INSTITUTO DE PESQUISAS TECNOLÓGICAS

COMUNICAÇÃO TÉCNICA

Nº 178792

Development of oxalate-based layers for temporary protection of NdFeB magnets

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Palestra apresentada no LATINCORR & INTERCORR 2023, Rio de Janeiro . 17 slides.

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Summary

- Overview - Rare Earths Permanent Magnets
- (Nd,Pr)FeB magnets microstructure
- Motivation
- Objective
- Methodology
  - Surface conditioning
  - Layer formation
- Results
- Conclusion

Overview – Rare Earths Permanent Magnets

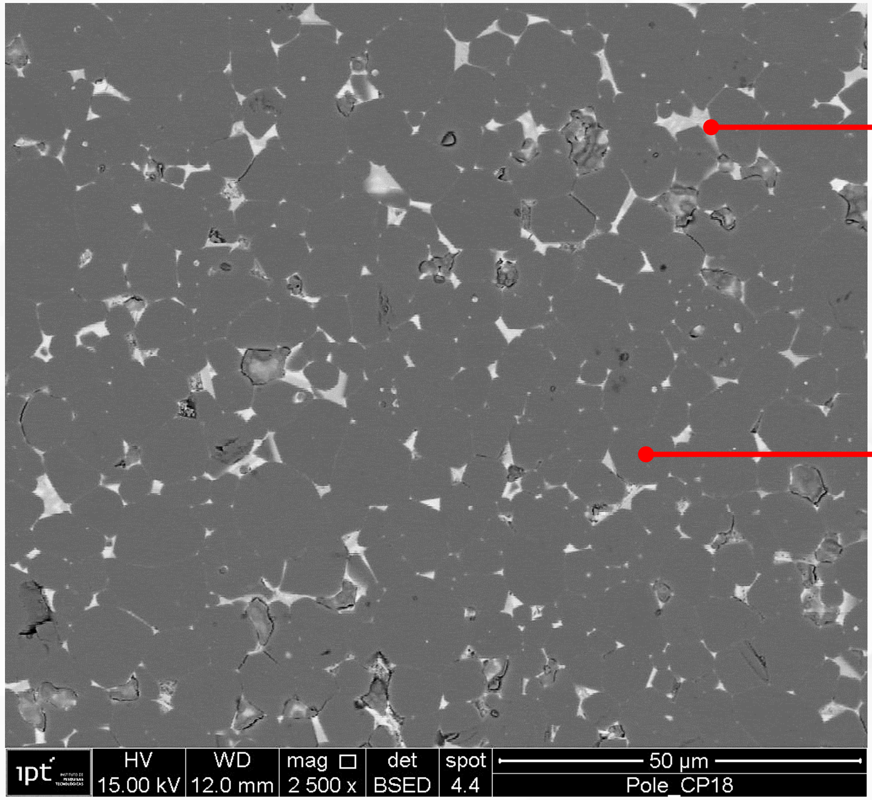
Rare earth PM AC machines are widely used in industry nowadays. Their high efficiency as well as high energy density bring unparalleled superiority. On the one hand, the **renewable energy** markets are estimated to gain explosive growth in the next decades, such as **Hybrid (H) and pure Electrical Vehicles (EV)**, E-scooter, **Wind Turbines**, Electrified Marines, and Aircrafts, etc.

Li, Z., Kedous-Lebouc, A., Dubus, J.M., Garbuio, L. and Personnaz, S., 2019. Direct reuse strategies of rare earth permanent magnets for PM electrical machines–an overview study. *The European Physical Journal Applied Physics*, *86*(2), p.20901.

https://exame.com/negocios/veiculos-eletricos-devem-dominar-vendas-ate-2033-diz-ey/

