

Ultrafine Co Powder Prepared by Hydrogen Reduction of $\text{Co}(\text{OH})_2$ Suspension in Ethylene glycol

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Introduction

One of the tendencies in materials science has been to prepare ultrafine metal powders with uniform size and shape. Great progress in this field has been made by Matijevic (forced hydrolysis) and Figlarz (polyol process). The powders made in their methods had good morphology and distribution, but the reaction time was too long (from several hours to several days), and polyols were not used again.

Over recent years, we have done some research work on preparing ultrafine metal powders (Ni, Co, Ag) from their hydroxide slurries by H_2 reduction. In the present work a new process combining the advantages of polyol process and hydrogen reduction process was proposed. E.G. was used as the solvent and H_2 was used as the reductant. The reaction was completed in 10min. and E.G. after reduction could be used many times. The particle size was in sub-micrometer with uniform size and shape.

Experimental

The experiments were carried out in a 2 liter stainless steel autoclave in which a suspension of 500ml E.G. containing $\text{Co}(\text{OH})_2$, PdCl_2 , PVP, and NaOH was charged. When temperature reached, H_2 gas was introduced into the autoclave to start reaction. After finishing the reaction, Co powder was filtered, washed with ethanol and acetone and dried in an oven at 55°C . The chemicals used for the study were reagent-graded in purity. The characterizations of Co powder were performed by SEM, XRD and FT-IR.

Results

The preparing conditions of submicrometer-sized Co powder with uniform size and shape can be summarized as follows: the end pH is from 4.5 to 12; temperature changes from 140 to 170°C ; hydrogen pressure varies from 0.8 to 3.0 MPa; Co concentration is from 20 to 60 g/l; PVP/Co weight ratio is 0.5; PdCl_2/Co weight ratio is 2×10^{-4} ; and the reduction can be completed in 3~10min. No obvious difference can be observed between the E.G. before and after hydrogen reduction, so E.G. can be used many times. Adjusting the end pH of the system can control the morphology of Co powder obtained. The lamellar or spherical Co powder can be obtained separately at the end pH about 10 or 5. This may relates the different reaction mechanism.

References

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