Dependence of wustite based iron catalyst crystallite size on ammonia synthesis reaction analysed by *in-situ* XRPD

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Wustyt is a non-stoichiometric form of iron(II) oxide with the general formula $Fe_{1-x}O$, which is currently used as a precursor of the iron catalyst for the synthesis of ammonia. The catalyst obtained from this precursor has higher activity compared to a traditional catalyst reduced from magnetite [1]. Promoters used in the magnetite precursor may have a different role in wustite catalysts. This is due to the different valence of iron in the structure of wustite and magnetite. As a result, some promoters are more or less likely to build in the catalyst grain [2]. In this study, the influence of magnesium oxide addition on the activity and thermal stability of the catalyst was investigated. It was suspected that as a result of similar valence, magnesium ions would be more likely to build into the grain of wustite, thus stabilize the active phase of the catalyst.

Iron catalyst precursors were obtained as a result of the melting of magnetite, aluminum oxides, calcium, magnesium, potassium nitrate, and metallic iron, which acts as a magnetite reducer. The XRPD method confirm the presence of wustite phase in each precursor. Chemical composition was determined by ICP-OES method. Evolution of the phase composition of obtained precursors during reduction with hydrogen were investigated by XRPD *in-situ* method, with the use of PANalytical X'pert Pro MPD diffractometer equipped with Anton Paar XRK 900 reaction chamber. Crystallite sizes of iron were calculated using Rietveld method. Activity tests in the ammonia synthesis reaction were carried under pressure 10 MPa in the temperature 450°C.

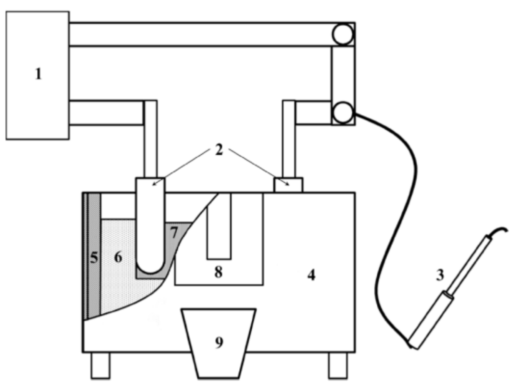

Figure 1. Scheme of the laboratory plant for fused catalysts synthesis: (1) multi-tap transformer with a control system, (2) water-cooled fusing electrodes, (3) auxiliary electrode, (4) fuse reactor, (5) (6) insulation, (7) batch, (8) casting chamber, and (9) lava pouring box.

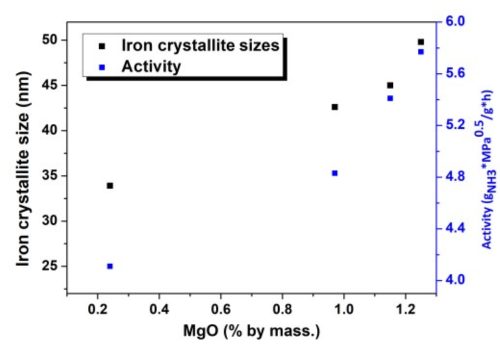

Figure 2. Differences in crystallite size and activity of catalysts promoted with magnesium oxide.

The promotion of wustite precursors with magnesium oxide contributes to the significantly increase of the crystallite sizes. The crystallite size increased by 47% comparing the catalysts with the lowest and the highest concentration of magnesium oxide. Similarly activity of obtained catalysts rate were also increased. The activity increased by over 40% comparing the catalysts with the lowest magnesium oxide concentration to the catalyst with the highest concentration of this promoter.

[1] Hua-Zhang, L., Xiao-Nian, L. & Zhang-Neng, H. (1996). *Appl. Catal. A General* **142**, 209.

[2] Lendzion-Bieluń, Z. & Arabczyk, W. (2001). *Appl. Catal. A General* **207**, 37.

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