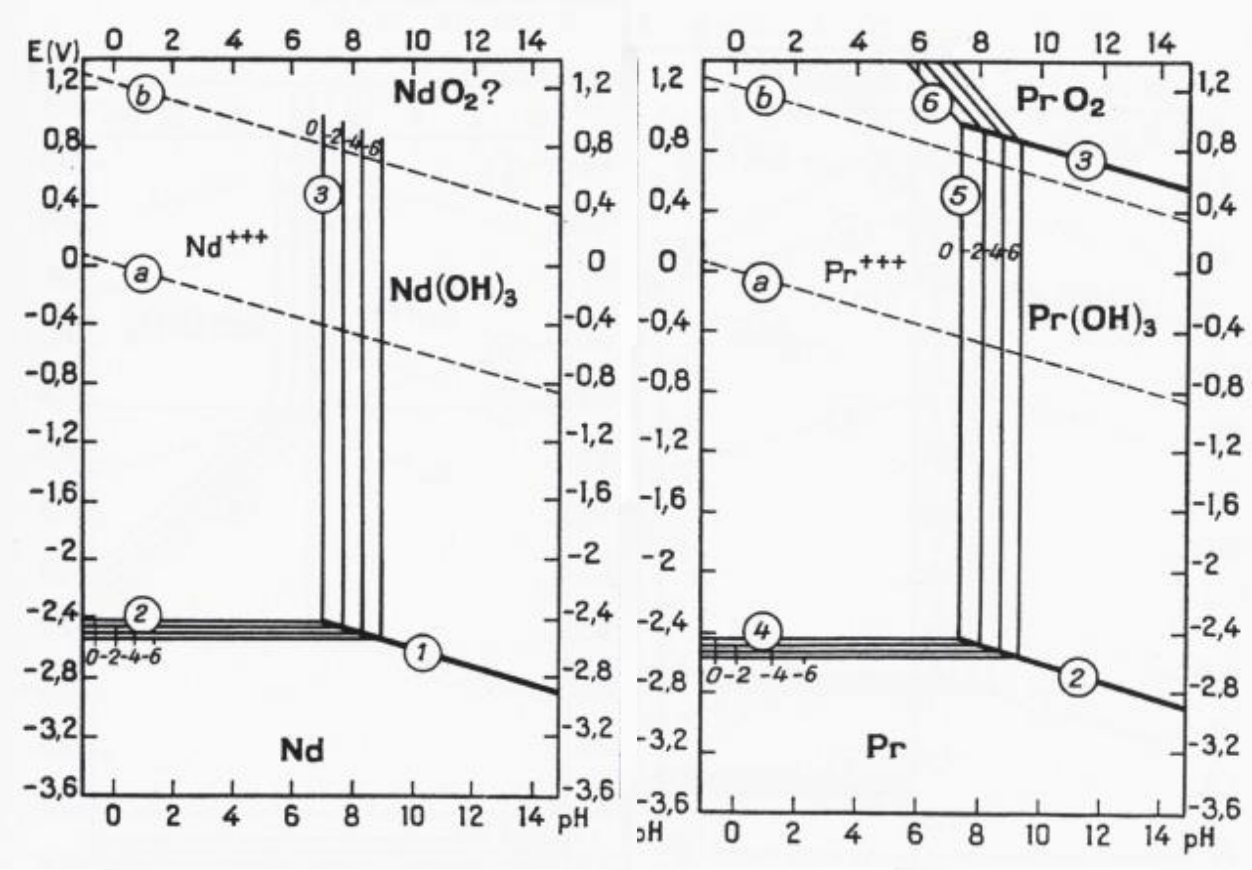
https://epocanegocios.globo.com/Brasil/noticia/2018/01/brasil-adere-agencia-internacional-de-energia-renovavel.html

(Nd,Pr)FeB Magnets Microstructure



Rare earth rich phase

Magnetic phase



Motivation

Phosphate chemical conversion processes **successfully produce** non-conductive phosphate coatings on different metals

While these processes are well established, they **produce sludges** that **need to be treated** and can generate **environmental liabilities**

Oxalate-based protective layers can be an alternative coating for temporary protection of the magnet and as a pretreatment for anchoring organic coatings

https://www.conserve-energy-future.com/wp-content/uploads/2015/12/eco-friendly-sustainable-living.jpg

Objective

Evaluate the use of oxalate-based protective layers as an alternative coating, obtained by immersing (Nd,Pr)FeB magnets in an oxalic acid solution

Methodology

Samples

- Commercial (Nd,Pr)FeB magnets, grade N42

Elements (mass fraction %)								
Fe	**Nd**	**Pr**	**Co**	**Gd**	**Al**	**Cu**	**Dy**	**Total**
68,95	22,71	6,91	0,54	0,42	0,27	0,13	0,06	99,99

Methodology

Samples

NdFeB cubes

- (Nd,Pr)FeB cubes (10 mm x 10 mm) uncoated and non-magnetized

Sanded

- (Nd,Pr)FeB cubes sanded with emery papers (from #400 to #1200) and sonicated with acetona

