

Nanocrystalline iron nitriding – in-situ XRPD analysis

Aleksander Albrecht^a, Paweł Adamski^a, Dariusz Moszyński^a

^a *Department of Inorganic Chemical Technology and Environment Engineering, West Pomeranian University of Technology in Szczecin, ul. Pułaskiego 10, 70-322 Szczecin, Poland*
Contact Author E-mail Address: aleksander.albrecht@zut.edu.pl

Iron nitrides are metastable compounds consisting of nitrogen dissolved in interstitial spaces of iron. Research on the Fe-N phase system was conducted since the 1920s and the main analytical method utilized for this purpose at that stage was XRD [1]. It was later discovered that the characteristics of bulk and nanometric iron nitrides differ considerably. The difference concerns among others both kinetic and thermodynamic parameters of nitriding reaction [2, 3].

Current knowledge on the topic requires complementation. One of the important omitted aspects is the influence of the nanocrystallite size on the thermodynamic parameters of crystalline phase changes. This study aims to describe differences between α -Fe \leftrightarrow γ' -Fe₄N \leftrightarrow ϵ -Fe₃N phase changes in various nitriding potential and several different temperatures for certain nanocrystalline iron sizes.

For this purpose commercial nanocrystalline iron with different particle sizes within the range of 25-80 nm was exposed to ammonia and hydrogen mixture in the Anton Paar XRK 900 reaction chamber mounted on Philips X'pert MPD X-ray diffractometer. *In-situ* X-ray powder diffraction (XRPD) analyses were conducted in the temperature range of 350-500°C. The apparatus worked in Bragg-Brentano geometry and a copper K α radiation source was used. Phase analysis was performed with use of the International Centre for Diffraction Data ICDD PDF 4+ database. A full-pattern fit based on the Rietveld method was conducted to calculate the weight fractions of the crystallographic phases identified in the material and their mean crystallite size. A semi-automatic procedure of Rietveld refinement included in the HighScore Plus 3.0e software by PANalytical was used. During this procedure, the following parameters were varied: scale factors, unit cell dimensions, atom coordinates, occupancy factors, peak profile shape and full width at half maximum (FWHM).

Acknowledgments

Aleksander Albrecht thanks the Polish Ministry of Science and Higher Education for support through the project “Diamentowy Grant” no. DI2018003348 funded in years 2019-2022.

References

- [1] S. Bhattacharyya, Iron Nitride Family at Reduced Dimensions: A Review of Their Synthesis Protocols and Structural and Magnetic Properties, *The Journal of Physical Chemistry C* 119 (2015) 1601-1622.
- [2] D. Moszyński, Nitriding of Nanocrystalline Iron in the Atmospheres with Variable Nitriding Potential, *The Journal of Physical Chemistry C* 118 (2014) 15440-15447.
- [3] R. Pelka, W. Arabczyk, Studies of the Kinetics of Reaction Between Iron Catalysts and Ammonia - Nitriding of Nanocrystalline Iron with Parallel Catalytic Ammonia Decomposition, *Top. Catal.* 52 (2009) 1506-1516.