Synthesis and Characterization of Nickel and Nickel Hydroxide Nanopowders

Catherine Huang and Teresa D. Golden

Abstract

- Nickel, Ni, and nickel hydroxide, $Ni(OH)_2$, powders with nanosized particles were synthesized using a chemical method.

- The polymer Poly(vinyl pyrrolidone) or PVP was added to solutions of Ni for purposes of particle protection.

- X-Ray analysis revealed the size of $Ni(OH)_2$ particles to be approximately 19 nanometers in diameter and the sizes of Ni particles to be between 7 and 12 nm.

Introduction

- Metal nanopowders are highly useful because they possess unique chemical and physical properties and have possible applications in optical and magnetic devices.
- Such powders are generally produced through the chemical reduction of metal ions using a reductant or are produced through electrochemically depositing metal particles as films.
- Research on metal nanopowders focuses on the prevention of agglomeration and oxidization of the particles [1], synthesizing powders with small particle sizes [2, 3], and quantity synthesis with a focus on industrializing of the process [4].

Introduction – Nickel Hydroxide

- $Ni(OH)_2$ is used as the cathode of rechargeable Nickel/Metal Hydride (NiMH) Cell batteries and is also used to produce NiO, which is commonly used in glass and ceramic industries. [5]

- Powders of $Ni(OH)_2$ were synthesized using a simple and efficient chemical method.

- The powders were characterized using X-Ray analysis and the crystallite sizes determined using X-Ray peak broadening analysis, as X-Ray peaks become broader due to the effect of small crystallite sizes.

- A Williamson-Hall plot was used to estimate particle size (Fig. 2).

Nickel Hydroxide Powder Preparation

- $Ni(OH)_2$ powders were produced by the chemical reaction between nickel (II) chloride, $NiCl_2$ sodium hydroxide, NaOH, and the reductant hydrazine hydrate, N_2H_4.

- $NiCl_2$ was dissolved in distilled water was mixed with absolute ethanol and formed a green solution. In a separate beaker, N_2H_4 and NaOH were thoroughly combined.

- The solution of N_2H_4 and NaOH was then slowly added to the dissolved $NiCl_2$, resulting in a cloudy green solution. Precipitate formed as soon as the two solutions were mixed.

- The precipitate was was filtered, washed, and dried using a standard funnel arrangement, then X-Rayed (Fig. 1).

