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Abstract: Nowadays, the circular economy is gaining more and more attention in sectors where the raw material supply is critical for both cost and geo-political reasons. Moreover, the environmental impact issue calls for recycling. From this perspective, the recovery of rare earth elements represents a strategic point. On the other hand, the high cost and the dangerous standard recovery methods that apply to NdFeB magnets limits options for traditional recycling. A new mechanical method is proposed, not requiring hydrogen, high temperature, or chemical processes, but instead using an impact mill designed to operate in vacuum. A traditional impact mill operating in a glove box filled with Ar atmosphere has also been used for comparison. The obtained NdFeB powders were analyzed in terms of magnetic properties and chemical composition, particularly in terms of the oxygen content.

Keywords: recycling; NdFeB powder; NdFeB magnets; powder metallurgy; mechanical process; impact mill; anisotropy; oxidation; magnetic characteristic; circular economy

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1. Introduction

Decarbonization, reduction of greenhouse gas emissions, and improvement in overall health are the main aims in several countries where some actions concerning environmental policies have been taken. The most popular way of reducing air pollution consists of introducing electric or hybrid vehicles (EV or HEV) [1–3]. Different strategies have been proposed and studied regarding this topic, and some tools like life cycle assessment (LCA) are used to analyze the product or processes [4–6]. LCA consists of a Life Cycle Inventory (LCI) analysis and Life Cycle Impact Assessment (LCIA), where LCI involves the compilation and quantification of inputs and outputs for a product throughout its life cycle. The adoption of LCA permits understanding and evaluating the magnitude and implication of the potential environmental impacts by considering all the steps related to the product's life, from the design to its re-use [7]. Therefore, recycling plays an important role in ecological aspects and economic savings. In some industrial sectors recycling is essential, while in other sectors it shows high potential [8], also resolving the problems of raw material supply [9,10].

The introduction of EV or HEV led to the necessity of evaluating new material solutions for battery systems and electrical machines. Nowadays, different types of electric motors are used; one is the permanent magnet synchronous machine (PMSM), which shows high torque density and good dynamic behavior [11–13]. However, the PMSM contains

Rare Earth elements, which significantly affects the cost of the device [14]. The high cost depends on various aspects: the extraction process is expensive and the price depends on the market and geopolitical situations, as Rare Earth elements are located in a few areas of the planet [15–19]. The Neodymium price variation changed significantly in the last decade: 30 $/kg in 2009, then 300 $/kg in 2011. After that, the price decreased to 60 $/kg in 2018 then increased again until 220 $/kg at the end of 2022, reaching a critical point. The same situation involves the Dysprosium powder used to produce the best NdFeB grade magnets, where the price was 750 $/kg in 2022. Some solutions are proposed to solve this issue [15]: opening new mines, researching new magnetic materials [20–22], designing appliances without NdFeB magnets [23], and recycling [24–28]. All listed points have aspects open to criticism: opening new mines faces ecological permission problems in many countries, while new magnetic research is generally based on alloys that are not cost-effective, the use of ferrite magnets (as substitutions for NdFeB) increase volume and weight with an overall reduction of performances, making them not applicable to many industrial sectors.

NdFeB recycling is based on existing recovery techniques [29–37]. The hydrometallurgical method is a chemical process in which the material is crushed and leached; its extraction is obtained through particular solvents. This technique is expensive and complex. Another is based on the pyrometallurgical method, which consists of recovering REO (Rare Earth Oxides) at high temperatures (1500 °C). The obtained REO need to be processed again via the typical extraction process of rare earth, which involves transporting REO in a few areas of the planet. The more popular NdFeB recycling technique is hydrogen decrepitation (HD), where the material is crushed by means of the adsorption of hydrogen. There are various dangerous aspects of the process, due to the use of hydrogen and very highly reactive NdFeB powder; for this reason, this method is also considered rather expensive. Moreover, the recycled product should be reused immediately without going through several production chain stages.

