# Temperature Effects on Phase Evolution and Particle Morphology in Ce-Rich Commercial NdFeB Magnets During High-Pressure Hydrogen Decrepitation

BILGIN Salih[1], ARIK M. Nasuh[2], SARITAS Senem[1] , ÇAKIR Öznur[1,3]

1. Turkish Energy, Nuclear and Mineral Research Agency (TENMAK), Rare Earth Elements Research Institute (NATEN), Ankara 06980, Turkey

2. Turkish Energy, Nuclear and Mineral Research Agency (TENMAK), Boron Research Institute (BOREN), Ankara, Türkiye

3. Department of Metallurgical and Materials Engineering, Yildiz Technical University, 34220, Istanbul, Türkiye

## AIM OF THE STUDY

NdFeB magnets are essential to clean energy and advanced technological applications due to their superior magnetic properties. However, the availability of rare earth elements is susceptible to geopolitical and environmental challenges. The recycling of NdFeB permanent magnets from secondary sources represents a strategic approach to mitigate this supply risks. Among the available recycling techniques, Hydrogen Decrepitation (HD) has demonstrated significant potential for the efficient recovery of these elements from commercial magnets. This study investigates the effect of temperature during high pressure hydrogen decrepitation on the structural, microstructural, and compositional evolution of a commercial NdFeB magnet with relatively high Ce content, which is confirmed by inductively coupled plasma (ICP-OES) analysis provided baseline compositional data for the starting material. Experiments were performed at 9 bar hydrogen pressure under constant conditions of time, with three processing temperatures investigated (25 °C, 50 °C, and 75 °C). Scanning electron microscopy (SEM) of the initial magnet illustrated Ce-rich phases, while X-ray diffraction (XRD) confirmed the presence of $CeFe_2$. According to XRD results, main $RE_2Fe_{14}B$ phase transformed $RE_2Fe_{14}BH_x$ hydride phase after hydrogenation process. However, the $CeFe_2$ peaks disappeared, indicating amorph phase transformation. In addition to hydride phase and $CeFe_2$ phase, the presence $Nd_2O_3$ phase was detected. LECO elemental analysis was performed to quantify the amount of the oxygen and hydrogen inside hydrogenated powders. These results confirm the presence of oxide and hydrides with different amount. The results demonstrated that powders processed at 75 °C retained higher oxygen levels compared to those treated at 25 °C and 50 °C. This behaviour is attributed to less efficient hydrogen desorption and increased surface oxidation caused by extensive fragmentation. Furthermore, higher processing temperatures produced more agglomerated morphologies with limited cracking, lowering surface reactivity. Overall, the study highlights the effect of temperature on particle morphology, particle fragmentation, and oxidation behaviour during HD. These insights provide valuable guidance for the optimization of NdFeB magnet recycling and reprocessing strategies..

## METHODS

Hydrogenation Reaction:

$$Nd_2Fe_{14}B + xH_2 \longrightarrow Nd_2Fe_{14}BH_x$$

$$\text{Nd-rich Phase} + H_2 \longrightarrow NdH_2 + \text{Cracking}$$

**Table 1.** ICP-OES results for initial sample

| | Nd | Fe | Ce | B | Pr | Al | Dy | Ho | Ti | Other |
|---|---|---|---|---|---|---|---|---|---|---|
| **Initial Sample** | 13.08 | 68.32 | 11.42 | 0.85 | 3.30 | 0.59 | 0.62 | 0.25 | 0.20 | 0.06 |