X-Ray Diffraction Pattern of Nickel Hydroxide Powder

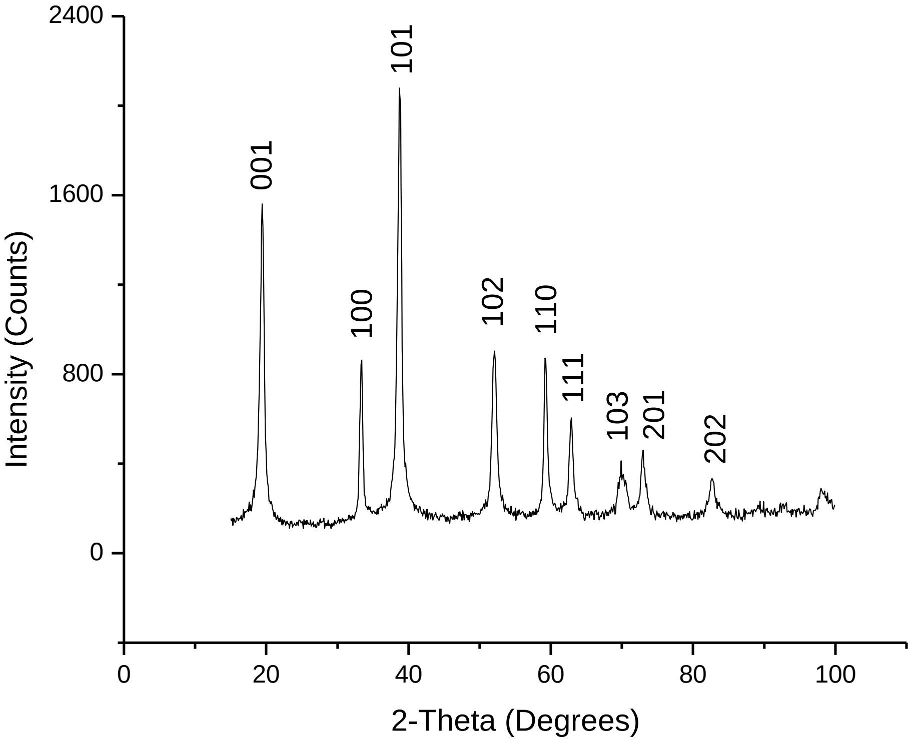

Figure 1. XRD pattern of $Ni(OH)_2$ powder

Peak (θ)	001 @ 19.47°	100 @ 33.62°	101 @ 38.85°	102 @ 52.29°
FWHM (deg)	0.78	0.69	0.80	1.16
FWHM (rad) $=B_0$	0.014	0.012	0.014	0.020
$B_0\cos\theta$	0.014	0.0115	0.013	0.018
$\sin\theta$	0.169	0.289	0.333	0.441

Table 1. FWHM calculation of $Ni(OH)_2$ powder

Williamson-Hall Plot of Nickel Hydroxide Powder

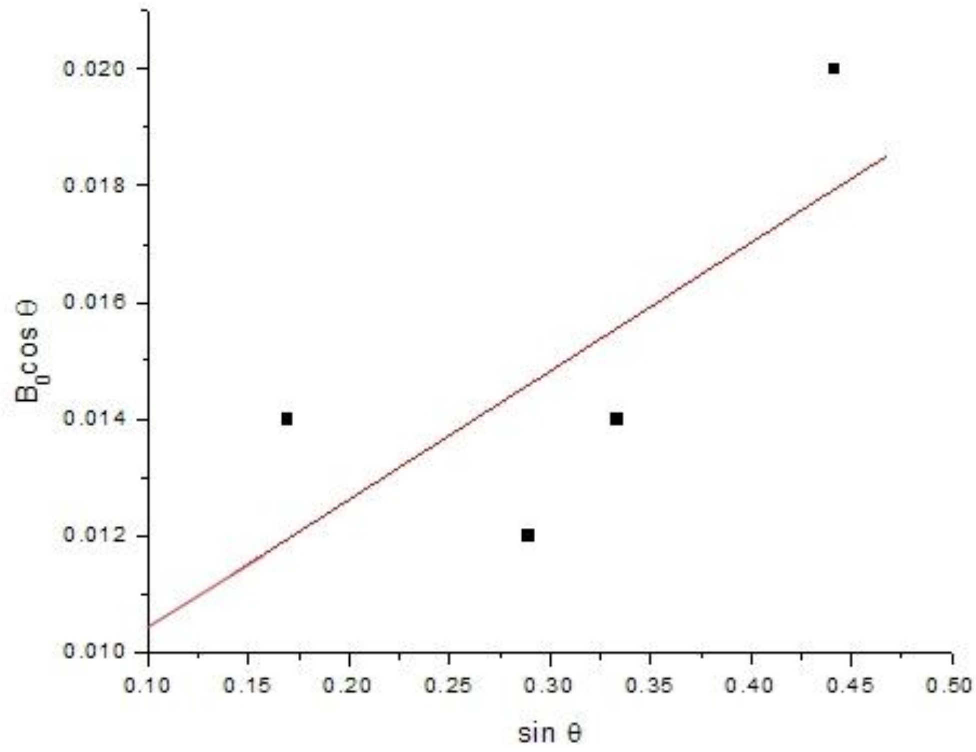

Figure 2. Williamson-Hall plot of $Ni(OH)_2$ powder

Y-intercept of W-H plot (rad)	0.0105
k	1.0
λ (nm)	0.154056
*k*λ/Y-intercept = ***L* (particle size)**	**19 nm**

Table 2. Particle size of $Ni(OH)_2$

Introduction - Nickel

- The many chemical and physical properties of nanosize nickel powder include a extremely high surface area per mass, conductive and magnetic properties, and control over the scattering of light.
- Nanosize nickel powders can be commercially utilized today as chemical catalysts, in fuel and solar cells, in paints and polymers, and in optical equipment.
- Stabilizers such as Poly(vinyl pyrrolidone) (PVP) are often used during synthesis for particle protection and are able to decrease particle size [6].

Introduction - Nickel (cont’d)

- Ni powders were synthesized using a chemical method.
- Different amounts of powder can be produced by varying the concentrations of the chemicals.
- PVP was added to prevent agglomeration of the particles and to promote small particle sizes.
- X-Ray analysis characterized the powders and the Williamson-Hall plot provided an estimate of the particle sizes (Fig 3, 4).