This paper analyzes an alternative method for producing NdFeB recycled powder, based on a cheaper mechanical process, without the use of hydrogen. The focus is to develop a more suitable, cheaper, and safer recycling process for industrial criteria. The goal of the research is to develop a reliable process able to guarantee the production of powders ready to be re-used, without changing the chemistry of the starting material. A vacuum impact mill has been designed and used to produce NdFeB powders. The device is a prototype of an attritor capable of working in a vacuum or controlled atmosphere, and it was designed and produced by the authors in Politecnico di Torino. The mill is composed of two chambers, both containing a controlled atmosphere during the operational phase. All of the grinding processes in this work were conducted within a vacuum. The proposed mechanical grinding process is a technique already used for the fracture, surface finish and coating of the powder, continuously. In the proposed work, the process is non-continuous, so when all the magnets are ground and reduced to a powder, the mill can be disassembled and loaded again with new material. Moreover, the activity does not require chemical preparations and does not result in high costs.

For comparison, a conventional impact mill was used, placing it in an inert environment in a glove box filled with Argon gas. NdFeB powders obtained in different conditions have been characterized and tested.

2. Materials and Methods

Hard disks used in computers in the last 15 years have been collected by RISTA s.r.l., an Italian Company working in the field of waste recovery and environmental sustainability. Magnets have been disassembled from the supports through an automatic precision system developed by OSAI Automation System, a leading Italian Company in the field of automation and LASER for industrial processes. The position of the magnets is shown in Figure 1. The variety of the magnets appears in several dimensions (Figure 2) related to different magnetic properties. Diversification of magnets was not implemented, and also the nickel protection coating was not removed [38].

Figure 1. Hard disk drive with the magnet position.

Figure 2. Different sizes of the recovered NdFeB magnets (some high-mu shields on the left).

Magnets were demagnetized before the crushing process. This was performed at 350 °C in a vacuum to avoid oxidation.

The crushing and grinding processes were performed using an impact mill (Figure 3). The scheme of its advanced version, that works in vacuum, is reported in Figure 4. Two types of impact mill have been evaluated. The first mill is made by a high-speed DC electric motor and a chamber with blades, which is filled with the recovered magnets to be chopped and transformed into recycled powder. The equipment has been placed in a glove box of approx. 0.5 m^3 (Figure 5a,b). Before starting the milling process, an Ar purging of 15 min has been carried out, and Ar backfilling has been adopted throughout the whole process.

The second mill consists of a vacuum impact system designed for this specific purpose (Figure 6a,b). It is made up of different parts. One part consists of a chamber with a high-speed DC motor, electrically and mechanically sealed off from the (second) chamber where the powder is collected, which is then separated by a mechanical grid from a glass storage container for the recycled NdFeB powder. A sealing gasket was used to retain vacuum.

Powders' granulometry and distribution size have been analyzed with the Malvern Mastersizer 3000, equipped with Aero S for dry dispersion.

The obtained powders were analyzed using X-ray diffraction (XRD) analysis (Bruker New D8 ADVANCE ECO diffractometer equipped with a Cu Kα [1.5406 Å] radiation source) and compared to the NdFeB reference standard (PDF 00-036-1296).

Figure 3. Proposed mechanical technique: impact mill.

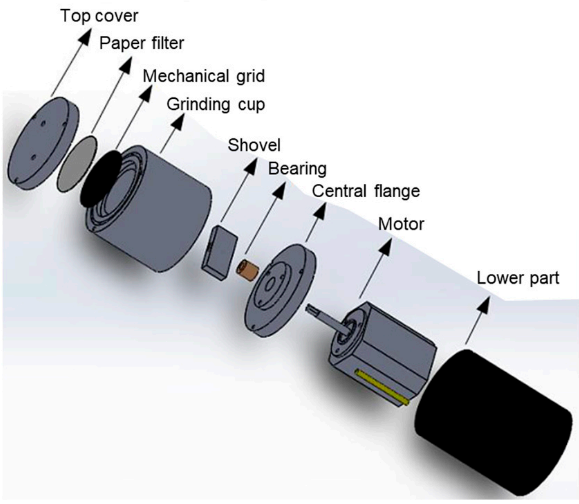
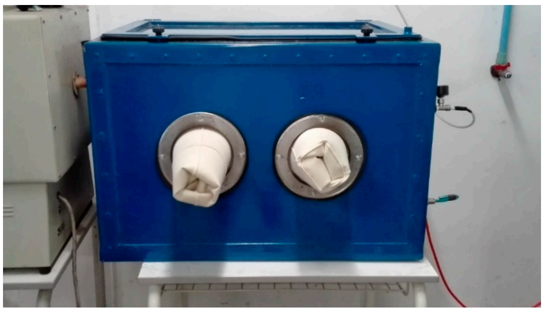
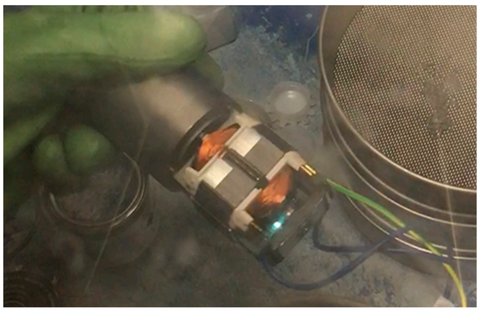


Figure 4. Vacuum impact mill scheme.