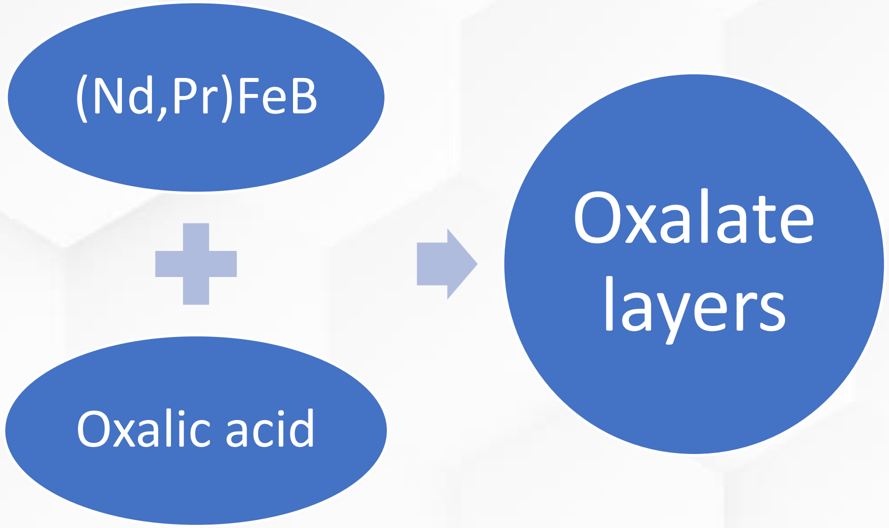
Oxalic acid solution

- The sanded cubes were exposed to a 0.11 mol/L oxalic acid solution, stirred at 500 rpm, and tested at two temperatures 25 °C and 50 °C

Methodology

Formation and monitoring of oxalate layers :

- E vs. t recording with SCE (VersaSTAT 3 – PAR)
- Two-electrode cell
  - **Ref** = SCE;
  - **WE** = (Nd,Pr)FeB
- 0.11 mol/L oxalic acid as electrolyte



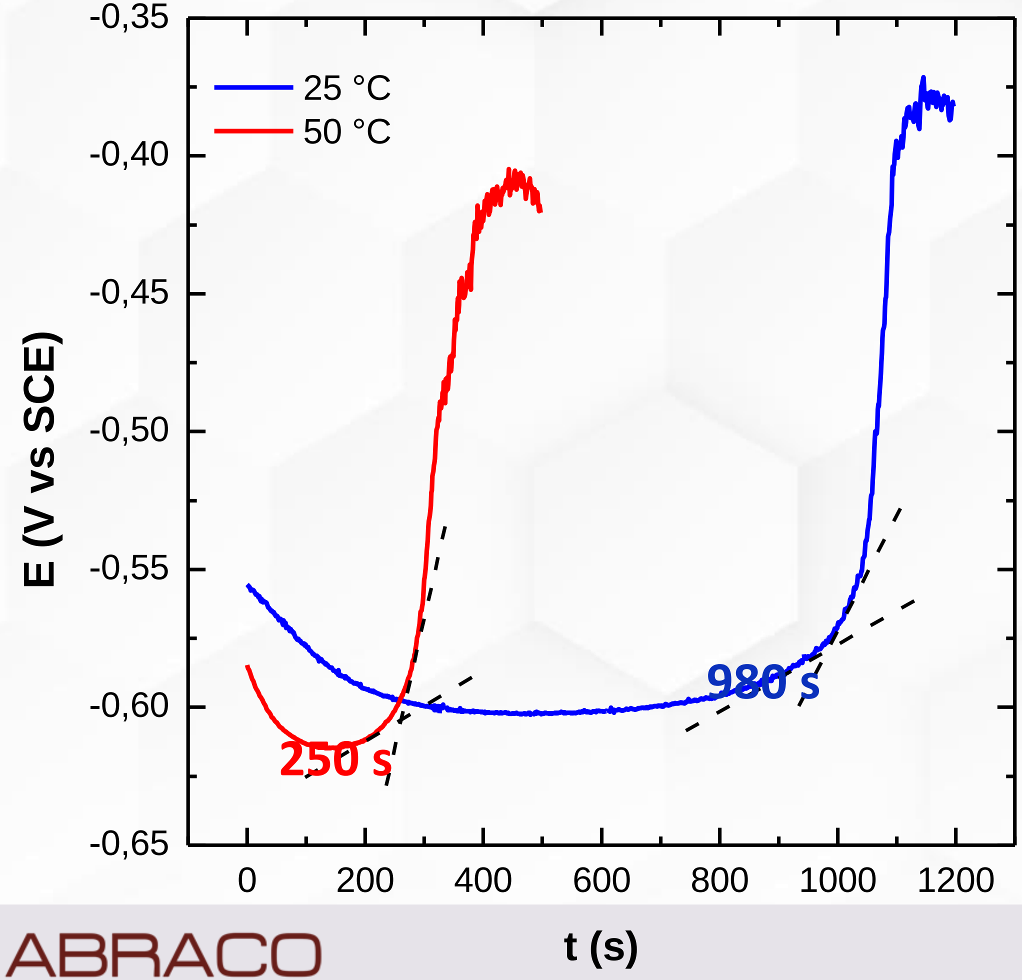
Methodology

Characterization of oxalate layers obtained:

- scanning electron microscopy (SEM) (FEG – FEI – Quanta 400)
- electrochemical impedance spectroscopy (EIS) (VersaSTAT 3 – PAR)
  - three electrodes cell
    - **Aux**= Pt;
    - **Ref** = Ag/AgCl/0.1 $molL^{-1}$ KCl;
    - **WE**= (Nd,Pr)FeB+oxalate layer, 0,33 cm^2 surface area
  - 0.1 $molL^{-1}$ NaCl electrolyte
- scanning vibrating electrode technique (SVET) (Workstation Model 370 – PAR)
  - 0.001 $molL^{-1}$ NaCl electrolyte

Results

E vs. t during oxalate layers formation

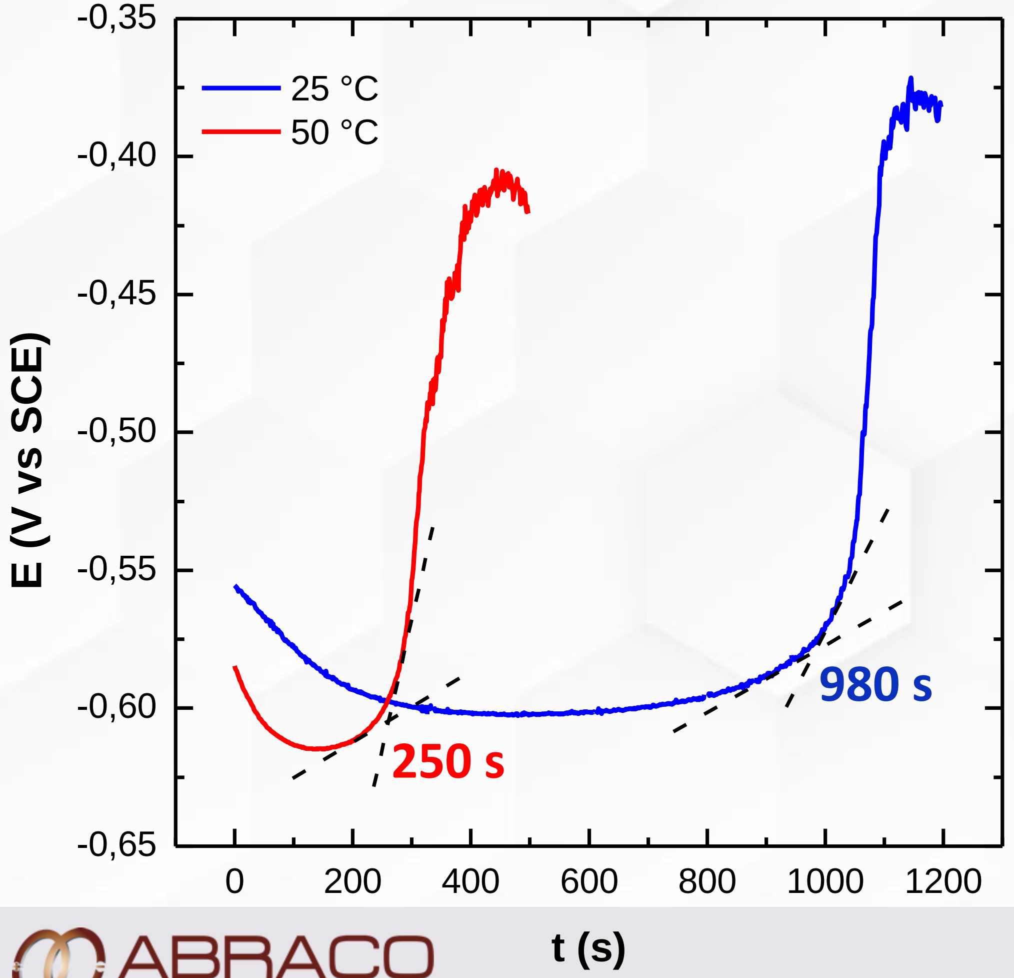


$$2Nd + 3H_2C_2O_4 \rightarrow Nd_2(C_2O_4)_3 + 3H_2$$

$$2Pr + 3H_2C_2O_4 \rightarrow Pr_2(C_2O_4)_3 + 3H_2$$

Results

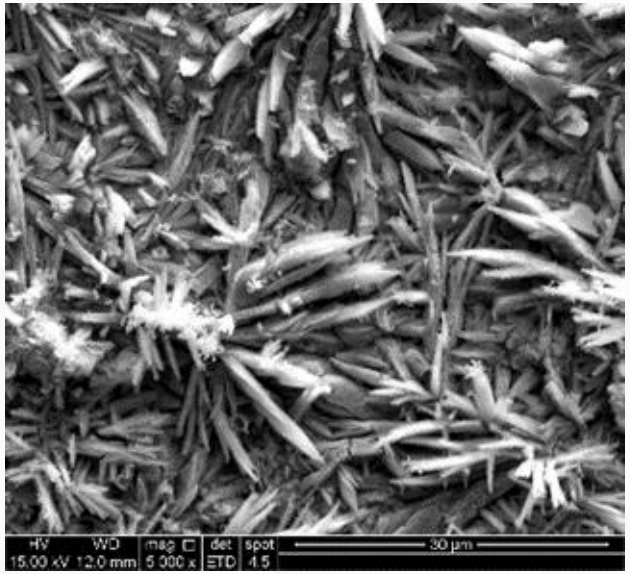
