

D6.5

15 PhDs awarded

Public Date: 30.10.2019





PhD status of DEMETER fellows at the end of the project:

- Three fellows obtained their PhD within their funded DEMETER period. Find details in section "Doctoral degrees awarded".
- Six fellows received an extension of contract where:
 - Three fellows have already obtained their PhD before the DEMETER Final reporting date (31st October 2019). Find details in section "Doctoral degrees awarded".
 - Three fellows have the defence date already set within a few months after the end of the project.
 - ESR10: Adolfo Garcia Gonzalez defense date on 14th November 2019
 - ESR2: Muhammad Awais defense date around November 2019
 - ESR1: Anas Eldosouky defense date around January 2020
- Funding for PhD of four fellows ended with the end of their contract with DEMETER. Three have finished the required research work and are in the dissertation-writing phase.
- The doctoral programme of two fellows was discontinued (Project Officer was informed), nevertheless DEMETER continued providing research supervision and high level trainings till the end of their contract.

Doctoral degrees awarded (before 31st October 2019)

A doctoral degree has been awarded to the following six DEMETER fellows by 31.10.2019 (in chronological order):



ESR11: Pranshu Upadhayay (VALEO/G2Elab)

PhD Thesis: "Electrical machine designs based on 3D flux paths with reuse & recycle magnet concepts for automotive applications". PhD awarded on December 11th, 2018. Abstract

The research work presented in this thesis aims at developing a permanent magnet based clawpole machine for automotive application with permanent magnet reuse and recycle concept. The aforesaid research is under the aegis of Project DEMETER which is in the framework of European Union's Horizon 2020 Marie Sklodowska-Curie actions. The project focuses on the recovery of rare earth permanent magnets utilized in automotive applications due to the prevailing problems of price fluctuations and supply-demand issues of these permanent magnets. The claw-pole machine is employed in almost all of the automobiles in the world for alternator application. With the increase in power demands, the claw-pole machine is also being developed as a motor-generator utilized in the hybrid electric vehicles. At present the permanent magnet based claw-pole machine is being used in mild hybrid electric vehicles for energy savings. The literature is replete with various configurations of claw-pole machines that can be developed to achieve better performances. However, easy assembly and disassembly of various parts of the machine is also important for the reuse and recycle of magnets. In this research work two concepts have been developed; first, the direct reuse concept i.e. easy assembly/disassembly of the rotor and magnets, so as to easily take out the magnets for direct reuse or recycle and; second, the direct recycle concept i.e. utilization of recycled magnets in the machine to achieve the desired performance. In the course of this research the base design of the claw-pole machine was developed, analyzed and optimized so as to attain best torque versus magnet-weight ratio. This helped in the reduction of magnet cost for almost the same torque. The optimization was carried out using 3-D numerical analysis. The optimized model was developed in a way that the assembly process of the magnets and claw-poles remained the same. However, during disassembly the magnets can easily be withdrawn without disassembling the complete rotor; therefore utilizing these magnets for direct reuse in other applications or sent for recycling. In the direct recycle concept, the magnets used in the machine are recycled magnets with deteriorated performance. The type of recycling process is a strong determinant of the deterioration in performance of these recycled magnets. The aim of the direct recycle



concept was to analyze the machine with virgin and recycled magnets, and evaluate the energy consumption of the machine under different drive cycles. It was observed that with utilization of recycled magnets in the claw-pole machine, the energy consumption was almost same as that of the machine with virgin magnets. Thus it can be concluded that for the permanent magnet based claw-pole machine, the utilization of recycled magnets is more sustainable for the environment as it can lead to consequential limits on the mining of rare earth materials. The price fluctuations and supply-demand problems can also be reduced with the increase in utilization of recycled magnets have been developed and tested for performance. It has been observed that the experimental results match fairly well with the design analysis results, hence validating the design process and methodology. Available: http://theses.fr/2018GREAT103

You may find the thesis here: <u>https://tel.archives-ouvertes.fr/tel-02130959/document</u>





ESR9: Amit Jha Kumar (G2Elab)

PhD Thesis "Design of Halbach Permanent Magnet External Rotor Machine with Reuse & Recycle Magnet Concepts for Automotive Applications ". PhD awarded on January 28th, 2019.

