



Recovery of Rare Earth Elements from permanent magnets

Speaker: Yannick Menard, y.menard@brgm.fr

Experts contacts:

N. Menad n.menad@brgm.fr

A. Seron a.seron@brgm.fr

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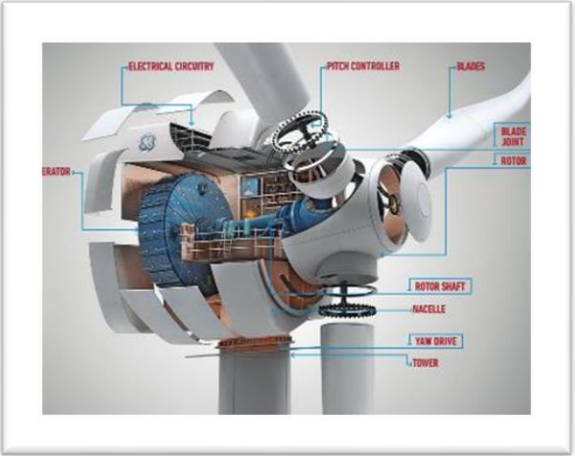


CONTEXT

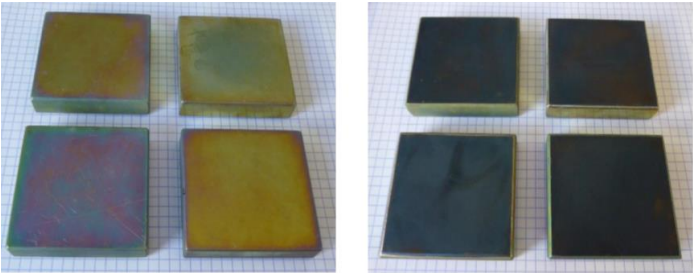
Research guidelines

- High content of REEs (strategic metals) in permanent magnets contained in WEEE (secondary resources)
- Development of innovative mechanical sorting process to recover magnets from HDD which is not actually industrialized
- Development of alternative route to short loop allowing fragmented and polluted magnet valorization using soft and green chemistry
- Implementation of developed processes at pilot scale

