conclusions**

In this study, we present the development of a novel electrochemical deposition process of the heavy rare earth element dysprosium on sintered Nd-Fe-B permanent magnets in order to enhance their permanent magnetic properties. A coating thickness of up to 2.5 μm dysprosium was achieved by galvanostatic deposition. XPS analysis after argon ion sputtering indicates that minor impurities are only superficial as their atomic fractions in the sample nearly disappear with increasing etching time. After a conventional heat-treatment at 900°C and 400°C inducing grain boundary diffusion of Dy, the coercivities increased up to 20.4%. Electrochemical deposition of Dy has the potential to be superior to coatings based on PVD processes in terms of versatility, speed, and investment costs.

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