E vs. t during oxalate layers formation



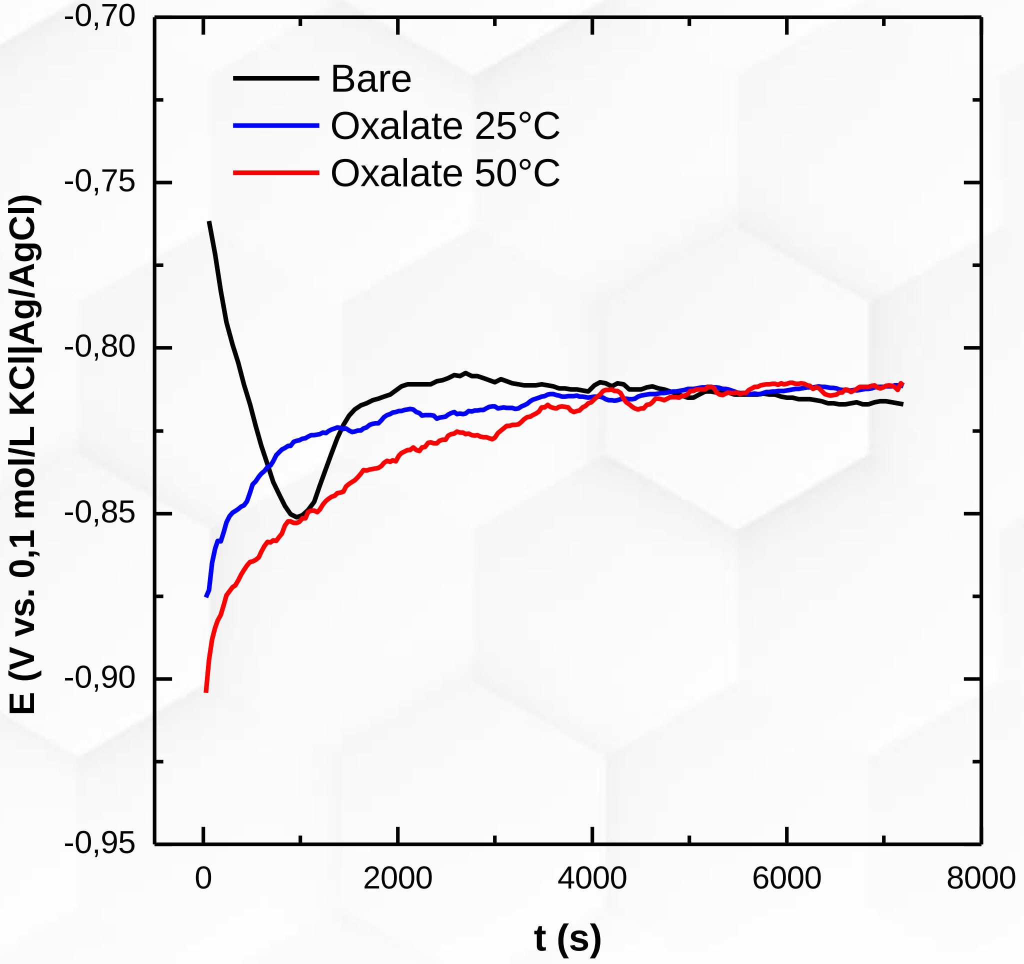
SEM Images

Oxalate layer at 25°C

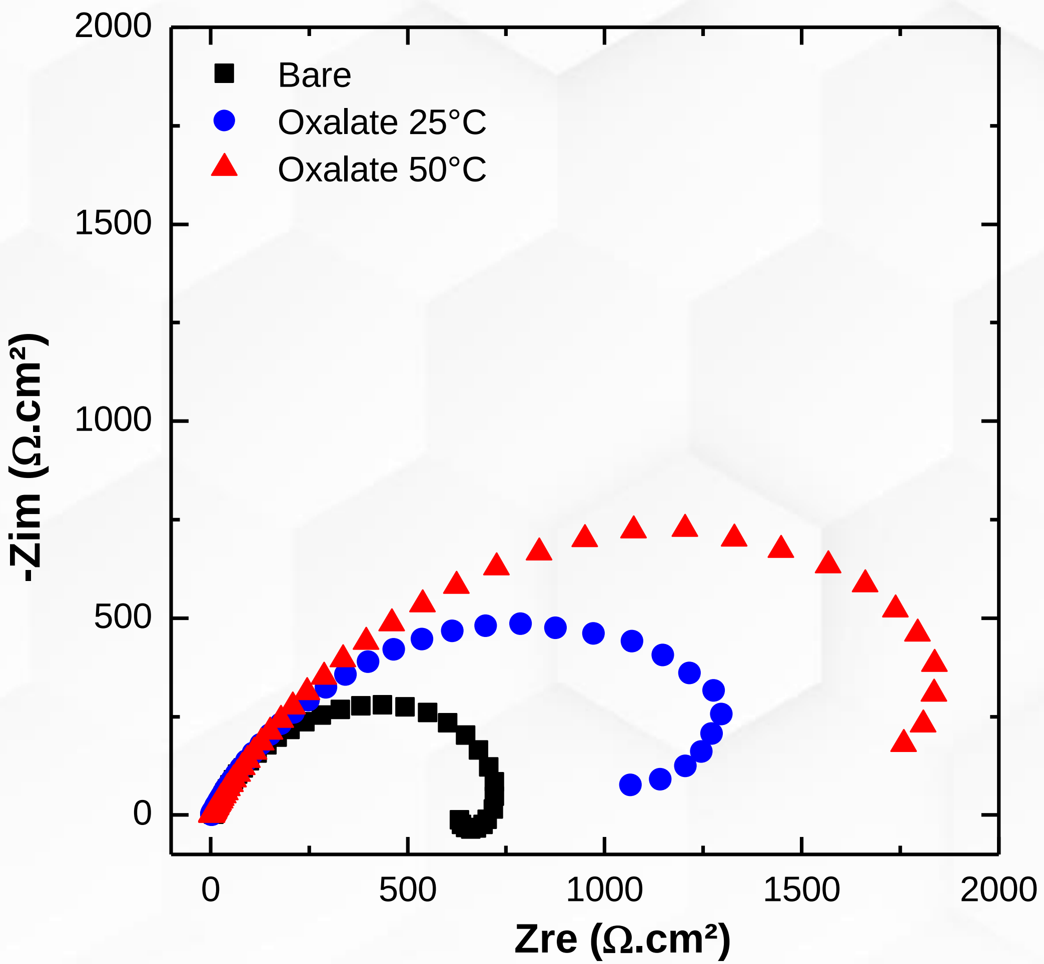
Oxalate layer at 50°C

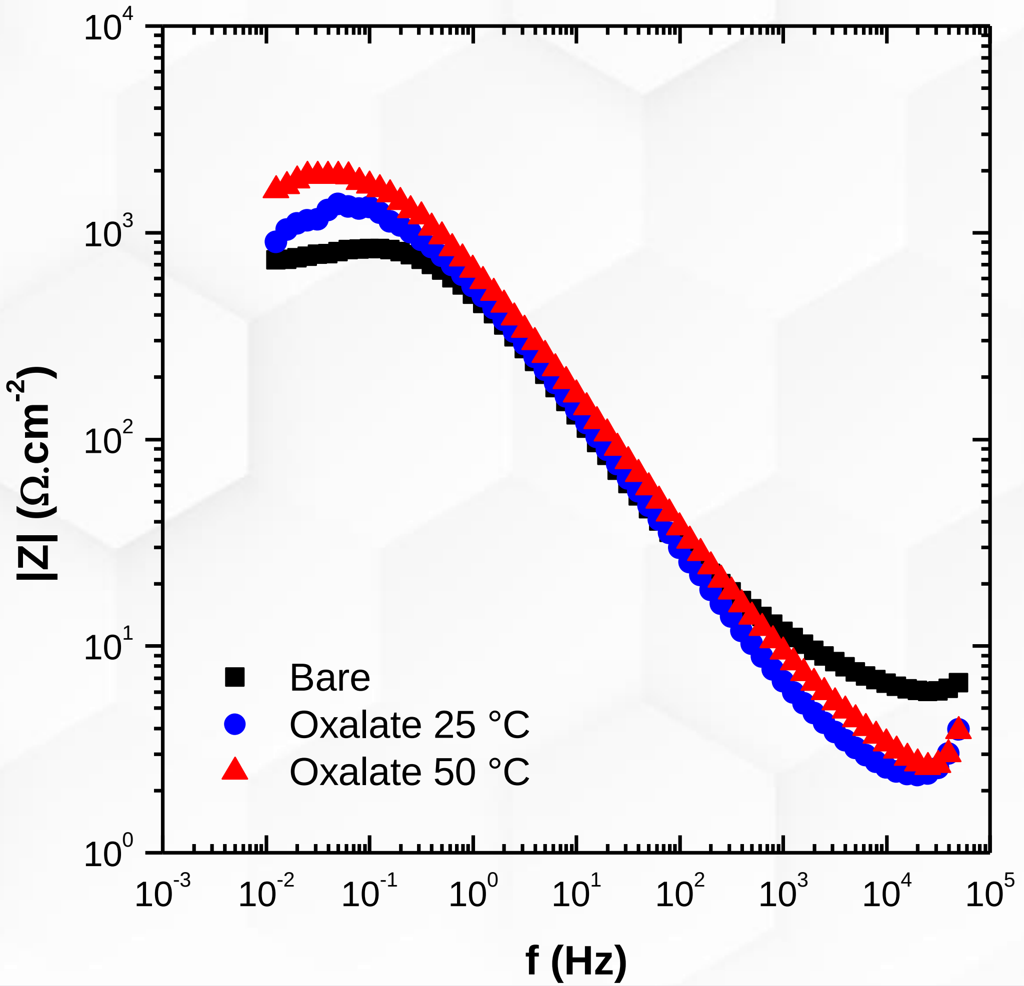


Results – OCP before EIS

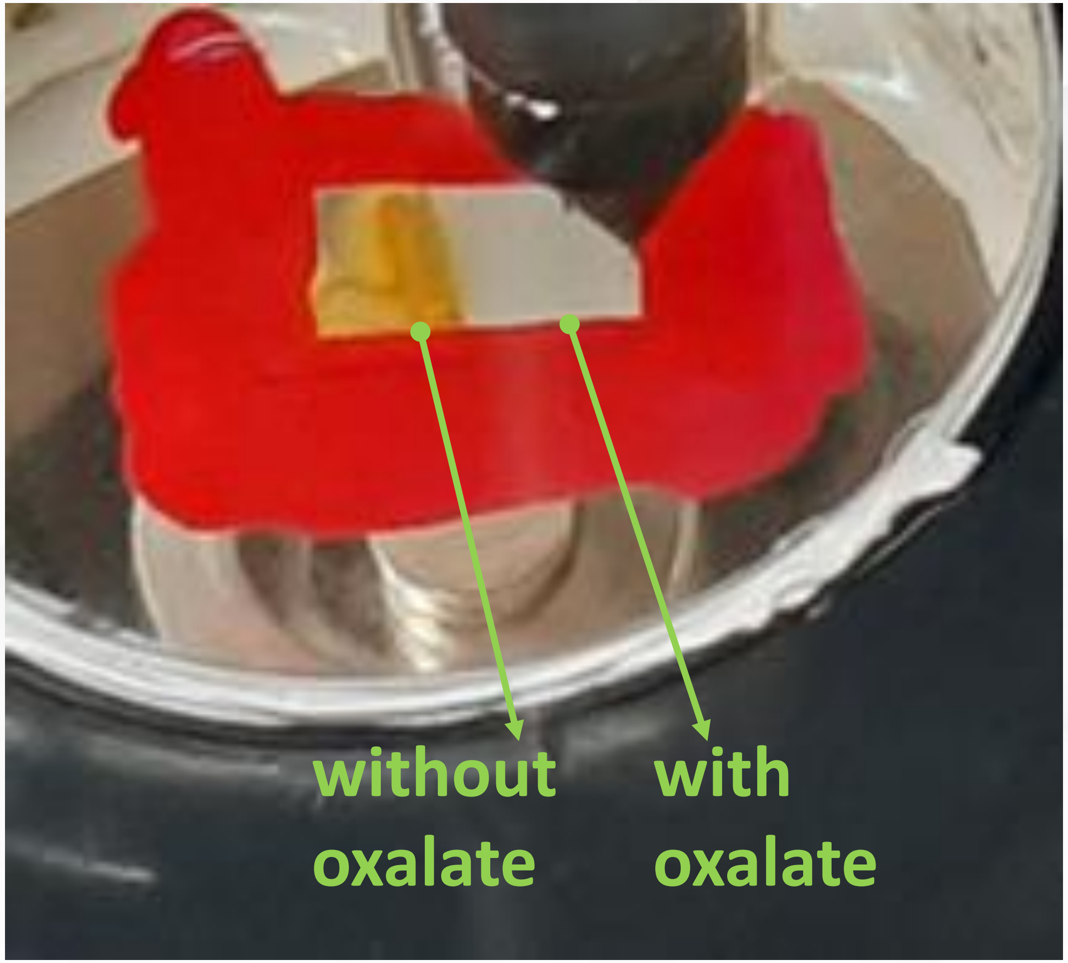


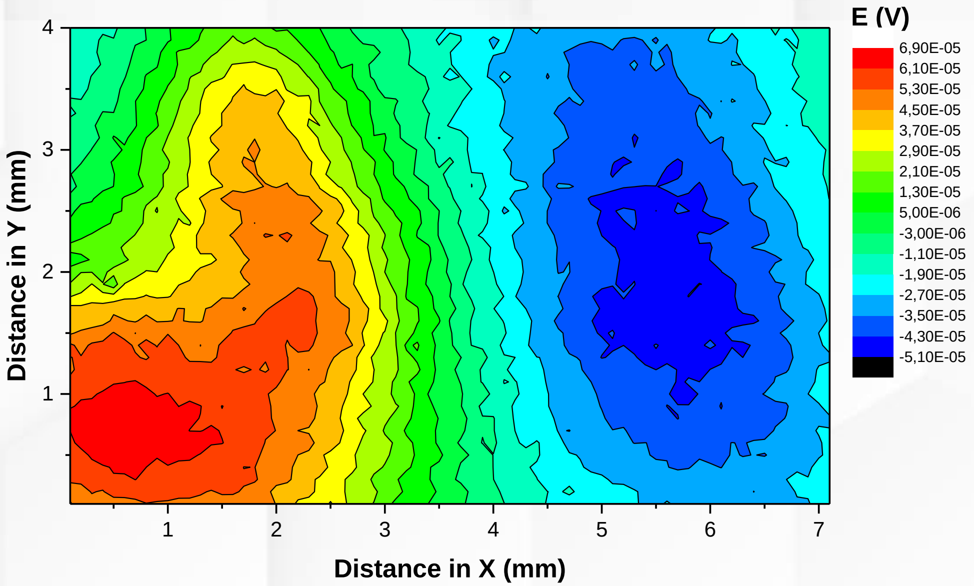