PERMANENT MAGNETS



Wind Turbine Magnets



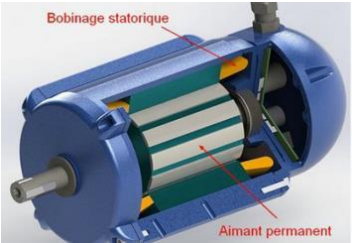
Hard disc drives from computers



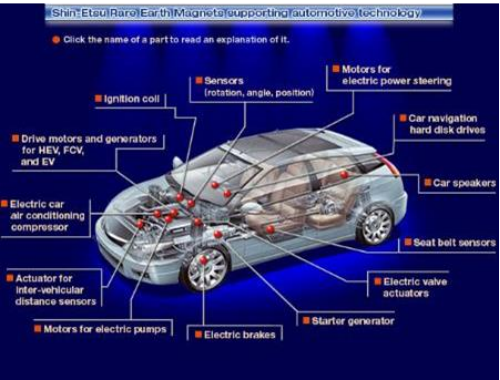
Electric scooter



Electrical motors



Electric vehicles



THE HISTORY OF THIS RESEARCH AT BRGM IN A FEW FIGURES

➤ 8 years of research

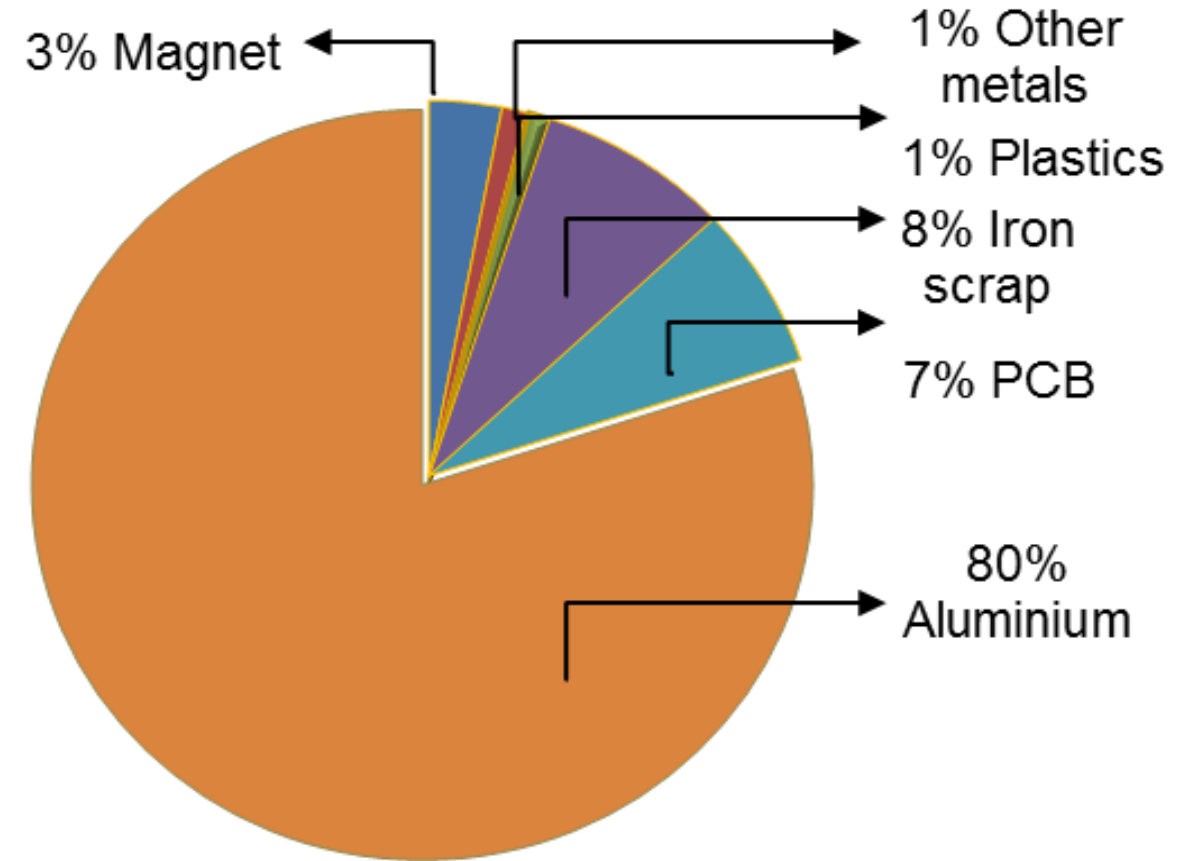
➤ 3 Research projects

- | | | |
|------------------------------------|----------------|-------------|
| ✓ EXTRADE (completed) | French funding | (2014-2017) |
| ✓ VALOMAG (completed) | EIT RM | (2020-2022) |
| ✓ PEPR Strategic metals (starting) | French funding | (2023-2025) |

➤ 13 partners where 6 research centres, 1 eco-organism, 6 companies

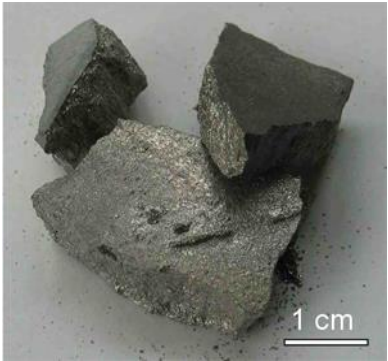


HARD DISC DRIVE COMPOSITION



PERMANENT MAGNETS

hydrogen																helium															
1 H 1.0079																2 He 4.0026															
lithium																beryllium															
3 Li 6.941																4 Be 9.0122															
sodium																magnesium															
11 Na 22.990																12 Mg 24.305															
potassium																calcium															
19 K 39.098																20 Ca 40.078															
rubidium																strontium															
37 Rb 85.468																38 Sr 87.62															
cesium																barium															
55 Cs 132.91																56 Ba 137.33															
francium																radium															
87 Fr [223]																88 Ra [226]															
lanthanide series																actinide series															
periodic table showing elements B, Fe, and Nd highlighted in red circles.																															