(**a**)



(**b**)

Figure 5. Impact mill in glove-box and the washing in Argon: exterior aspect of the glove box (**a**) and the impact mill used with this configuration (**b**).

Furthermore, powders were examined by means of X-ray fluorescence (XRD) analysis (Thermo Scientific EDX-7000) to evaluate the amount of neodymium and iron. The oxygen content (wt. %) of the two set of powders was obtained by means of an elemental analyzer (LECO ONH836).

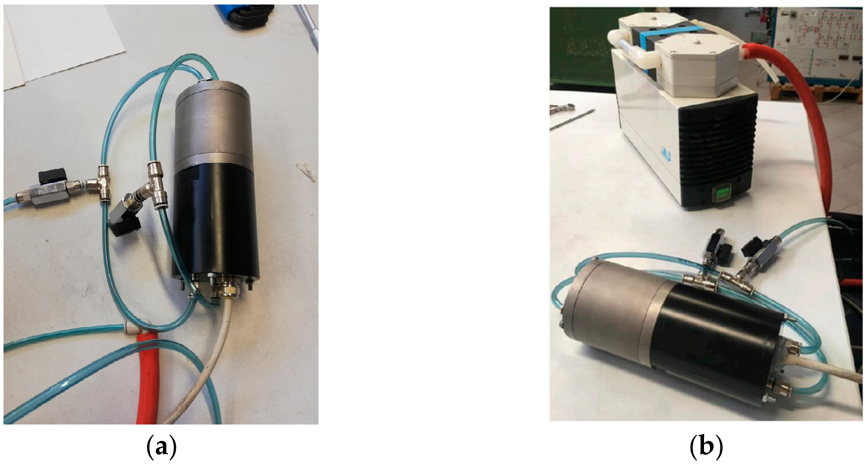


Figure 6. Vacuum impact mill: mill (**a**) and system connection with the pump (**b**).

The powders' magnetic performance has been evaluated by means of a Permanent Magnet Hysteresisgraph (Laboratorio Elettrofisico I-200 A/S, equipped with Powder testing fixture).

All produced powders were stored in vacuum glass bottles. This operation is mandatory to avoid powder oxidation and to facilitate transport. Figure 7 illustrates the scheme from the recovery of NdFeB magnets from hard disks to powder characterization.

