



# Magnetic properties of aristate spherical Ni nanoparticles synthesized through ultrasound reduction method



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## ABSTRACT

Aristate spherical Ni nanoparticles with 5 nm–65 nm grain size range were synthesized via ultrasound reduction method using NiCl<sub>2</sub>·6H<sub>2</sub>O as raw materials, N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O as reducing agent and PVP as surfactant. Phase and microstructure of as-synthesized Ni nanoparticles were characterized by X-ray diffraction (XRD) and high resolution transmission electron microscope (HRTEM). Magnetic properties were measured using vibrating sample magnetometer (VSM) at room temperature. Results indicated that the aristate spherical morphology of Ni nanoparticles could be ascribed to PVP surfactant effect on its nucleation and growth. Specific saturation magnetization  $M_s$  of the specimens increased from 26.3 A m<sup>2</sup>/kg<sup>-1</sup> to 48.0 A m<sup>2</sup>/kg<sup>-1</sup> as their grain size increased from 5 nm to 65 nm. Coercivity increased with their grain size. The maximum coercivity  $H_c$  reached up to 23.20 KA/m as their grain size increased to about 42 nm, indicating the experimental critical grain size for the aristate spherical Ni nanoparticles. The theory value for the ferromagnetic exchange length of Ni nanoparticles was about 49.1 nm, which was consistent with the experimental result. Their magnetic behavior maybe attributed to the effect of nanometer size on magnetic particles.

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## 1. Introduction

Increasing attention has been paid to Ni nanoparticles in recent years due to their important applications in absorbing materials, magnetic recording, magnetorheological fluid, catalyst and medical materials [1–4]. In comparison with bulk nickel, nanostructured Ni particles show high reactivity, excellent catalytic activity and magnetic properties [5,6]. Many studies found that these excellent properties are closely related to their grain size, microstructure and synthesis method [7,8]. The synthesis and morphology control of Ni nanoparticles are therefore of great significance. Many physical and chemical methods have been used to synthesize Ni nanoparticles, such as aqueous reduction method [9], thermal decomposition of organic complexes [10], sol-gel method [11], polyol process [12], etc. However, these methods have shown several disadvantages, including harsh reaction conditions, easy agglomeration and oxidation of specimens.

Ultrasound reduction method has recently attracted much scientific interest due to its unique advantages. This method can accelerate and improve reaction efficiency by ultrasonic cavitation energy, thus trigger new chemical reactions [13,14]. This method has been used in the preparation of amorphous iron and nanometer cobalt powders [15]. In this paper, we report synthesis of aristate spherical Ni nanoparticles by the ultrasonic reduction method. The microstructure and the grain size effect on magnetic properties for the aristate spherical Ni nanoparticles were also discussed.

## 2. Experimental

The aristate spherical Ni nanoparticles were synthesized using an ultrasonic reduction method. Briefly, a solution was first prepared by dissolving reagent grade NiCl<sub>2</sub>·6H<sub>2</sub>O and PVP (PVP-K30, Sinopharm Chemical Reagent Co., Ltd.) surfactant in distilled water. The molecular weight of the used PVP was about 40000. Subsequently, N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O solution was added into the above solution as a reducing agent, and NaOH solution was used to adjust its final pH value. The obtained mixture continued to react by ultrasonic reduction for different time at 90 °C so as to form

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phase and alter size. The obtained powder particles were washed with distilled water and ethanol for several times, followed by drying at 40 °C in a vacuum oven for 12 h.

Phase composition of the specimens was investigated through X-ray diffraction spectrometer (XRD, Shimadzu LabX XRD-6000) with Cu-K $\alpha$  radiation. Average grain size was evaluated based on the Scherrer formula. Microstructural characteristics were observed by using high resolution transmission electron microscope (HRTEM, JEOL JEM-2010). Magnetization measurements were performed with vibrating sample magnetometer (VSM) at room temperature.

### 3. Results and discussion

#### 3.1. Phase composition

Fig. 1. Shows XRD patterns of Ni nanoparticles synthesized by the ultrasonic reduction method at different reaction time. All characteristic diffraction peaks were identified to be typical face central cubic (fcc) structure (JCPDS 04-0850). No any other phases were detected in these specimens. The main features of XRD patterns did not change with increased reduction time, except for the sharpening of the reflection peaks. This sharpening of the characteristic diffraction peaks revealed the grain growth of the Ni nanoparticles during the ultrasonic reducing process. The average grain size  $d$  of the specimens was obtained by Scherrer formula as follows,

$$d = k\lambda / (\beta \cos\theta) \quad (1)$$

Where  $\lambda$  is the wave length of the X-ray radiation,  $K$  is a constant taken as 0.89,  $\beta$  is the line width at half-maximum height, and  $\theta$  is the diffracting angle. According to the calculation, variation of the average grain size  $d$  of the specimens with ultrasonic reduction time is shown in Fig. 2. It was observed that the average grain size of the specimens increased from 5 nm to 65 nm as the ultrasonic reduction time was increased from 10 min to 150 min.