Rare earths : 17 elements

60

Nd

Neodymium

144.24

Pr

26

Fe

Iron

55.847

Dy

5

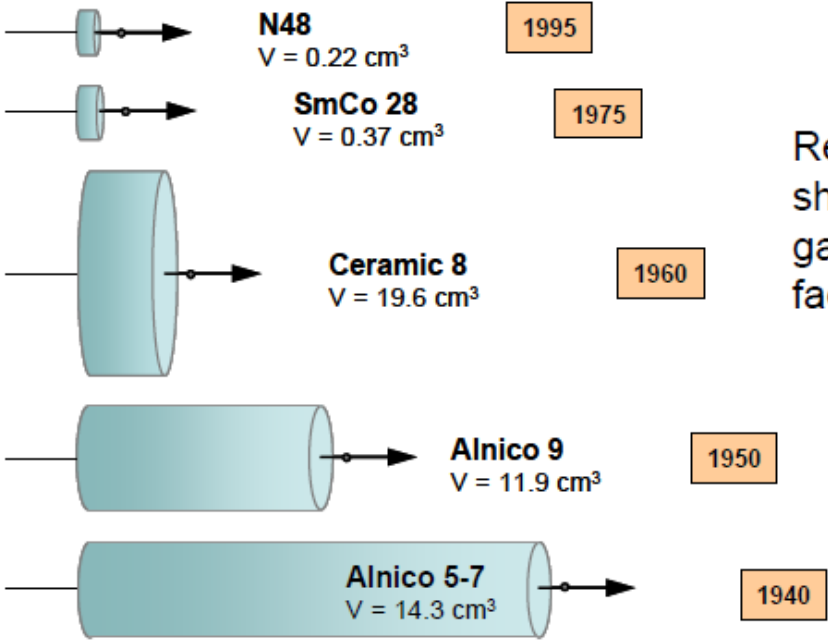
B

Boron

10.811

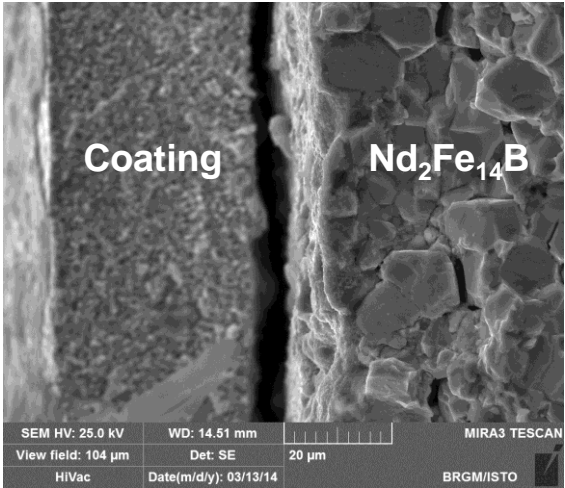
Nd-Fe-B ingot produced by arc-melting

Nd-Fe-B magnets with nickel plating

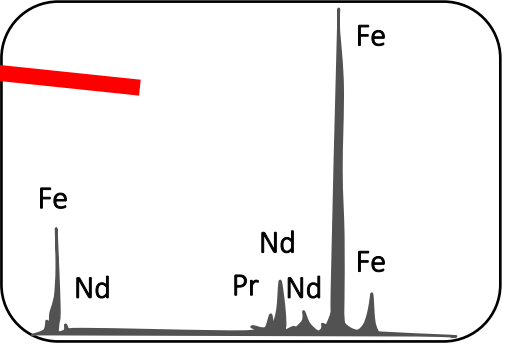
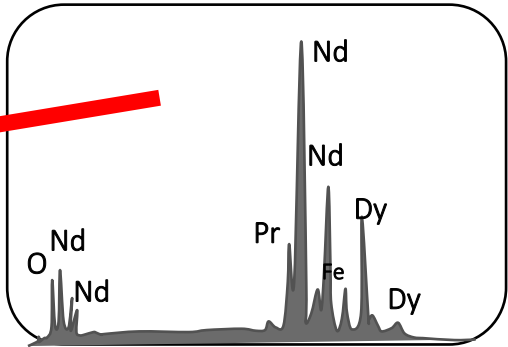
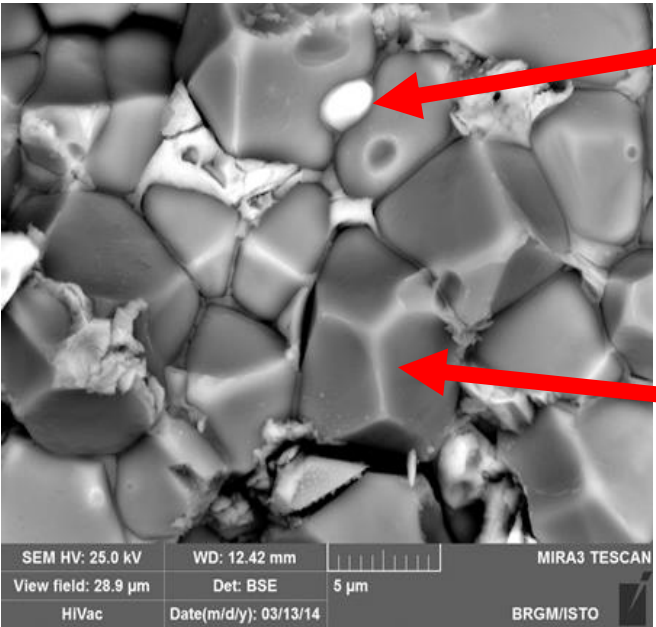


Relative magnet size and shape to generate 1000 gauss at 5 mm from the pole face of the magnet.