Abstract

Abstract : Electric vehicles (EVs) or Hybrid electric vehicles (HEVs) offer many advantages over the conventional IC engine vehicles. According to recent trends, the demand for efficient (H)EVs is expected to grow significantly. For a high-power range, permanent magnet based motor technology has been the preferred choice for motors deployed in (H)EVs. Growing demand of highly efficient motors is in direct correlation to the demand of strong magnets (NdFeB or SmCo), which uses rare earth elements (REE). The availability and supply of REEs specially heavy REEs is very critical. Therefore, the aim of this doctoral thesis is to design an outer rotor Halbach motor for a (H)EV application with easy recycling and reuse of the magnet. Further, the project aims to investigate and propose the manufacturing of a Halbach magnet used in a high power motor EV applications. Firstly, the manufacturing of Halbach magnet using a sintered and a bonded NdFeB magnet was investigated. The study shows that the manufacturing of Halbach array using a bonded magnet is much easier and more cost effective than the sintered magnet. The characterisation of a bonded NdFeB magnet used for manufacturing a Halbach magnet was also performed. Various recycling routes for both sintered and bonded magnets were analysed and it can be inferred that bonded magnets are much easier to recycle in a cost effective and environment friendly manner. The thesis also proposes the recycling route for the bonded magnet used in the motor. Secondly, a motor with bonded Halbach magnet was designed using 2D and 3D FEM. To achieve a highly efficient and compact motor, fractional slot tooth coil winding was used. The properties of Halbach magnet was calculated using FEM model and benchmarked against the analytical model. The results obtained from the two approaches were in close agreement. Further, the impact of slot pole combinations on motor losses and the subsequent torque were investigated, specifically eddy loss (considering all the design constraints). Different strategies to use recycled magnet with lower remanence is also presented. It is shown that using a recycled magnet with increased axial length of the motor could be the best choice considering different factors, specially manufacturing of the Halbach



magnet. Based on different parametric studies a design of the motor was proposed and prototype was built. It was demonstrated that a high power Halbach magnet could be built economically using a bonded NdFeB magnet. The airgap flux density of the rotor, measured on the prototype is in close agreement with the calculated values. Additionally, WIRE (Weighted Index of Recycling and Energy) methodology was presented to benchmark different motor designs on the basis of performance and recyclability. The method developed produces two indices based on: • Ease of motor recyclability considering material, assembly and disassembly of magnets. • Impact of a recycled magnet on the energy consumption of a motor during its operational lifetime. Using both the above indices, one can easily analyse the pros and cons of different motor designs on the basis of recyclability and energy efficiency. The proposed motor design was evaluated using the developed method and it is shown that the motor is easy to assemble and disassemble. In addition, the motor assembly (glue free) enables easy magnet extraction and direct reuse. The evaluated energy index of the motor shows the impact of using a recycled magnet and its viability for EV applications in different scenarios. Finally, a motor prototype was built and measurements were done. The measured results are in good agreement with the calculated values. The assembly and disassembly of the motor were done manually using standard tools with ease.







ESR12: Ziwei Li (VALEO/G2Elab)

PhD Thesis: "Electrical radial flux machine design focusing on magnet recycling and reuse: Application to hybrid or electric vehicles". PhD awarded on May 2nd, 2019.