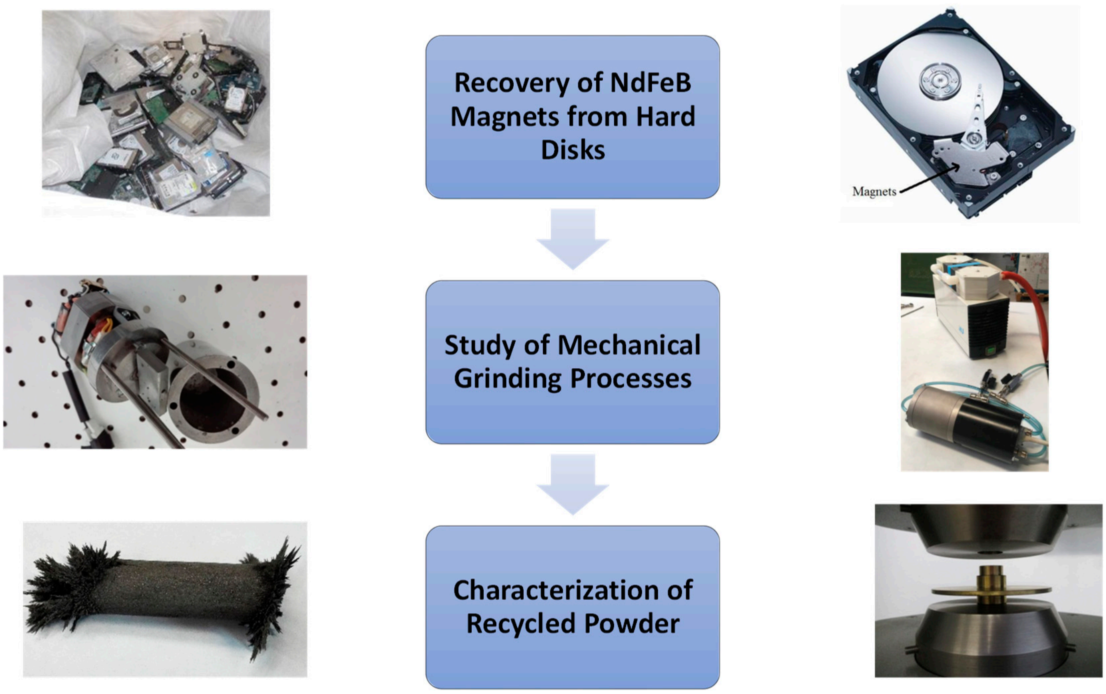


Figure 7. The scheme represents different steps of the proposed technique: supply of disused hard disks, recovery of NdFeB magnets, crushing with two different impact mills, and recycled powder characterization.

3. Experimental Results

After the milling process, powders have been sieved between 50 µm and 500 µm. Their distribution size is reported in Figure 8.

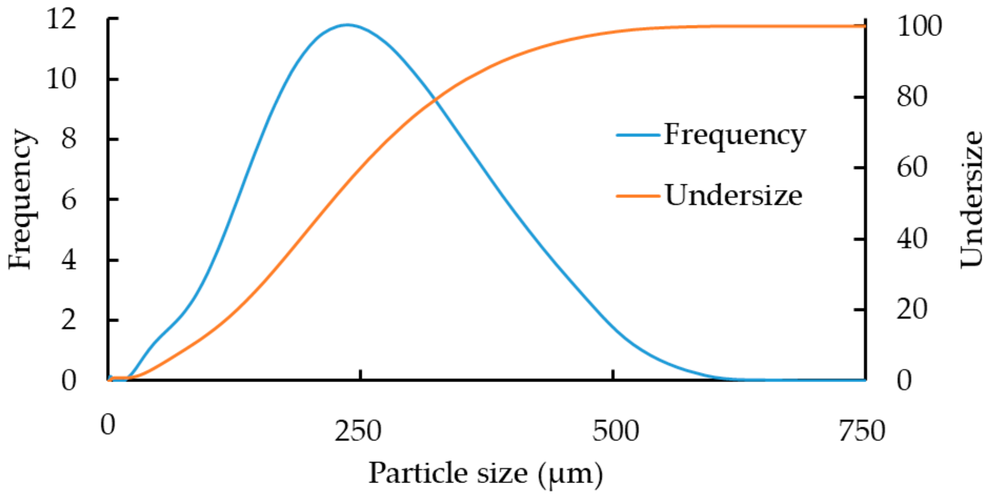


Figure 8. Distribution size of milled powders.

3.1. Chemical and Microstructural Analyses

The presence of oxygen has been investigated through elemental analysis for both impact mill processes. Results are reported in Table 1, where the operation with a vacuum impact mill highlights an oxygen content highly reduced in comparison to the process in the Argon-washed glove box [39].

Table 1. Elementary analyses (LECO-ONH) as a function of different processes with impact mills.

Type of Impact Mill Operation	Oxygen Content wt.%
Operation in a glove box (Argon)	1.59
Operation in Vacuum Impact Mill	0.375

In order to preserve the magnetic properties of the material, the value of oxygen should be minimized; in fact, the obtained value is similar to a general indication of 0.4%, as reported in the literature [40–42]. The result of the vacuum impact mill confirms two things: firstly, the process does not add further oxidation; secondly, the recycled powder can be used immediately and not stored like an REO.

Considering the oxygen contents of the two powders and the magnetic properties of the different systems (ref par 3.2), only results relative to vacuum-processed powders will be reported. Several batches of materials were used that were derived from magnets recovered from landfills. The analyzed lots were found to be free of Dy and Pr. As reported later in the text, this does not affect the basic magnetic characteristics.

Table 2 shows the chemical composition of the powder obtained in vacuum evaluated by XRF.

Table 2. XRF composition of recycled powder in vacuum impact mill.