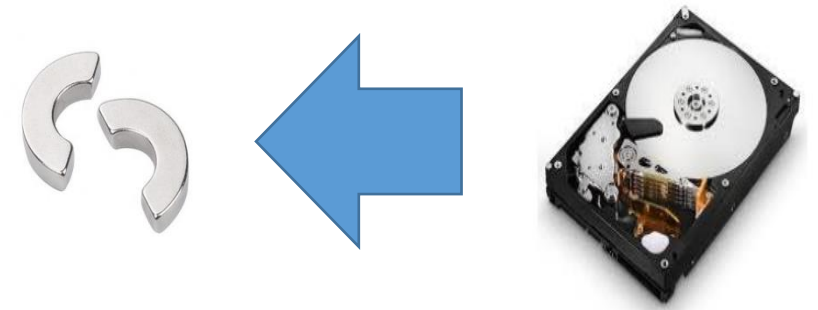
CHARACTERISATION



- ❖ Nd₂Fe₁₄B magnet
- ❖ Coating (Ni or Ni/Cu) thickness 28µm



Elem	Weight %	Nd ₂ Fe ₁₄ B Theoretical
Fe	69.6	72.0
Nd	28.1	27.0
Dy	2.3	--
B	--	1,0
Total	100.0	100.0



LOCKS OF RECYCLING NdFeB MAGNETS

- ✓ Difficulties related to the dismantling of products due to their often compact and complex design
- ✓ Variation in chemical composition, even within the same IT application
- ✓ Strict requirement of the REEs market in terms of purity degree
- ✓ Difficulties related to the separation between REEs due to their similar physico-chemical properties
- ✓ Presence of other undesirable metals (coating Cu/Ni and Fe)

OBJECTIVES

- To propose an outlet allowing REEs that can be recovered from end of life permanent magnets extracted under forms (fine powders, specific composition, mixtures) that cannot be reused in the short recycling route
- To optimize leaching procedure (nature of leaching media, residence time, concentration of the leaching agent, temperature, ...);
- To improve kinetics of leaching and conversion efficiency

MECHANICAL SORTING

Patented results

- ✓ 1ton HDD treated
- ✓ 350Kg HDD/day
- ✓ Recovery: 95% of magnets
- ✓ Purity: 88%
- ✓ Other fractions to be recovered (aluminum, circuit board, steel)



HDD Components obtained after sorting

Al & PCB rich
fraction
+ 40 mm



Iron scrap rich fraction
-20 +16 mm and -40 +20
mm



Magnet rich
fraction
-16 +5 mm



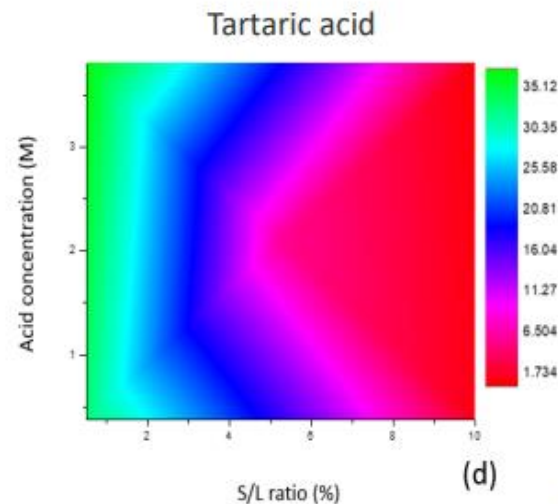
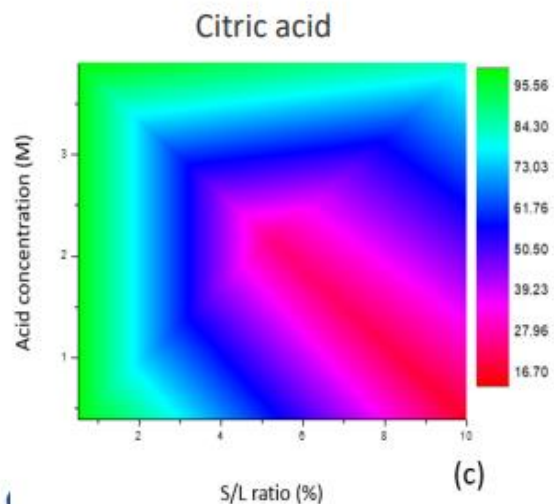
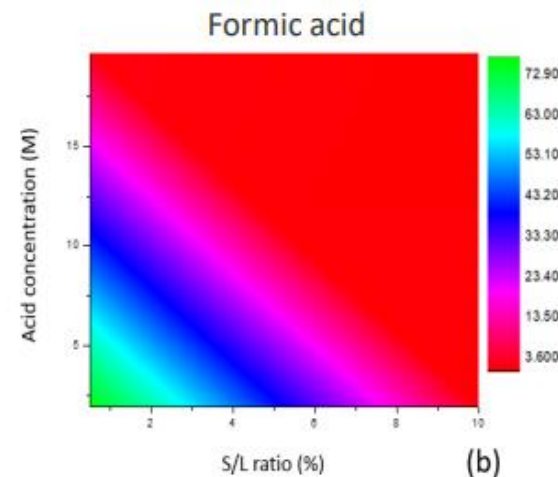
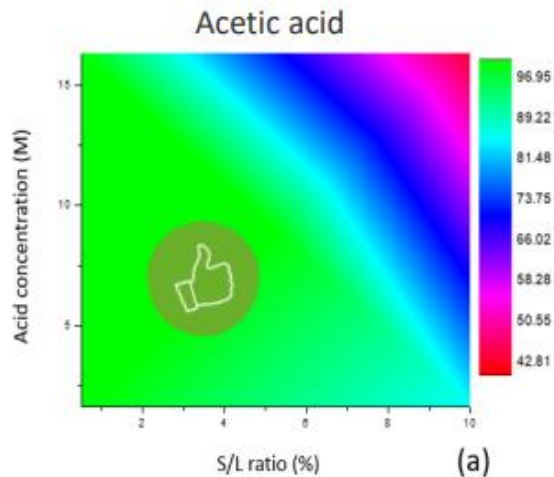
Metal mixtures
rich fraction - 5
mm