Nickel Nanopowder Preparation

- Preparation of the solution is as follows: 0.5 g of $NiCl_2$ was dissolved in 60 mL of distilled water for Ni^{2+} ions. 0.1 g of PVP was dissolved in the Ni solution.
- In a separate beaker, approximately 1.0 g of a strong base, NaOH, was dissolved in 20 mL distilled water.
- 20 mL of the reductant, $N_2H_{4,}$ was added to the beaker containing NaOH; the resulting solution was then added to the solution of $NiCl_2$.
- The solution turned a royal blue color and was allowed to sit overnight.

Nickel Nanopowder Preparation (cont'd)

- During post-processing using sonication or heating (Table 3), the solution became grey, a black precipitate began forming along the sides and bottom of the beaker, and a shiny silver substance coated the sides of the beaker.
- The solutions were left to precipitate overnight.
- The precipitate was filtered, washed, and dried using a standard funnel arrangement, and X-Rayed.

X-Ray Diffraction Pattern of Nickel Powder

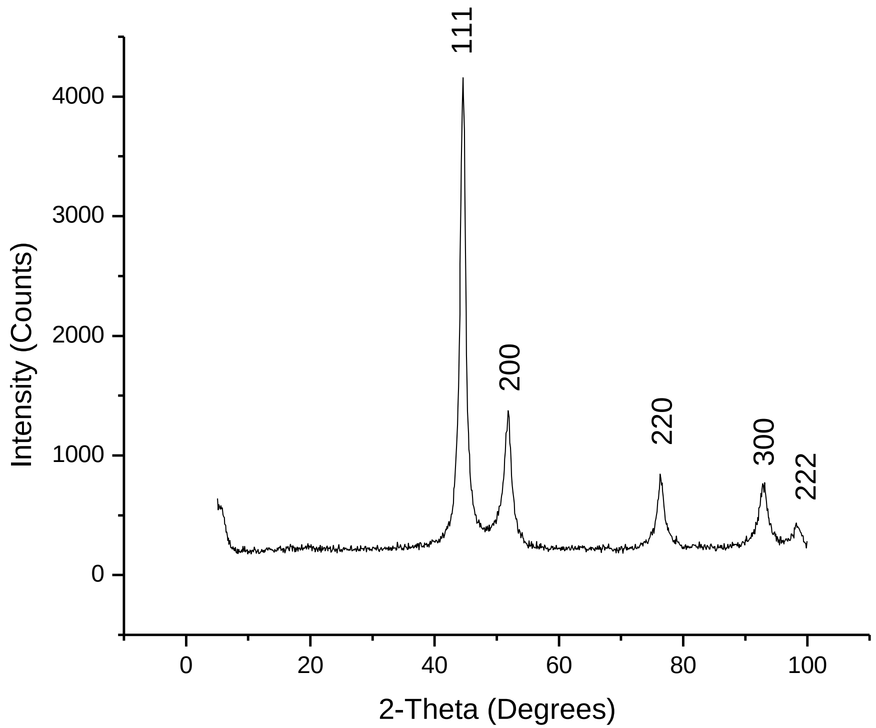

Figure 3. XRD Pattern of Ni Powder stabilized by PVP

Peak (θ)	111 @ 44.57°	200 @ 51.81°	220 @ 76.41°	311 @ 92.92°
FWHM (deg)	1.12	2.81	3.90	5.73
FWHM (rad) = B_0	0.020	0.049	0.068	0.100
$B_0\cos\theta$	0.0185	0.0441	0.0534	0.0689
B_1†	0.0073	0.0073	0.0085	0.0103
Br ($Br^2 = B_0^2 - B_1^2$)	0.019	0.048	0.067	0.11
Brcosθ	0.0176	0.0432	0.0526	0.0689
sinθ	0.379	0.437	0.618	0.725