Elements	Composition %
Fe	63.394
Nd	28.514
Ni	5.653
V	1.053
La	0.987
Cu	0.182
Ca	0.087
Nb	0.057
Zr	0.039
Os	0.020
Mo	0.013

It is possible to note that Iron and Neodymium are about 63.4% and 28.5%, respectively, which are typical amounts for NdFeB recycled materials [43]. These values correspond to medium Rare Earth element scraps. Additionally, the presence of Nickel is high due to the protective coating, which was not removed before the crushing and grinding process.

The recycled powder was tested through the XRD; the obtained spectrum is shown in Figure 9, blue line. The cell results are as follows: space group is P42/mnm, lattice parameters are a = 8.7865 Å, c = 12.1822 Å. The crystallite size was 96 nm. For verification, the NdFeB standard lattice parameters (PDF 00-036-1296, measured as bulk sample) are reported: space group is P42/mnm, a = 8.7933 Å and c = 12.1799 Å. The experimental spectrum shows the errors with respect to standard lattice parameters of 0.0773% for a and 0.01888% for c. This comparison confirms the similarity of the recycled powder to an NdFeB permanent magnet, evidencing the lack of oxidation effect [44]. Therefore, the magnetic phase is maintained. The comparison of the recycled powder spectrum and NdFeB reference is reported in Figure 9, blue and red lines, respectively.

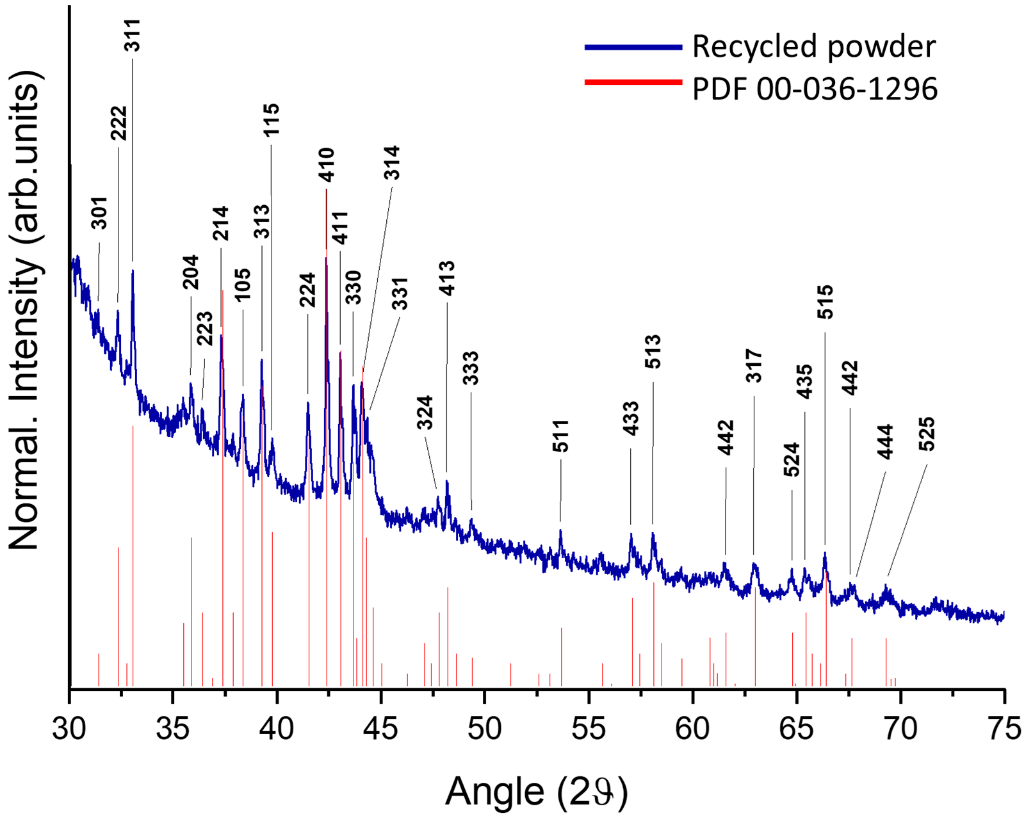


Figure 9. Comparison between Experimental XRD spectrum of recycled powder in vacuum impact mill (numbers denote the Miller's indices, (hkl)) and NdFeB bulk reference standard.

