

# Microscopic characteristic of Nd-Fe-B magnets structure - magnets recovered from electric motors

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**Abstract:** The rare earths (RE) are characterized with their strong metallic atomic bonds. They are used in magnetic materials used in computer technology and electric powered vehicles. The advancing globalization and the increasing standard of living both lead to massive waste disposal problems, therefore, the methods of recovering used metallic materials are sought. In this paper, the microscopic characteristic of magnetic powder from old electric engines has been presented.

**Key words:** NdFeB magnets, Rare earth elements, microscopic analysis, structure of NdFeB

## 1. Introduction

The hard magnetic materials play a large role in modern technology (automotive, electronics and many daily use equipment), the overview of statistics indicate that over the next few years the continuous increase of their consumption will be observed. Currently, China is the largest importer of rare earths elements, from which RE-M (rare earth elements, transition metal) magnets are produced. This situation is inconvenient for European countries because they depend on the largest producer.

For the research, the new magnetic material had been prepared from recycled hard magnetic elements from electric motors. The magnetic material characteristic includes, a brief outline of the recovery technology (treatment and processing of recycled permanent magnets) as well as a package of materials research – Digital microscope and Scanning electron microscope (SEM) analysis.

However, the NdFeB-sintered magnets have an issue that the magnetic properties is degraded by downsizing process, because the machined and damaged surface causes nucleation of magnetic domains. As demands for the higher magnetic performance of small-sized magnets are increasing in the application fields such as micro-motors and mobile electronic equipments, this problem is getting serious.

Hard magnetic materials are characterized by high coercivity, remanence, saturation flux density, high hysteresis energy losses, as well as low initial permeability. Like soft magnetic materials, restricting domain wall motion will enhance the coercivity of the material. Hard magnetic materials are often desired in numerous applications because they require no external power source and consequently generate no heat during usage. Hard magnets are essentially energy storage devices, which proper handling can retain their magnetism [2].

The coercivity of hard magnetic materials is strongly dependent on the microstructural features of the material. The experimental coercivity always factors approximately 3-5 times less than the theoretical prediction of the material; this is known as Brown's paradox [3]. This results when particle size increases to the point where the particle can contain domain walls which aid in the nucleation and propagation of reverse domains decreasing the material's coercivity.

Dimagnetic materials have the unique of creating magnetic fields in direct opposition of the applied magnetic field. Diamagnetism is a weak form of magnetism, all materials exhibit a diamagnetic signal. Diamagnetism persists only while an external field is present. When a diamagnetic material is placed between the poles of a strong electromagnet, the material is attracted to weak regions in the field [1]. Diamagnetic materials are typically those which are considered non-magnetic. Most organic compounds fall in this classification, as well as many metals such as copper, mercury and gold. Metals which are typically diamagnetic are heavy metals with core electrons.

## 2. Material preparation

The study has been performed on samples of the Nd-Fe-B type magnets - powder obtained by mechanical crushing of magnets from old electric motors (Fig.1).

Taking into account the multi-phase structure of the magnets and neodymium high affinity to oxygen, the preparation of materials has been conducted in a noble gas atmosphere (Ar). For demagnetization, the magnetic material has been heated in an oven to a temperature above the Curie temperature.



Fig.1. Magnets from electric motors

Subsequently grinding in ball mill has been performed, keeping in mind that excessive fragmentation of the powder will reduce the magnetic coercivity, which in turn would result in the loss of magnetic properties of the starting material [4,5]. Figure 2 shows the example of demagnetization curve for permanent magnets depicting the process of magnetization and magnetic spins during this process.

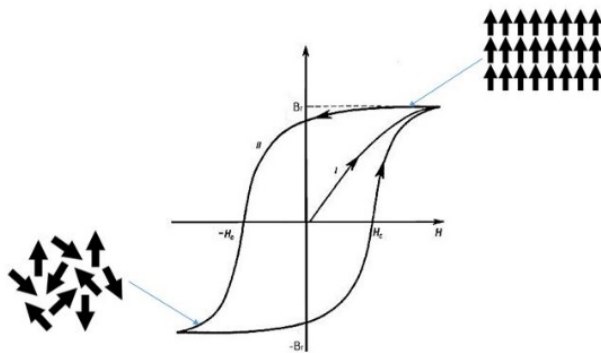


Fig.2. Important features of the ferromagnetic hysteresis loop

The crystal structure and symmetry of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase result from mirror symmetry in two orthogonal planes and contribute to its strong magnetic properties. The structure of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  is tetragonal in structure and had space group symmetry of  $\text{P4}_2/\text{mm}$ , neodymium and iron are in parallel alignment within the sublattices. This symmetry produces a magnetization of approximately 1,6 T at room temperature and an anisotropy of resulting from 'mm' symmetry through the two neodymium sites [6].