Table 3. FWHM of PVP-protected nickel particles

† B_1 is the correction for instrumental broadening

Williamson-Hall Plot of Nickel Powder

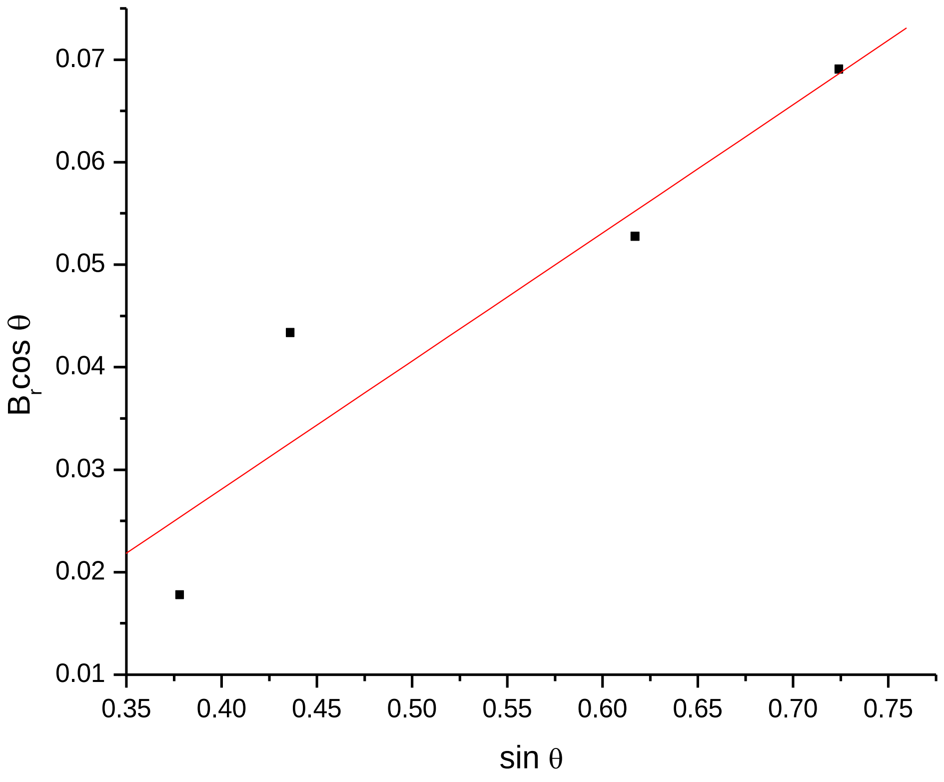

Figure 4. Williamson-Hall plot of PVP-protected Ni particles

Y-intercept of W-H plot ($k\lambda/L$)	0.022
k	1.0
λ (nm)	0.154056
$k\lambda$/Y-intercept = ***L* (particle size)**	**7 nm**

Table 4. Particle size of PVP-protected Ni particles

Production Methods

Ni Procedures	Trial 1	Trial 2	Trial 3
Chemical Conditions	NiCl2: 0.499 g H2O: 60 mL N2H4: 20 mL NaOH: 20 mL	NiCl2: 0.499 g H2O: 60 mL N2H4: 20 mL NaOH: 20 mL	NiCl2: 0.499 g H2O: 60 mL N2H4: 20 mL NaOH: 20 mL PVP: 0.5 g
Post-processing method	Sonication in a bath sonicator	Low heat	Sonication in a bath sonicator
Resulting particle size	**12 nm**	**19 nm**	**7 nm**

Table 5. Comparison of three different methods of Ni powder synthesis

Discussion

- A Williamson-Hall plot indicated that PVP-protected particles had an estimated size of 7 nm, compared to an estimate of 12 nm for non-PVP protected Ni particles.

- A possible formula for the formation of Ni:

  $NiCl_2 \rightarrow Ni^{2+} + 2Cl^-$

  $Ni^{2+} + 6N_2H_4 \rightarrow Ni(N_2H_4)_6^{2+}$

  $Ni(N_2H_4)_6^{2+} + 2OH^- \rightarrow Ni + N_2 + 5N_2H_4 + 2H_2O$

- The procedures for $Ni(OH)_2$ and Ni are nearly identical, except for the concentrations of the chemicals. One possible explanation for the different results is an insufficient amount of NaOH in the solution that produced $Ni(OH)_2$ (Fig. 5).