Abstract

Nowadays it is imperative to reduce the CO2 emission of automotives due to the climate changes. One of the essential strategies is to use new energy vehicles, such as Hybrid and pure Electrical Vehicles ((H)EVs). However, no matter what the energy storage devices (H)EVs have, they always need electrical machines to transfer electrical energy into mechanical energy. Permanent Magnet (PM) electrical machines seem to be the best candidates for (H)EV applications in terms of their outstanding performances. However, the supply and cost of PMs are essential for PM machines. The strongest rare earth PM is Neodymium-Iron-Boron (Nd2Fe14B) type magnet, or simply written as NdFeB. Commonly, in order to improve the temperature stability as well as resistant demagnetization of magnets, small portion of heavy rare earth element, Dysprosium (Dy) or Terbium (Tb), is added to the alloy. However, with a high demand of high grade NdFeB magnets, the supplies of these rare earth elements, including Neodymium (Nd), face serious challenge, especially for Europe. In this case, one of the possible solutions for Europe to tackle the rare earth supply risks is to recycle rare earth magnets. Demeter -European Training Network for the Design and Recycling of Rare-Earth Permanent Magnet Motors and Generators in Hybrid and Full Electric Vehicles, is an Europe Union registered project. DEMETER envisaged three routes for the recovery of rare earth PM from these devices, which are so called direct re-use, direct recycling and indirect recycling. Valeo and G2Elab are the principal partners in this project, and they mainly focus on the route of PM direct re-use. This doctor thesis is supervised by Valeo and G2Elab, and mainly focuses on radial flux type PM electrical machines, which are the most widely used type of electrical machines nowadays. The applications include Mild Hybrid Electric Vehicles (MHEV) or small Electric Vehicles (EV). The new motor design not only needs to be recycle friendly for PMs, but also needs to meet all the strict requirements for the applications. With thorough literature studies, FEM optimization and thermal/mechanical analysis, it was found that an IPMSM design can fairly fulfill all the requirements and constraints. Then new magnet materials and assembly methods



were implemented for the magnet recycling - a kind of bonded magnet was used for the IPMSM. This bonded magnet was made from a Hydrogen Decrepitation Deabsorbation Recombination (HDDR) anisotropic NdFeB magnet powder, with Sulfide (PPS) binder. It has the possibility to directly assemble the magnet into the rotor by injection molding. Thus the assembly of the magnets would not be constraint by their shapes. The disassembly of the magnets became easy as well it is possible to heated up the rotors so that the bonded magnets can be melted down for extraction. Then they can be mixed with a certain percent of virgin magnets compound to make new bonded magnets without remarkable changes on performances. In summary, the entire recycling process is relatively easy and ecologically sustainable. Thus, based on this new concept, an IPMSM with bonded NdFeB magnets were fabricated. Series benchmark tests were carried out, for instance measurements of back-EMF, torque, efficiency, short circuit current and stator temperatures. In this thesis, apart from new design ideas of electrical machines, another goal is to evaluate e-machines with respect to the recyclability. The recyclability is quantified by two indexes, together they can be named Weighted Index of Recycling and Energy (WIRE). By using WIRE, the recyclability between different machines can be comparable, even with different dimension or performances. It was found that by using WIRE to evaluate the new designed PM machine, promising results can be obtained. The magnet reuse and recycling approach can gain environment benefit without economic losses.

For more info: <u>http://www.theses.fr/s160930</u>





ESR3: Martina Orefice (KU Leuven)

PhD Thesis: "Solvometallurgical recovery of metal values of Sm-Co and Ne-Fe-B permanent magnets". PhD awarded on June 21st, 2019.

<u>Abstract</u>

Two main features of (hybrid) electric vehicles are sensitive to a sustainable end-of-life treatment: the battery and the traction motor. Compared to the large extent of research about battery recycling, studies on the sustainability of electric traction motors had so far a marginal attention. However, the most efficient and convenient of these motors rely on rare-earth permanent magnets and are, together with wind turbines, the main cause of depletion of the critical rare-earth elements. Recycling of end-of-life permanent magnets from electric motors will be a valid support to keep the production and demand of rare earths in balance. Furthermore, rare-earth permanent magnets are also an interesting secondary source of cobalt, primarily extracted in a critical geopolitical context.