LEACHING OF NdFeB POWDER USING ORGANIC ACIDS

Patented results

Iso-response curves of the leaching yield (%) of Nd from NdFeB magnet powder in different organic acids at 60°C for 24 hours as a function of the S/L ratio (%) and the concentration of acids



Nd, Pr and Dy have the same leaching behavior in all tested acids

Acetic acid: Best candidate for leaching REEs under industrially favorable conditions; high S/L ratios and low acid concentrations

> 90% of REEs leached:

S/L ratio (%) [0.5 - 5]

Acetic acid concentration (M) [1.6-10]



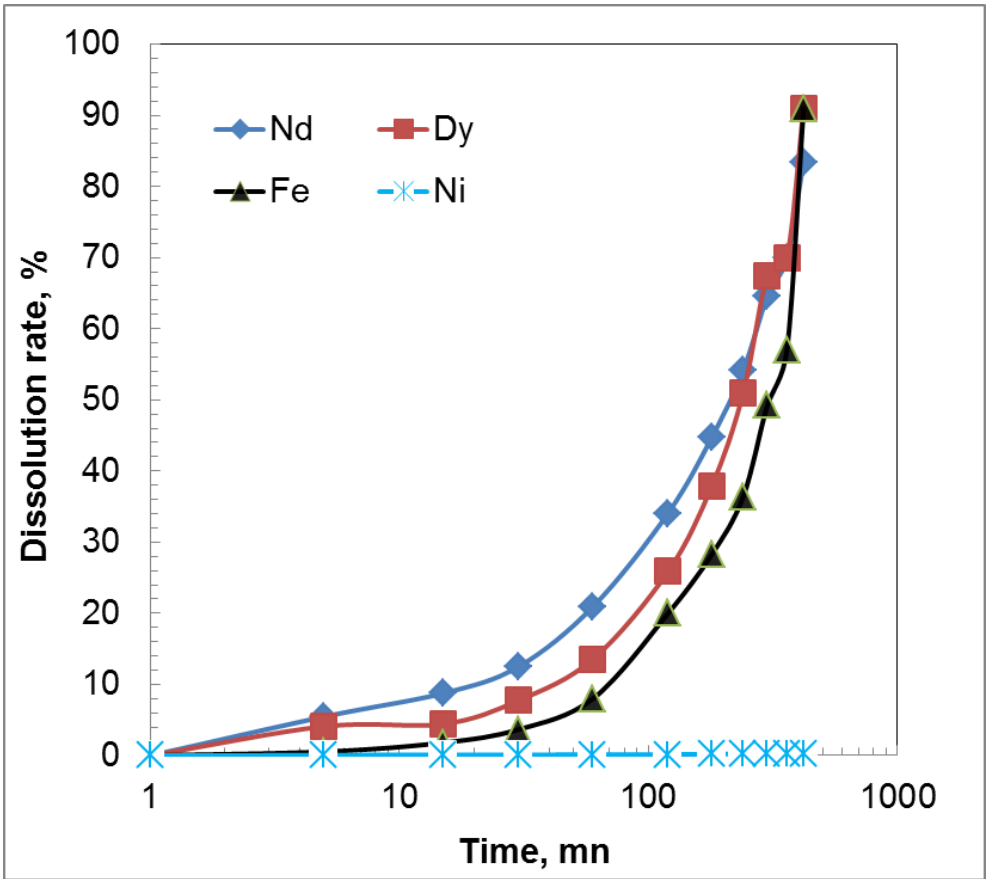
Partial/ total co-leaching of Fe, Co and B

