

PHASE STABILITY STUDY OF $\text{Ni}_x\text{Mg}_{1-x}\text{Al}_2\text{O}_4$ VIA XRD AND COMPLEMENTARY TECHNIQUES

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Treatment of $\text{Ni}_x\text{Mg}_{1-x}\text{Al}_2\text{O}_4$ with H_2 results in the progressive reduction of Ni with increasing temperature. The formation of a charge compensating oxygen vacancy accompanies the transition of Ni^{2+} to Ni^0 . For compositions $x \leq 0.5$, it has been shown that the vacancy rich spinel structure remains stable with all Ni^{2+} removed, up to at least 1000 °C. For compositions $x = 0.75$ and $x = 1$, the spinel phase becomes unstable when approaching full reduction, and transitions to a θ or α alumina phase. The addition of a small amount of ZrO_2 (2.5 weight %) has been found to suppress this phase transition and maintain the spinel structure. The nature of the system encourages the use of complementary techniques such as XPS and in-situ thermogravimetric analysis under flowing hydrogen. Refined fractional occupancies of Ni on the tetrahedral and octahedral sites, and Ni metal phase fraction allows for the deconvolution of the complex Ni2p electron emission spectra. In-situ TGA gives confirmation of the refined Ni metal phase fraction by monitoring a mass change due to charge compensating oxygen vacancies. A detailed picture of structural changes, vacancy formation and phase development as a function of composition and reduction temperature will be demonstrated by Rietveld refinement of X-ray diffraction data, XPS, TGA, and in-situ high temperature X-ray diffraction under flowing hydrogen.