This PhD thesis explores innovative recycling routes of Sm–Co and Nd–Fe–B permanent magnets, based on solvometallurgy, which is an emerging branch of extractive metallurgy. Although the concept of solvometallurgy itself is not new, there exist only few examples of solvometallurgy applied to urban mining. Since most electric motors contain sintered Nd–Fe–B permanent magnets, research in this PhD thesis was mainly focused on this type of magnets.

First of all, the microstructure of Nd–Fe–B permanent magnets was investigated. Microscopy and image analysis revealed a reactivity order of the three phases of the Nd–Fe–B permanent magnets different from what reported in the literature. An ionic liquid comprising a concentrated strong acid was used as etchant. Afterwards, a leaching process based solely on water-free ionic liquids revealed limits of solvometallurgy when water is removed from the system. Possible causes and solutions have been investigated in detail and it was found out that the leaching efficiency was also effected by the formation in the roasting preliminary step of the ternary oxide NdFeO₃ instead of the expected binary oxide Nd₂O₃.

Controlling the roasting atmosphere allowed to obtain the desired Nd_2O_3 phase and the leaching yields were increased. In this way, a combined pyrometallurgical–solvometallurgical recycling route was developed. Finally, two flowsheets were designed, with the aim to use solely



solvometallurgical process steps, one flowsheet for the recycling of Sm–Co magnets and another one for the recycling of Nd–Fe–B magnets.

Another limit of solvometallurgy became evident: precipitation of the metals of interest did not occur in the solvent used. However, a step of precipitative stripping helped to save one operation unit and to mitigate the disadvantage. Solvometallurgy, in fact, resulted in improved leaching and solvent-extraction processes: the processing time can be reduced, the temperature can be increased above 100 °C, without the need of using an autoclave, and differences in solvation of the ions occur, affecting the equilibria in solution.

In conclusion, solvometallurgy can be a valid technology for the recycling of rare-earth permanent magnets, but the replacement of the aqueous phase in hydrometallurgy by an organic phase in solvometallurgy is not trivial. In particular, attention needs to be paid to the choice of the solvents and to the way of recovering the metals from the solvent phases.

You may find the thesis here: <u>https://limo.libis.be/primo-</u>

explore/fulldisplay?docid=LIRIAS2806167&context=L&vid=Lirias&lang=en_US&search_scop e=Lirias&adaptor=Local%20Search%20Engine&tab=default_tab&query=any,contains,Solvomet allurgical%20recovery%20of%20metal%20values%20of





ESR7 – Awais Ikram (Institut Jozef Stefan)

PhD Thesis: "Reprocessing of recycled Nd-Fe-B and Sm-Co-based magnets with contemporary sintering technique". PhD awarded on June 28th, 2019.

<u>Abstract</u>

The rare-earth-based permanent magnets (REPM) are vital for modern electrical appliances and green energy technologies which has created a massive supply-demand frontier for the industries. Usually high thermal stability and resistance to demagnetization comes with the addition of heavy rare earth elements (HREEs) like Dy and Tb in Nd2Fe14B-type permanent magnets, which has the highest energy products (BHmax) per unit volume. The addition of HREEs to Nd-Fe-B permanent magnets has strong implications on natural abundance, costs as well as the reduction in magnetization (and BHmax) due to the antiferromagnetic coupling of the magnetic moments of the HREE atoms with that of iron. Since the 2011 rare earth (RE) crisis, which caused the prices of raw material (especially Nd, Dy, Tb) to surge by 700%; the global REPM producers are looking for supply chain and economic sustainability to fulfill this commercial demand. On the contrary, the scientific front has been looking to explore a new class of permanent magnets without the costly rare earth elements or try to develop the methods to recycle the end-of-life (EOL) REPMs. The former has not been very successful since the past decade or so, whereby the later route has shown high potential in commercial up-scaling if the processing parameters are controlled to preserve and control the microstructure. Direct reusage or direct/indirect recycling of EOL magnets is an area of interest for the researchers as well as the magnet producers. Slight modification of chemical composition towards rare earth rich compositions has helped developed REPMs with better magnetic properties than the starting EOLs, but the higher costs due to the addition of rare earth rich compositions to the existing systems may be a problem.