Formic acid: Precipitation of REEs in formates

Tartaric acid: Precipitation of REEs in hydroxides

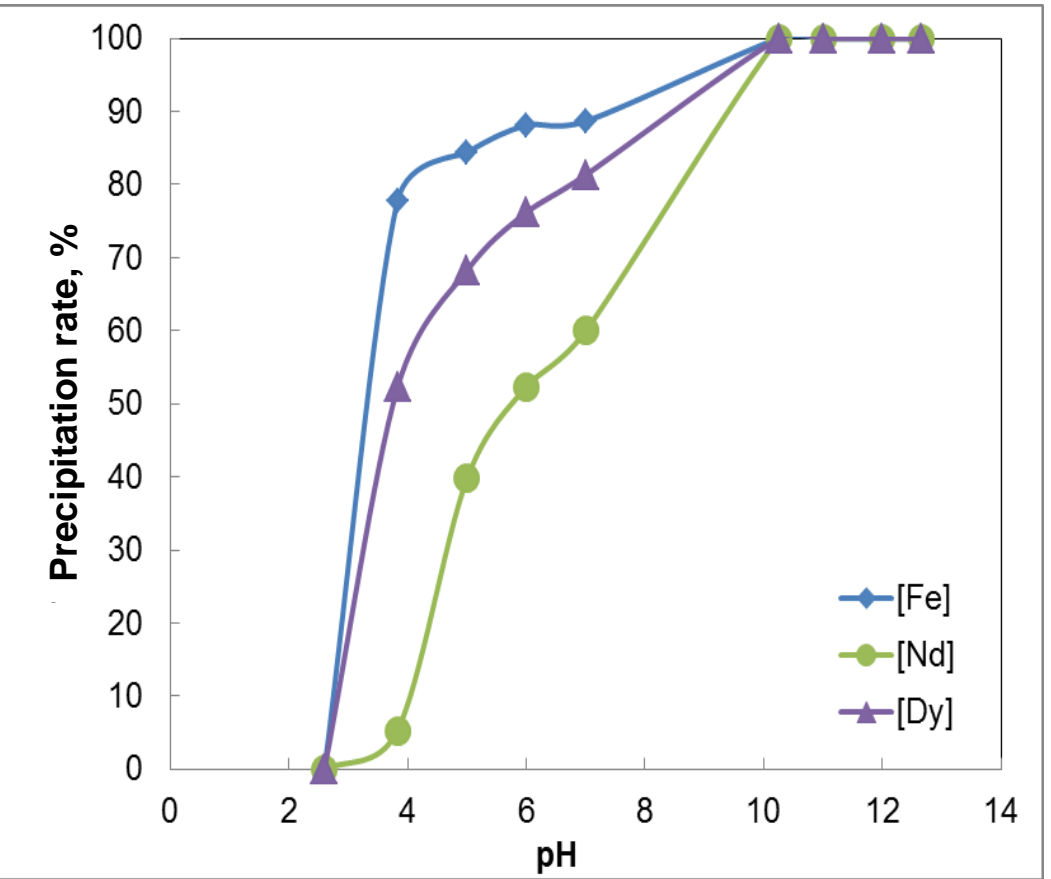
ROUTE 2: EXTRACTION OF REES USING INNOVATIVE HYDROMETALLURGICAL TECHNIQUES

Dissolution kinetics of permanent magnets



Selective leaching of Fe, Nd, Dy

Precipitation of Fe, Nd, Dy using soda solution



Co-precipitation of Fe, Nd, Dy at pH 2-3

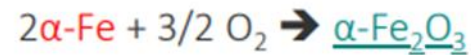
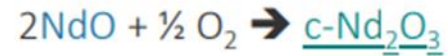