3.2. Magnetic Characterization and Magnetographic Observation for Recycled Powder

Figure 10 shows the demagnetization curves for two recycled powders obtained through the mechanical process, using impact mills. The exact quantity of powder for both techniques is filled in a particular holder. The holder with powder is magnetized and then characterized by a permanent magnet hysteresisgraph. The obtained results are used to compare the process effect, without estimating the final magnetic properties of a possible magnet. The coercivity fields in a vacuum impact mill increase by 33% with respect to the operation in argon. A slight reduction in remanence has been noted for the process in a vacuum. The difference in the coercivity field is due to the oxygen content, which is 4 times higher than the typical reference value.

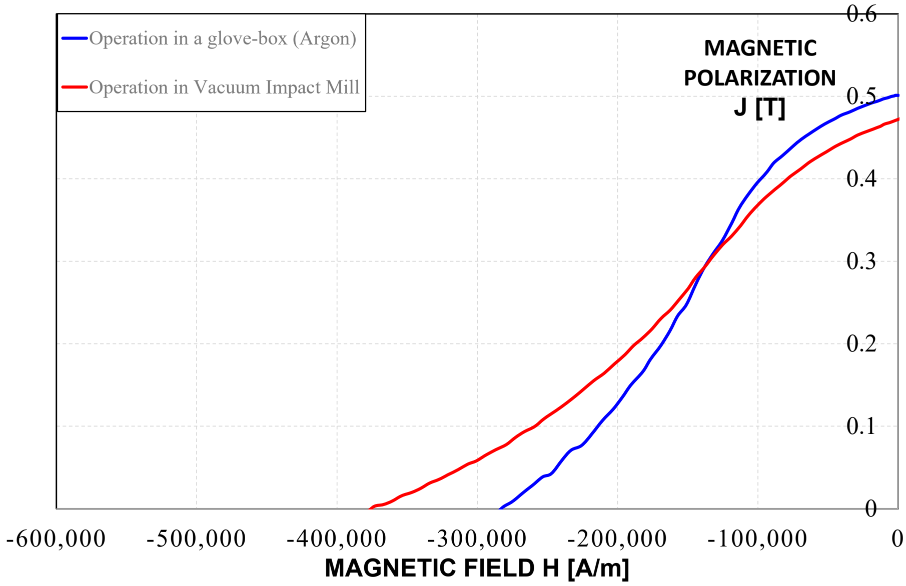


Figure 10. Demagnetization curves for two impact mill processes.

With the magnetic characterization of the powders, it is possible to study the density effect on magnetic performances (Figure 11). The density is changed by inserting the powder into a specific cylinder with dimensions 15 mm in diameter and 20 mm in height. Three density values have been implemented. The starting density (low density) corresponds to the density obtained after the powder is added into the calibrated cylinder (Figure 12a,b). The second density measurement (medium density) corresponds to the density obtained after manual powder compaction through the hysteresisgraph joke and dedicated expansion pole (Figure 12b). The third density value (high density) is similar to the second, but also considers an orientation through an external magnetic field.

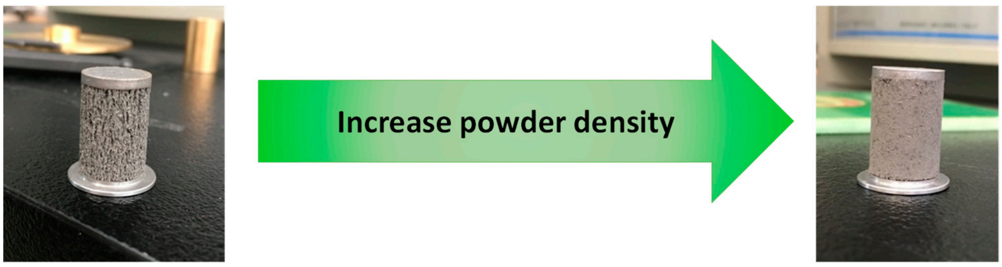


Figure 11. The density variation for the same volume cylinder of recycled NdFeB powder.

(**a**) (**b**)

Figure 12. The specific cylinder with dedicated tools: brass cylinder and separators discs with dedicated extension poles (**a**); insert in hysteresisgraph joke (**b**).