Microstructure plays a crucial role in the magnetic properties of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  magnets. The magnetization and demagnetization can be altered by controlling the size, shape, and orientation of the grains. To achieve different microstructures, three different processing techniques are currently employed powdered metallurgical methods- sintering and melt spinning and mechanical alloying.

### 3. Results and Discussion

The typical microstructure of a NdFeB sintered magnets is shown in the optical microscopy image in Figure 3. The  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grain boundaries and larger grains at the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grain junctions. The larger, more rounded Nd-rich grains are usually neodymium oxides, whereas the smaller, angular metallic in character. The thin metallic grain boundary layers tend to be amorphous when the thickness is below 1 nm and become crystalline at greater thicknesses.

During the tests of microscopic analysis of the magnetic material three phases were observed. As the typical NdFeB-magnets structure consists of three phases: the ferromagnetic phase  $\text{Nd}_2\text{Fe}_{14}\text{B}$  ( $\Phi$ ) about 85% of the phase volume, the boron-rich phase  $\text{NdFe}_4\text{B}_4$  ( $\eta$ ) about 3% and the neodymium-rich phase NdFe about 12% mass. Neodymium magnets are structured in the multi-phase skeletal manner, i.e. phases rich in neodymium fill intergranular areas around grains of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase.

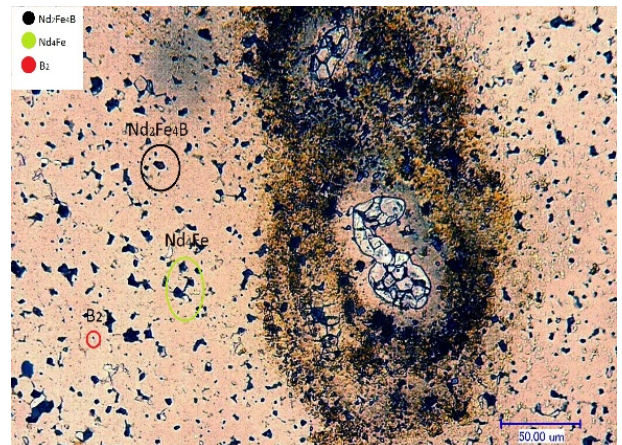


Fig.3. The microstructure images of RE-M-B magnetic material recovered from electric motors (marked ferromagnetic, Nd-rich and B-rich phases)

After a grinding process, a ready-to-use powder is present in particle sizes of 20  $\mu\text{m}$  to 320  $\mu\text{m}$ , which consists of a magnetically isotropic conglomerate of small NdFeB crystals with crystal sizes between 50 and a few hundred nanometers. This microcrystalline structure is responsible for the high magnetic and chemical stability of the material. Samples of the SEM analysis (Figure 5, 6) show a finely ground powder.

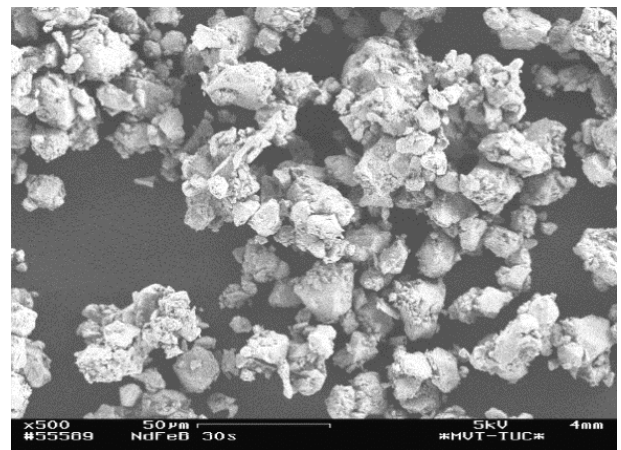
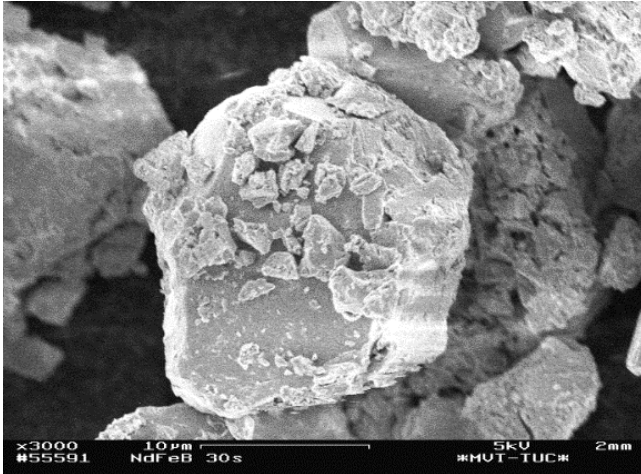
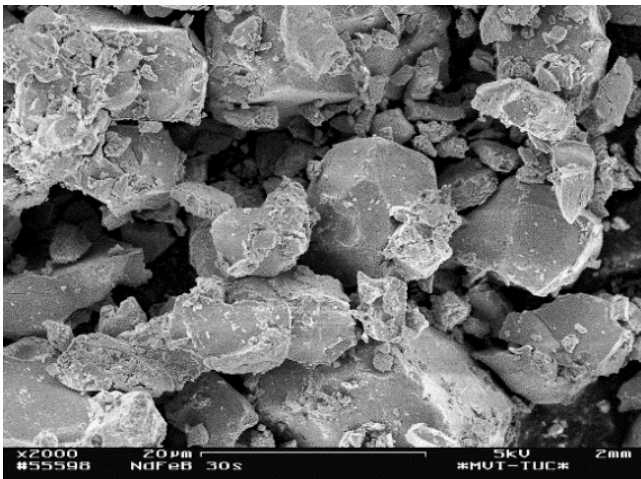