Discussion (cont'd)

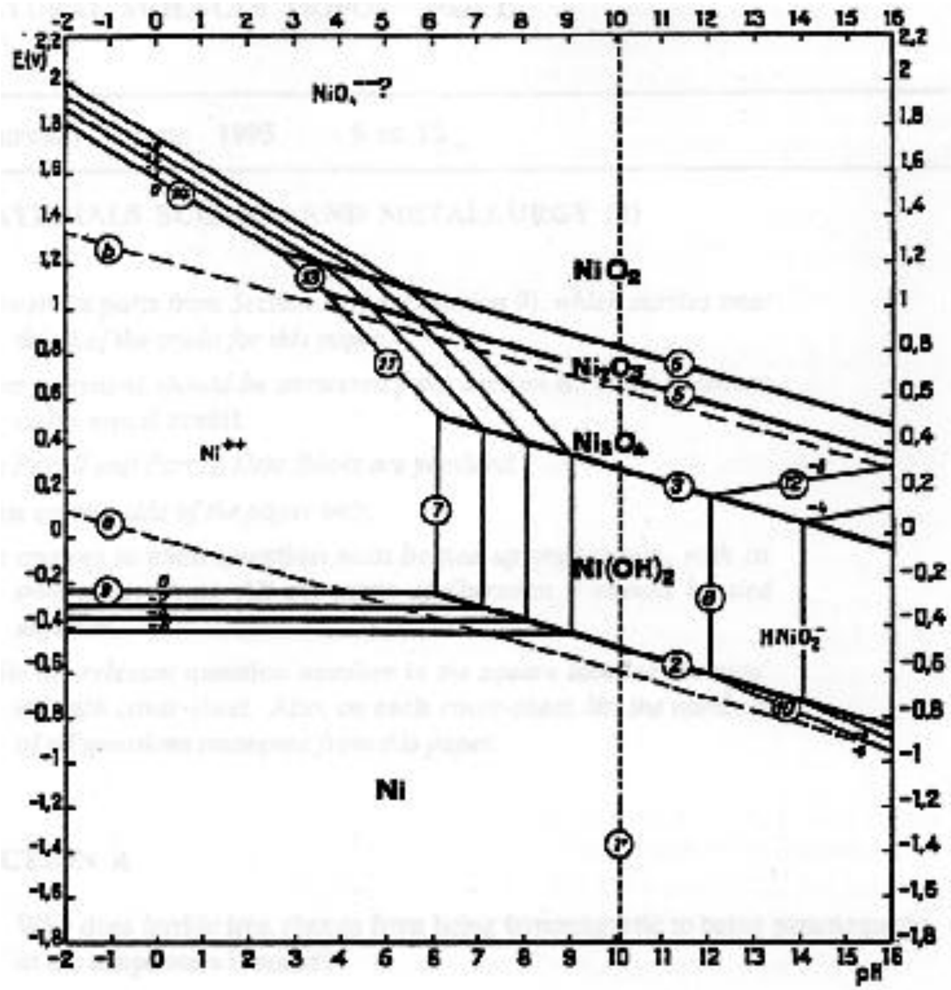

Figure 5. The Pourbaix diagram for Ni

Conclusions

- This chemical method for synthesis of Ni nanopowders can be slightly modified to produce $Ni(OH)_2$ powders.
- The addition of PVP prevents agglomeration of particles and promotes smaller particle sizes.
- The most notable features of this method are the simple operation, high yield, and small particle sizes.
- Future research on this method include synthesizing nanopowders of various metals and investigating the applications of the metal nanopowders in optical equipment.

Acknowledgements

- TAMS Summer Research Scholarship
- Dr. Teresa D. Golden
- University of North Texas Chemistry Department

References

- (1) H. Feng, Z. Zhengyi, X. Yaofu, W. Wuxiang, H. Yafang, W. J. Run, *Mater*. *Proc*. *Tech*., **2003**, 137, 201-203
- (2) L. Rodriguez-Sanchez, M. C. Blanco, M. A. Lopez-Quintela, *J*. *Phys*. *Chem*. *B*., **2000,** 104, 9683-9688
- (3) M. T. Reetz, M. Winter, R. Breinbauer, T. Thurn-Albrecht, W. Vogel, *Chem*. *Eur*. *J*., **2001**, 7, 1084-1094
- (4) W. Yu, H. Liu, *Chem*, *Mater*., **1998**, 10, 1205-1207
- (5) J. M. Boyer, B. Repetti, R. Garrigos, M. Meyer, A. Bee, *J. Meta. Nano. Mater.,* **2004**, 22, 1-11
- (6) N. Aihara, K. Torigoe, K. Esumi, *Langmuir*, **1998**, 14, 4945-4949