Results - EIS

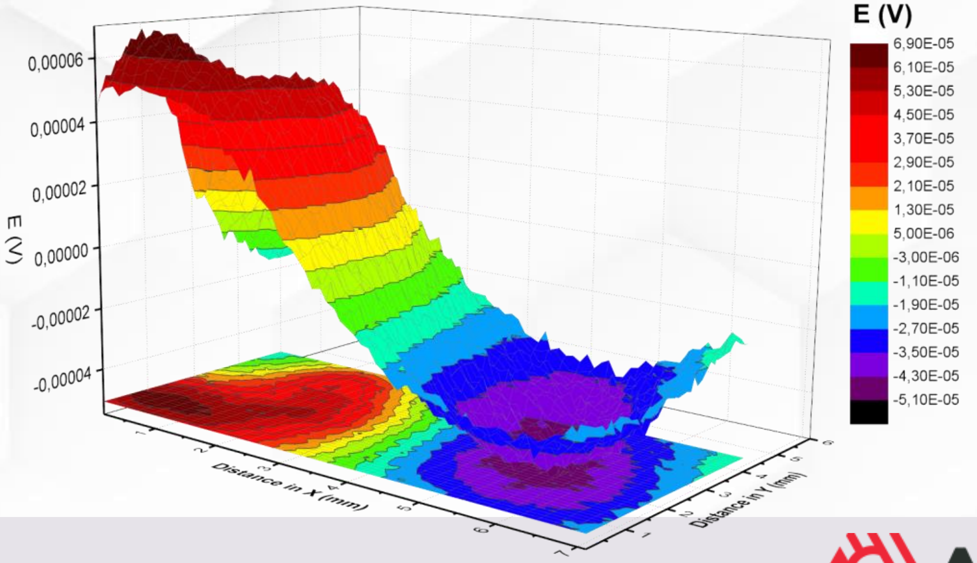

Bare
Oxalate 25°C
Oxalate 50°C
-Zim (Ω.cm²)
Zre (Ω.cm²)
0
500
1000
1500
2000


Bare
Oxalate 25 °C
Oxalate 50 °C
|Z| (Ω.cm⁻²)
f (Hz)
10^0
10^1
10^2
10^3
10^4
10^-3
10^-2
10^-1
10^5

Results - SVET







Conclusion

A needle-shaped crystal oxalate layer was obtained that adhered strongly to the (Nd,Pr)FeB surface was obtained

Increasing the temperature accelerated the formation of the layers, and this process showed practically **no sludge** formation, which could be an environmentally friendly option for temporary corrosion protection of the (Nd,Pr)FeB magnets

THANK YOU!!!

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