The Synthesis and Characterization of the Ni-B Catalyst

The nickel boron catalyst was synthesized by the reduction of nickel acetate with sodium borohydride in aqueous solution. The reactions were carried out in a N_2 atmosphere using standard vacuum-line techniques. For a typical preparation, a solution of 452 mg $Ni(CH_3COO)_2 \cdot 4H_2O$ was reacted with 160 mg $NaBH_4$ at 80 °C under vigorous stirring. The solution was turned into black immediately after mixing the two reagents, indicating the formation of the nickel boron nanoparticles. The product precipitated out of the solution after about 20 h of stand-still at room temperature.

HRTEM observations were performed with a JEOL JEM-3011 spectrometer operated at 300 kV. Images were recorded at magnifications up to 800,000x. The TEM samples were prepared by using 400 mesh Cu grids with holey carbon supporting film. For the XRD studies, the precipitated sample was repeatedly washed with distilled water and then dried under vacuum. The XRD spectra were recorded by a step size of 0.05° using the Philip Xpert spectrometer (Cu $K\alpha_1$, λ = 1.5418 Å). The annealing treatment was carried out at 600 °C in argon (Ar) atmosphere for 10 minutes. For the nano-beam electron diffractions, the camera was calibrated by the diffraction patterns of colloidal gold, and care was taken to ensure a constant objective lens excitation, to ensure that there was no change in the focal length between different diffraction patterns. In the EDS analyses, given the relatively low detection rates at the low energy for the detection of boron, a standard deviation of around 10% was probably the best accuracy that could be achieved. With the actual specimens, due to the superposition of the x-ray peaks from the amorphous coating of the particles and the supporting carbon film, the stronger carbon K emission line partly masked the boron K emission line. Therefore, the background subtraction was necessary. Fortunately, the spectrometer resolution at the low energies was approximately the same order of magnitude as the 92 eV separation of boron and carbon K emission lines, a correction was therefore possible. This was carried out by recording a spectrum from a region of the specimen, then recording a second spectrum from an adjacent area of the carbon film, such that the height of the carbon K emission peak was the same in both. Subtraction of the second from the first produced a corrected spectrum.