Fig.4. Results of scanning electron microscope of RE-M-B magnetic powder microscopic magnif. x500

After the chemical reaction, the material is comminuted by means of the grinding processes in a disk swing mill to form powders of different grain sizes. The grain sizes must be matched to the type of magnet modification to be produced. For the production of magnetic anisotropic, plastic-bound hard ferrite magnets, single-crystalline, magnetic, directional powder particles with a grain size of 10  $\mu\text{m}$  are required.



*Fig.5.Results of scanning electron microscope of RE-M-B magnetic powder microscopic magnif. x3000*



*Fig.5.Results of scanning electron microscope of RE-M-B magnetic powder microscopic magnif. x2000*

## Literature

- [1] Callister, William D. (2002) Materials Science and Engineering an Introduction, New York, Hoffman Press, pp. 673-700. Vol. 11.
- [2] Cohen, Morris, [ed.] (1972), Introduction to Magnetic Materials, Reasing Massachusetts, Addison-Wesley Publishing Company, pp. 181-202, 287-351, 556-626, Vol.11.
- [3] Weng, Robert S., WIJ, Jiin-Chuan and Kryder, Mark H. (1991) The effect of random coercivity on domain growth processes in rare earth-transition metal alloys, Vol. 69,8, pp. 4856-4856.
- [4] Klimecka-Tatar D., Bala H., Ślusarek B., Jagielska-Wiaderek K. (2009) The Effect of Consolidation Method on Electrochemical Corrosion of Polymer Bonded Nd-Fe-B Type Magnetic Material. Arch Metall Mater 54/1, s. 247-256.
- [5] Elwert, T., & Goldmann, D. (2015) Entwicklung eines hydrometallurgischen Recyclingverfahrens für NdFeB-Magnete. Univer-sitätsbibliothek Clausthal.
- [6] Berkowitz, Ami E. and Kneller, Eckart (1969) Magnetism and Metallurgy, New York, Academic Press, Inc.
- [7] Zakotnik, M., Harris, I. R., & Williams (2008) A. J. Possible methods of recycling NdFeB-type sintered magnets using the HD/degassing process. Journal of Alloys and Compounds, 450(1), 525-531.
- [8] Wnuk I., Wyslocki J. and Pryzbyl A. (2007) Phase structure of nanocrystalline Nd<sub>2</sub>Fe<sub>14</sub>B magnets with different grain size, pp. 85-88.

## 9. Summary and conclusions

The grain size of the starting material was determined using Digital Microscopy and Scanning electron microscopy (SEM). Microstructure plays a crucial role in the magnetic properties of Nd<sub>2</sub>Fe<sub>14</sub>B magnets. The magnetization and demagnetization can be altered by controlling the size, shape, and orientation of the grains. The achieve different microstructures, three different processing techniques are currently employed powdered metallurgical methods-sintering and melt spinning and mechanical alloying.

Grain size and grain distribution are important criteria for the intrinsic coercivity of the final magnet. Depending on the type of processing technique different grain sizes are found in the magnet. The microstructure of cast and extruded magnets is rather unknown, but first investigations show that the grain diameters are comparable with the one of sintered magnets.