Look for alternative treatment to avoid iron leaching during REEs dissolution

ROUTE 2: EXTRACTION OF REES USING INNOVATIVE HYDROMETALLURGICAL TECHNIQUES

Patented results

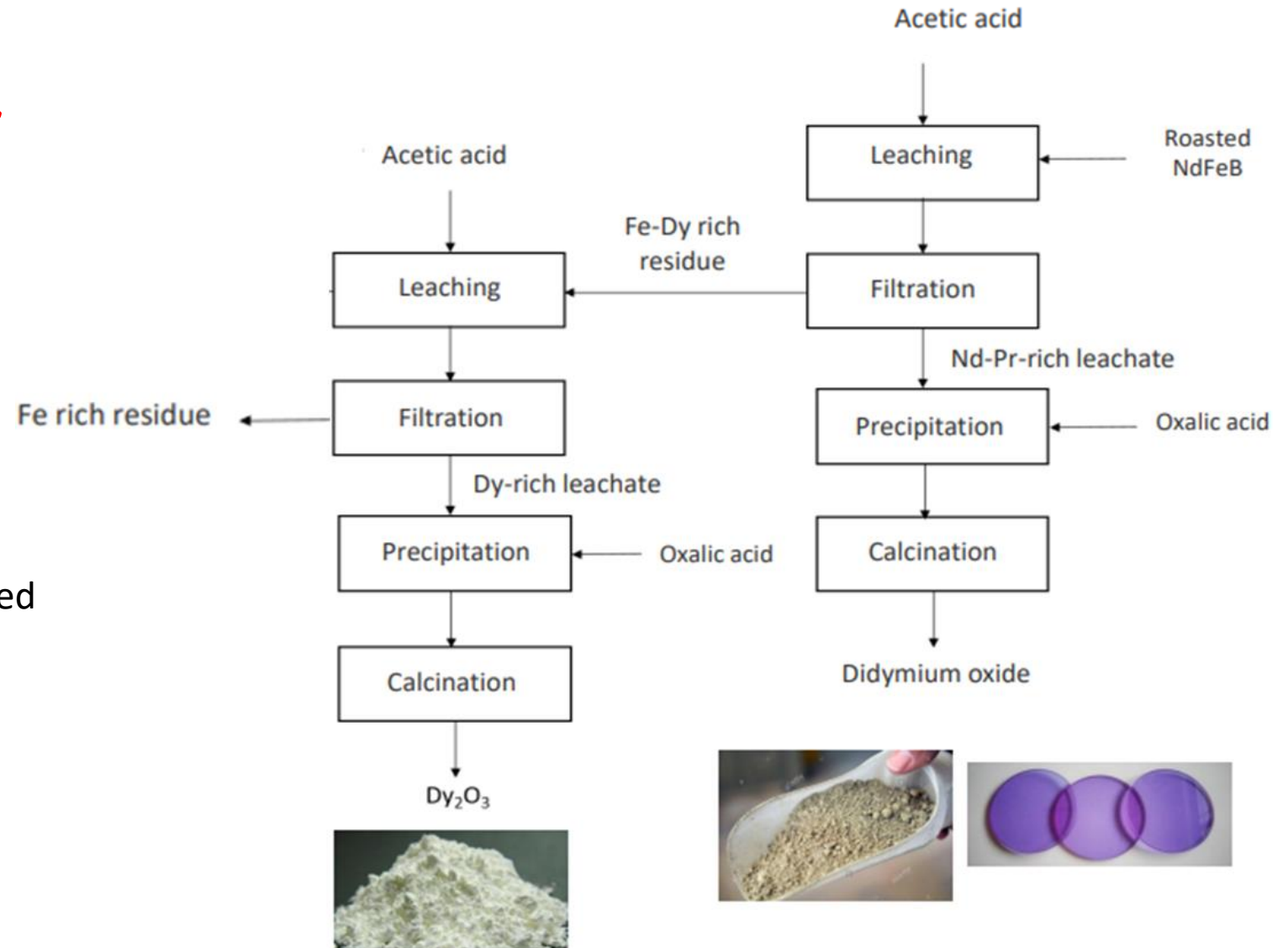
Oxidative roasting of NdFeB powder



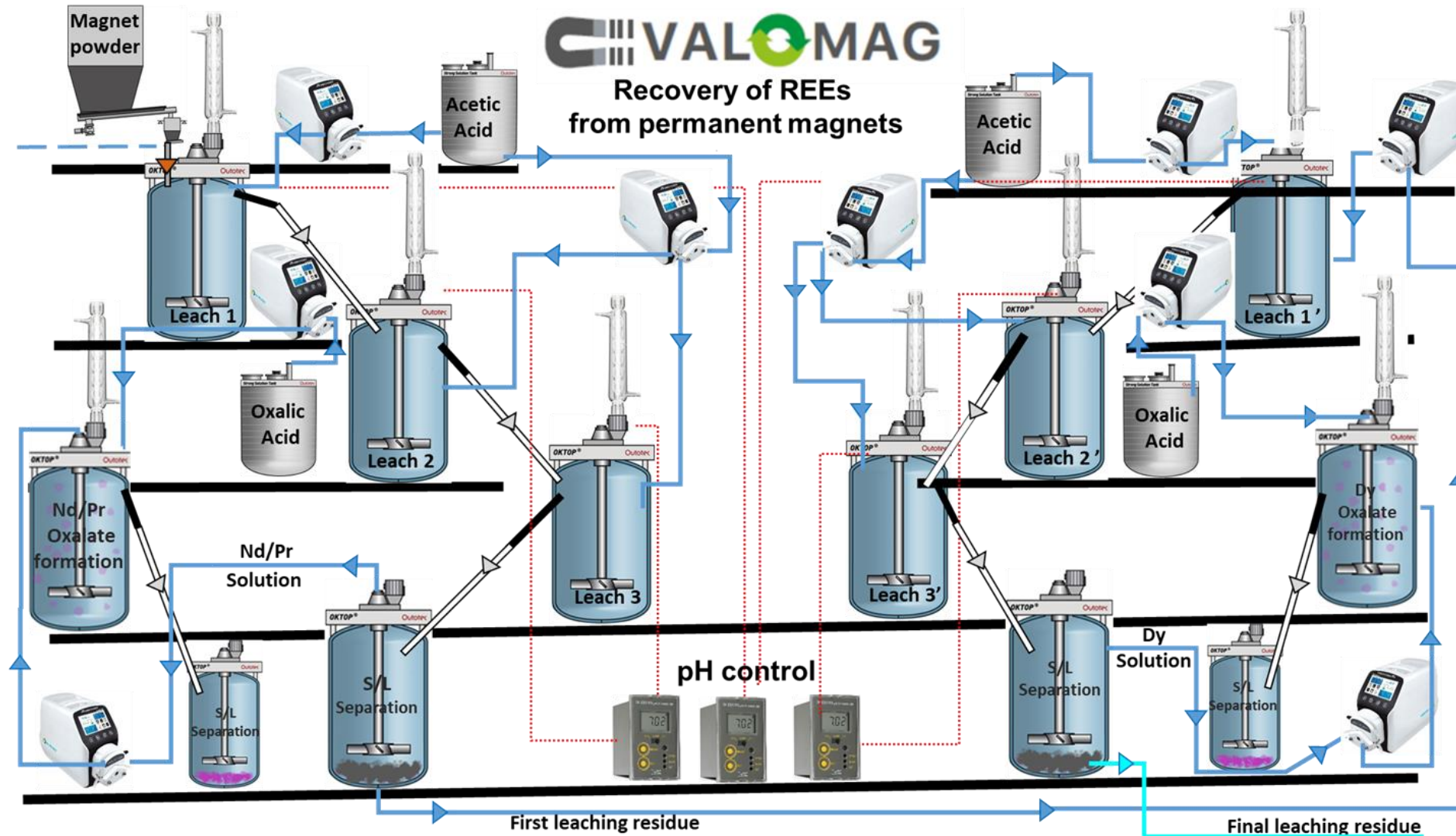
From 500°C:



- Selective leaching of REE / Fe confirmed when starting from oxides:
 - Recovery Nd, Pr, Dy > 95%
 - Purity > 99%
- On going studies en NdFeB magnets



PILOT TEST: EXTRACTION OF REES FROM MAGNETS BY HYDROMETALLURGICAL TREATMENT



- Magnet powder
- 600g/hour
- $T^{\circ}\text{C} = 30^{\circ}\text{C}$
- Reactants
 - Acetic acid
 - Oxalic acid

*REEs precipitation
using oxalic acid*



*Nd + Pr
oxalate*

*Dy
oxalate*

Recovery by precipitation: Nd+Pr 80%, Dy 100%

Residue: almost iron



CONCLUSIONS

- ❖ The recycling of permanent magnets: an ecological solution to help reducing our dependence on REEs supply
- ❖ Identification of Nd-Fe-B magnets present in WEEE:
 - ✓ 98% recoverable in small electronic appliances feed
 - ✓ The rest is disseminated in other equipments such as fixed phones, mobiles, internet boxes (more difficult to recover)
- ❖ Two sampling campaigns were conducted on two different industrial sites to collect a representative sample of hard drives and loudspeakers
