Preparation of Nano-sized Nickel Metal Particles by Microwave Irradiation

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Nano-sized nickel metal particles having narrow size-distribution were prepared through reduction of $Ni(OH)_2$ with ethylene glycol under microwave irradiation. The average particle size was controlled between 5-8 nm by changing the irradiation time and irradiation power.

Small metal particles having narrow size-distribution have attracted attention due to their potential applications as functional materials, *i.e.*, catalysts, magnetic materials, conducting inks, and ferrofluids.[1-3] Various preparation and stabilization methods of nano-sized metal particles, metal oxide particles, and metal sulfide particles have been reported.

In contrast to conventional heating by convection and agitation, microwave irradiation induces direct heating of substances through interaction of the dipole moment and/or ions with the electric field, resulting in homogeneous and rapid chemical reactions. Microwave heating has recently begun to attract the attention of chemists as a new method for synthesizing chemical compounds[4-7] or even for reducing metal salts to bulk metal.[8]

In the present study, microwave irradiation was used to prepare nano-sized particles of Ni metal through the reduction of nickel hydroxide in ethylene glycol in the presence of a platinum salt and nano-sized nickel metal particles much smaller than those obtainable through the conventional heating method were successfully obtained.

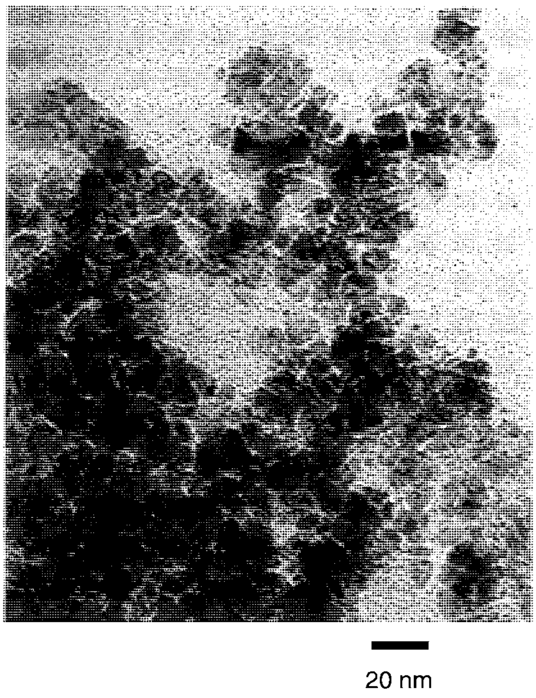

Figure 1a. TEM image of Ni nanoparticles prepared by microwave irradiation (magnification: 5 x 10^5).

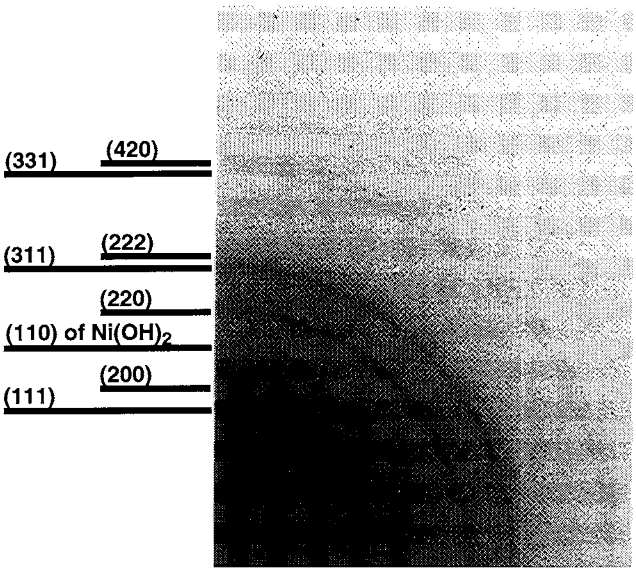

Figure 1b. Electron diffraction pattern of the sample used for the measurement of the particles in Figure 1a.