The hydrogen-based recycling route of REPM is an economically feasible and clean method due to its very low environmental footprint compared to pyro- and hydrometallurgical recycling methods. Normally the rejected waste in REPM production goes up to 25%, which is huge considering the aggregate global magnet production. This rejected scrap or EOL waste magnets can be processed by hydrogen-based recycling methods like: hydrogen decrepitation (HD) used



for Nd-Fe-B and Sm-Co or hydrogenation–disproportionation–desorption–recombination (HDDR) used for Nd-Fe-B to develop magnetic powder can either be sintered by conventional powder metallurgical routes or readymade to assorted shapes in the form of plastic bonded magnets. The HD treatment facilitates the intergranular cracking in EOL Nd-Fe-B magnets due to formation of NdHX (causing volume expansion) and this friable demagnetized powder can be further jetmilled to $3 - _5 \mu m$ size, magnetically aligned and sintered to final shape. The chemical composition can be easily modified by blending fine RE-rich alloy powder/dopant (DyF3) with the HD-treated recycled powder to achieve the desirable level of magnetic properties.

Identically the HDDR reprocessing has the additional benefit since it can develop anisotropic nanocrystalline grains in the size range of ~400 nm for achieving higher coercivities than the microcrystalline counterparts. The HDDR-treated recycled powders can be formed to shape by mixing them with the polymeric binders or sintered by rapid consolidation techniques like hot deformation or spark plasma sintering (SPS) to bulk magnets. Normally these nanocrystalline powders cannot be conventionally sintered, as higher temperatures for prolonged periods would result in exaggerated grain coarsening and the coercivity will decline severely. Therefore, rapid compaction by SPS is an alternate method in magnet recycling to achieve the magnetic properties at par with the commercial grade or fresh HD/HDDR powder. The SPS process is very rapid and heating rates can be controlled to prevent the deterioration to the microstructure, which in turn preserves the magnetic properties of the original powder and fully dense magnets can be formed in a matter of minutes at lower sintering temperatures than those in conventional sintering. Simultaneous application of pressure in the SPS is beneficial for achieving full densification and ramping it rapidly (> 100 MPa) and is beneficial in the texture in the Nd-Fe-B nanocrystalline material by hot forging (deformation).

Within this thesis two different kinds of recycled magnetic powders HD-treated SmCo5 and HDDR Nd-Fe-B, were investigated by consolidation with the SPS. In the former case the SPS temperature range was identified around 900 – _950 °C with holding time of 1 – _5 minutes and 100 MPa pressure in 0.2 mbar Ar pressure for achieving > 97 % densification. The resultant magnetic properties of isotropic SPS-ed SmCo5 magnets (HCi > 2000 kA/m & Br = 0.47 T) were slightly better than the vacuum-sintered magnets due to better microstructural control (optimal distribution of Sm-rich Sm2Co7 phase in the SmCo5 matrix) achieved with the SPS. As compared to the commercial grade material, the finer HD-treated recycled powder had a tighter grain size



control (4 - _20 μ m), which resulted in nearly full density and room temperature HCi > 2000 kA/m and better thermal stability at 180 °C.

Similarly, the recycled HDDR Nd-Fe-B powder was optimally SPS-ed at 750 – _800 °C (approx. 30 % lower sintering temperature) for 1 minute and 100 MPa to reach HCi ~ 1200 kA/m which was slightly better than the fresh HDDR powder MF-15P (1180 kA/m). The starting HDDR powder with Nd13.4Dy0.6Fe78.6B6.1Nb0.4Al0.7 composition had the HCi = 830 kA/m. The effect of post SPS thermal treatments was thoroughly investigated for the recycled HDDR powder and with HR-SEM and EDXS it was confirmed the redistribution of Nd-rich grain boundary phase is important for achieving higher coercivities after the SPS fabrication. These optimally SPS-ed magnets were hot deformed at 750 °C by varying the hot deformation pressure to 100 and 150 MPa in WC + Co cermet dies to induce texture in the isotropic bulk magnets. As compared to the starting HDDR powder with Br = 0.9 T, the resultant improvement after 150 MPa hot deformation in Br was ~ 1.01 T (BHmax = 180 kJ/m3) with 55% height reduction of the sample.