- ❖ Results of characterization, Wt% of magnet in electronic components:
4 - 6% in the loudspeakers, 2.5 - 2.8% in fixed computer hard drives, 2 - 3% in laptops hard drives, and between 0.8 and 2% in small electric motors

CONCLUSIONS

- ❖ Scanning electron microscopy (SEM) was used to:
 - ✓ Identify Nd-Fe-B magnets
 - ✓ Observe Nd-Fe matrix in a tetrahedral form and an inter-granular space rich in REEs (Nd, Dy, Pr).
 - ✓ Characterize the coating layer of Ni, Zn or Cu / Ni alloy (20-30µm thick).
- ❖ XRD reveals:
 - ✓ Two types of magnets: Nd-Fe-B and ferrites
- ❖ TGA shows:
 - ✓ Working T°C range of Nd-Fe-B type magnet goes from ambient to 150°C
 - ✓ Curie point is close to 300°C for Nd-Fe-B type magnet, 500°C for ferrite magnets.
- ❖ Mechanical sorting process developed and patented by BRGM allows recovering more than 95% of magnets from HDD
- ❖ Organic acid (acetic) may selectively REEs contained in magnets, and oxalic acid was used to precipitate REEs.
- ❖ Products obtained from the process developed and patented are: Didymium and Dysprosium oxides



Thank you for your attention !



Virtual visit of BRGM Piloting Facility :
<https://www.brgm.fr/fr/laboratoire-plateforme-technologique/plat-inn-economie-circulaire-entre-laboratoire-echelle>