A dispersion solution of nickel hydroxide (nacalai tesque, light-green powder, 3.0 x 10^{-4} mol) in ethylene glycol (2 ml) containing polyvinylpyrrolidone (PVP, average molecular weight: 40000, 0.133 g) and hydrogen tetrachloroplatinate(II) (1.6 x 10^{-6} mol) was placed in a glass vial. Hydrogen tetrachloroplatinate(II) and PVP should act as a precursor of Pt catalyst for nucleation of Ni metal and a stabilizer of small Ni metal particles, respectively, as suggested in the literature.[9] When $Ni(OH)_2$ was introduced into the solution and stirred, a light-green powder of $Ni(OH)_2$ was dispersed therein. When the dispersion solution was irradiated by microwave (2.45 GHz) at 200 W, the temperature of the solution was raised to 423 K in 5 min and the color of the solution changed to gray. Droplets of the solution were placed onto a carbon-coated copper mesh and dried at about 373 K under a vacuum for observation via transmission electron microscopy (TEM). The TEM image (Figure1a) showed the formation of aggregated cubic nano-sized particles. The primary size distribution determined via TEM was centered at 7 nm and ranged between 4 and 12 nm (Figure 2). Analysis of the diffraction ring pattern indicated that the particles consisted of metal nickel and traces of remaining $Ni(OH)_2$ (Figure 1b). Observation of the solution sample after irradiation

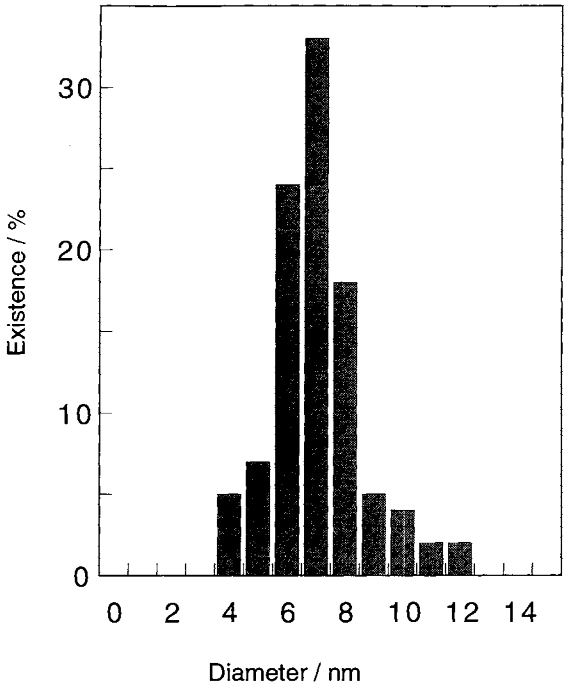

Figure 2. Size distribution of Ni nanoparticles determined by TEM. The sizes were measured for 100 particles selected at random.

via dynamic laser scattering revealed a size and distribution almost identical to those obtained in the TEM observation, indicating that the particles were mono-dispersed in the solution. Centrifugation (20000 G) did not cause sedimentation of the Ni metal particles in the solution.

The preparation of Ni metal particles using ethylene glycol and polyvinylpyrroridone was performed in the presence of hydrogen tetrachloroplatinate(II) previously under conventional heating by Hegde et al.[9] The particle size of the Ni nanoparticles prepared in the present work was ten times smaller than those reported by Hegde. We also reproduced Hegde's experiments and confirmed the clear difference in the particle sizes between the microwave irradiation and the conventional heating. It should be emphasized that the Ni particles formed via the microwave method are cube-shaped, whereas Hegde reported that the Ni particles prepared by the conventional heating method were spherical.[9] Thus, the growth process of Ni particles for the microwave-irradiating method would be different from that for the conventional method. Unusual phenomena, such as superheating[4] and nonthermal acceleration of chemical reaction[10] were reported to be observed under microwave irradiation. Microwave irradiation may cause different nucleation and growth of Ni metal particles due to the high dielectric loss properties of $Ni(OH)_2$ and ethylene glycol.

The growth of Ni particles can be controlled by the irradiation power and time. Further research on a new method for preparing inorganic nano-sized materials is currently in progress.

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