The demagnetization curves for the three densities of recycled NdFeB powder processed in vacuum are reported in Figure 13. As expected, the low-density sample shows the lowest magnetic characteristic, while the medium density one has a higher magnetic remanence. The demagnetization curve of the high-density recycled powder has the best coercivity properties, at the expense of a reduction in magnetic remanence. After analysis using the magnetographic method described in Figure 14, the presence of residual anisotropy can be observed, especially in Figure 14b,c. This parasitic phenomenon does not permit maximizing the magnetic performance of recycled NdFeB powder. The solution consists of using a specific magnetic orientation with a dedicated tool [45].

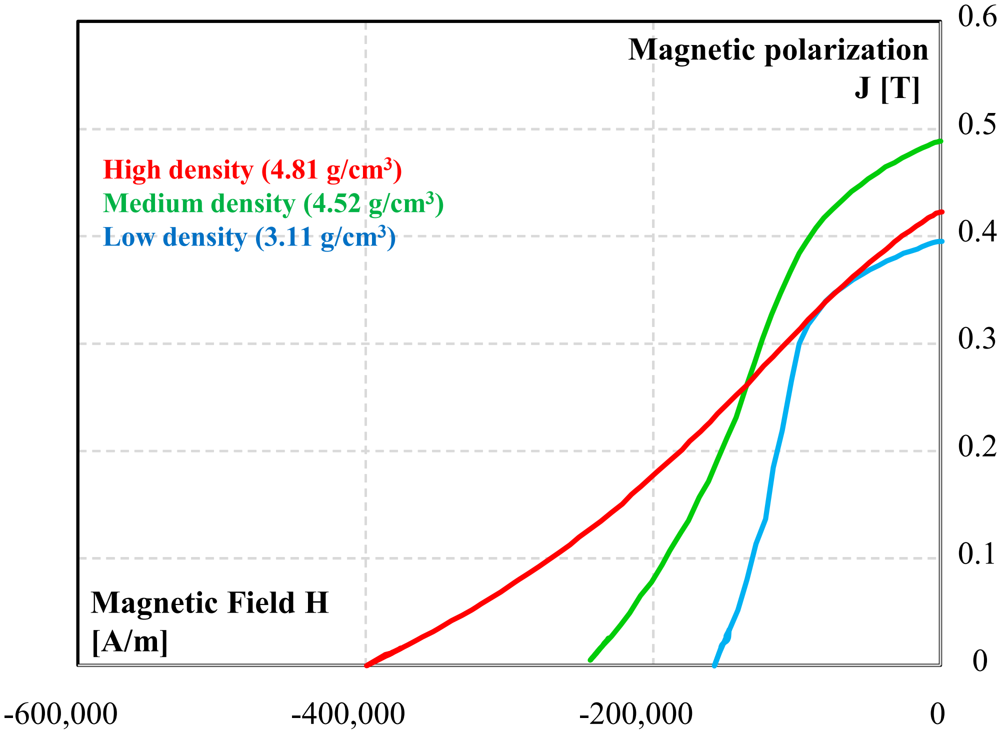


Figure 13. Demagnetization curves of the three powder densities for recycled NdFeB: low density 3.11 g/cm^3, medium density 4.52 g/cm^3, and high density 4.81 g/cm^3.

(**a**) (**b**) (**c**)

Figure 14. Magnetographic observation for three different densities: 3.11 g/cm^3 (**a**), 4.52 g/cm^3 (**b**), 4.81 g/cm^3 (**c**). The presence of residual anisotropy (**b**,**c**).

4. Conclusions

This paper investigated the quality and properties of NdFeB powders obtained from hard disk scraps.

The vacuum milling process represents an industrially viable and safe alternative to generate immediate re-usable powders from Nd-Fe-B magnets, rather than using more complex and expensive processes to recover Nd, Pr and Dy through use of hydrogen and/or chemical solvents. XRD and XRF analyses confirmed the good quality of the recycled powder, without any trace of added oxides. Furthermore, elemental analyses evidenced that a low amount of oxygen is present after operations in a vacuum impact mill, making this technique preferable to washing in an inert atmosphere. This is possible owing to simple equipment: heat treatment in the vacuum at 350 °C, and milling by means of a pump.

Although the presence of residual anisotropy represents a drawback, it does not necessarily imply negative potentiality of the proposed method, since this presence can be reduced with particular arrangements in the stages during the molding processes of the magnets. In this context, further activities will be performed to obtain recycled magnets through the use of dedicated tools, based on magnetic orientation.

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