It is important to consider that hydrogen-based recycling is effective only if the protective coating on EOL magnets is non present or ruptured for decrepitation to occur. In secondary recycling, these coatings are removed from the fragmented powder with an average particle size less than 100 μ m by mechanically crushing the magnets. On the contrary, the HDDR powder is prone to excessive oxidation if the reduction in average particle size is too significant. But this critical particle size range has not been identified previously, which also restrained the commercial scale up. The in-depth studies were performed to verify the relationship of HDDR powder particle size with the oxygen content, sinterability and the magnetic properties. The particle fractions from 700 μ m to < 50 μ m were individually tested for the oxygen content, the magnetic properties and the sinterability. It was evaluated that particles sized below 100 µm took up twice the oxygen (> 7000 ppm) and as a result, the overall Nd-rich phase declined due to excessive Nd2O3 formation. This resulted in poor sinterability and severe degradation of the magnetic properties (HCi < 200 kA/m). Therefore, it was suggested to limit sizing down the HDDR powder particles not less than the 100 µm range for achieving optimal magnetic properties. Lastly the improvement in HCi of the recycled HDDR Nd-Fe-B powder was realized by blending it with DyF3 in varying weight fractions from 1 - 5 %. The blends were compacted at 750 °C for 1 minute and 100 MPa. These magnets were subjected to vacuum thermal treatments at 600 -





DyF3 resulted in HCi ~ 1410 kA/m which is approx. 70% higher than the starting HDDR powder. This doping technique developed uniform a (Dy,Nd)2Fe14B core-shell structure throughout the sample due to localized Dy diffusion and solution reprecipitation upon solidification below the ternary eutectic temperature 665 °C. Excessive DyF3 $c_0_n_t_e_n_t \ge 5$ wt.%, prolonged thermal treatment (\ge 3 hours) and higher temperatures (900 °C) deteriorated the magnetic properties due to the decomposition of core-shell structure and formation of complex Nd-Fe-F-type intermetallic phases besides the excess of unreacted, non-ferromagnetic DyF4 & NdF4 phases.



ESR15: Gwendolyn Bailey – (KU Leuven)

"Life Cycle Assessment of new recycling and reuse routes for Rare Earth Element machines in hybrid/electric vehicles" on 30th October 2019.

<u>Abstract</u>

The rare earths contained in permanent magnet motors symbolize the building blocks of our technological world. However, the rare earth technologies which are essential for the transition towards sustainable mobility, contain critical metals with the highest supply risk for Europe.



Furthermore, these crucial metals come with some environmental challenges: the process of extracting and separating them from one another often has serious health and environmental impacts. Europe needs to look for solutions that ensure its industry can remain competitive in areas like electric vehicles. Of course, recycling is an obvious strategy and thus the DEMETER project was formed to develop innovative recycling and reuse technologies. My role in the project was to provide the environmental and economic perspective on the recycling/reuse technologies being developed within the project by adopting the life cycle approach.

As the project evolved, and the technologies being developed began to take shape, it became clear during the modelling for these assessments that some of the recycling processes were not automatically cheaper or better for the environment than the primary production route. It also became clear, after the work performed in conjunction with the Joint Research Center, that in order to provide a long-term meaningful benefit in terms of climate protection for sustainable mobility, additional solutions need to be sought such as decarbonizing the grid mix as well as recycling critical raw materials at a large scale.

You may read the full text here: <u>https://limo.libis.be/primo-</u>

explore/fulldisplay?docid=LIRIAS2853151&context=L&vid=Lirias&search_scope=Lirias&tab=defa ult_tab&lang=en_US&fromSitemap=1