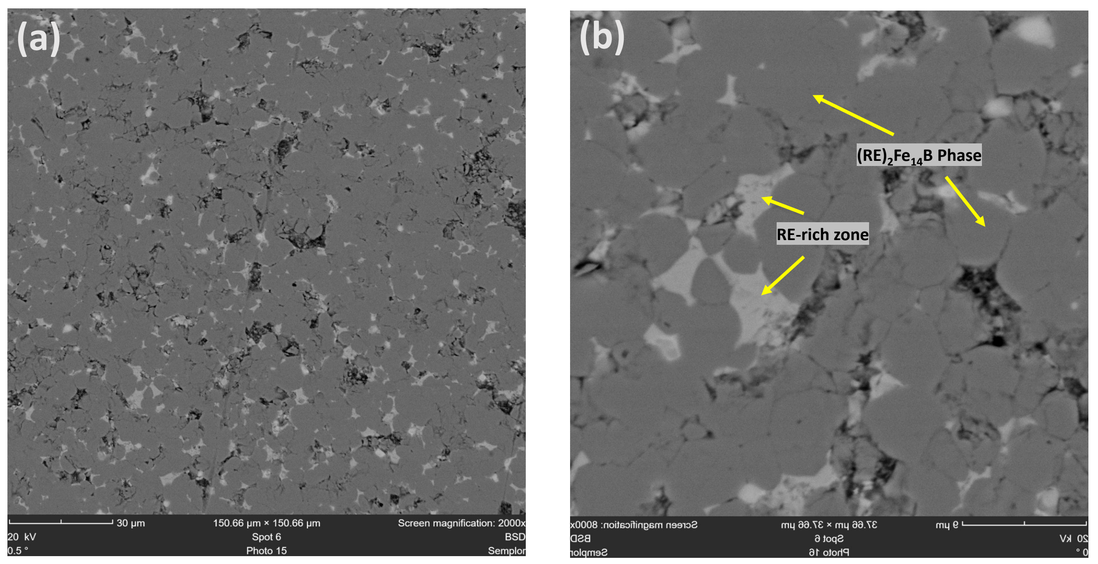



**Figure 1.** SEM images for initial sample (a) x2000 (b) x8000

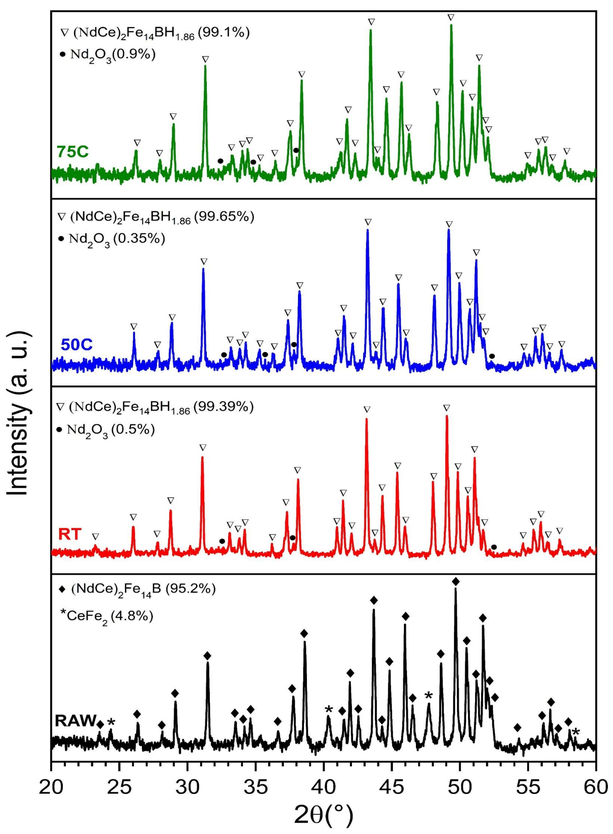



**Figure 2**. XRD patterns of the commercial NdFeB magnet (Raw) and hydrogenated powders processed at different temperatures (HD-RT, HD-50, HD-75)

## RESULTS AND DISCUSSION

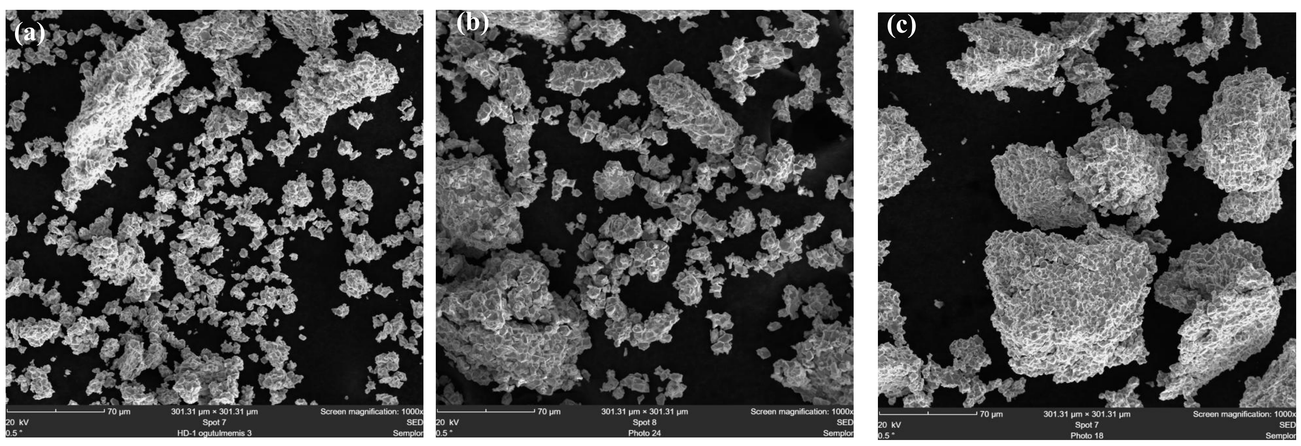



**Figure 3.** XRD patterns of the hydrogenated powders processed at different temperatures under 9.5 bar hydrogen pressure. (a) HD-RT, (b)HD-50, (c) HD-75)

**Table 2.** Hydrogen and oxygen content table for raw magnet and treated samples

| | Oxygen (wt.%) | Hydrogen (wt.%) |
|---|---|---|
| **Raw Magnet** | 0.250 | 0.02 |
| **HD_RT** | 0.368 | 0.517 |
| **HD_50** | 0.347 | 0.399 |
| **HD_75** | 0.410 | 0.404 |

## CONCLUSION

In conclusion, the processing temperature during high-pressure hydrogen decrepitation was found to strongly influence phase stability, particle morphology, and oxidation behavior in Ce-rich NdFeB magnets. Optimizing temperature conditions is therefore critical to controlling fragmentation and surface reactivity, offering key insights for improving recycling and reprocessing strategies for rare-earth magnets.

## ACKNOWLEDGEMENT

This work was supported by the Presidency of the Republic of Türkiye within the scope of the A2.H1.P42 Project conducted under the Rare Earth Elements Research Institute (NATEN), Turkish Energy, Nuclear and Mineral Research Agency (TENMAK)..

## REFERENCES

1. Zakotnik, M., I.R. Harris, and A.J. Williams, *Possible methods of recycling NdFeB-type sintered magnets using the HD/degassing process.* Journal of Alloys and Compounds, 2008. **450**(1-2): p. 525-531.
2. Zakotnik, M., I.R. Harris, and A.J. Williams, *Multiple recycling of NdFeB-type sintered magnets.* Journal of Alloys and Compounds, 2009. **469**(1-2): p. 314-321.
3. Burkhardt, C., et al., *An overview of Hydrogen assisted (Direct) recycling of Rare earth permanent magnets.* Journal of Magnetism and Magnetic Materials, 2023. **588**: p. 171475.
4. *Habibzadeh, A., et al., Microstructural Investigation of Discarded NdFeB Magnets After Low-Temperature Hydrogenation. Journal of Sustainable Metallurgy, 2024. 10(3): p. 1141-1155.*