#### 3.2. Microstructural characteristics

Fig. 3. Shows typical HRTEM images of the Ni nanoparticles synthesized at different ultrasonic reduction time. It was observed that most of the particles were of an approximately aristate

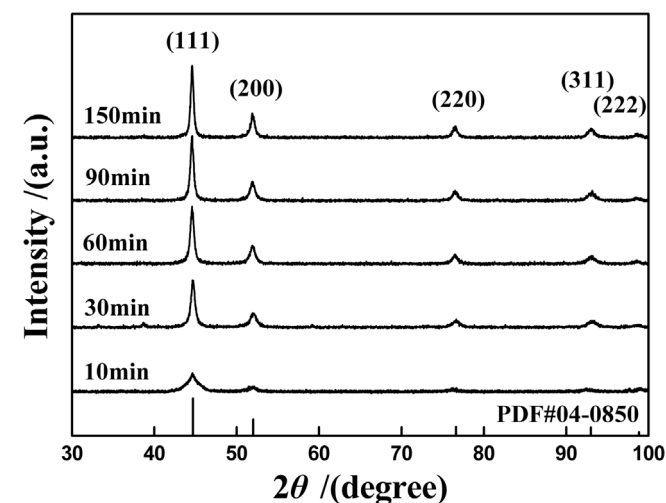


Fig. 1. XRD patterns of Ni nanoparticles synthesized by the ultrasonic reduction at different reaction time.

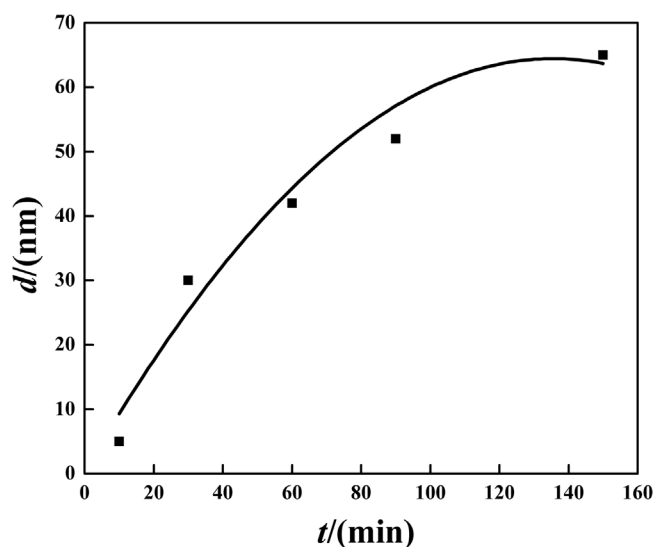


Fig. 2. Variation of the average grain size for Ni nanoparticles with different ultrasonic reduction time.

spherical shape. Fig. 4. Shows the average particle size of Ni nanoparticles synthesized at different ultrasonic reduction time. The particle size of the specimens increased from ~10 nm to ~160 nm as the ultrasonic reduction time was increased from 10 min to 150 min (Seen into Fig. 3(a)–(e)). Fig. 3 (f) depicts the selected area for electron diffraction (SAED) pattern of the aristate spherical Ni nanoparticles synthesized for 150 min. The plane spacing corresponding to (111), (200), (220), (311) and (222) of the Ni nanoparticles could also be clearly observed, indicating that the Ni nanoparticles were of polycrystalline nature.

Fig. 5. shows the schematic diagram for formation of aristate spherical Ni nanoparticles. During Ni nanoparticles synthesis by the ultrasonic reduction method, the free electrons from oxygen and nitrogen atoms in pyrrole alkyl of PVP molecules could easily form coordination bonds with nickel ions and then become stable Ni<sup>2+</sup>-PVP complex. The nickel ions were then reduced to nickel atoms with addition of N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O and NaOH reducing agents. Once the concentration of the nickel atoms exceeded the critical nucleation concentration, nickel crystal nucleus was formed.

During the synthesis of nanocrystalline materials, PVP was widely used as a surfactant. There is significant effect on size and shape control of nanometer-size powders [16,17]. When proper PVP molecular adsorbed on nickel crystal in spread state, aristate spherical nickel will form. It maybe ascribed to the fact that PVP molecular arrayed in the same direction served as the soft template of the growth. Some small crystal nucleus will form in N and O atoms of PVP molecular chain. The crystal nucleus will then grow faster following the PVP stretching state directions, thus preferring to form the aristate spherical Ni nanoparticles [18,19].

#### 3.3. Magnetic properties

Fig. 6. shows hysteresis loops of the aristate spherical Ni nanoparticles. Magnetization reached complete saturation at an external field of 200 kA/m, and specific saturation magnetization  $M_s$  of specimens increased with increasing ultrasonic reduction time. Fig. 7. shows variations of the specific saturation magnetization  $M_s$  and coercivity  $H_c$  of specimens with average grain size  $d$ . It was seen that the specific saturation magnetization increased from 26.3 A m<sup>2</sup>/kg<sup>-1</sup> to 48.0 A m<sup>2</sup>/kg<sup>-1</sup> as the average grain size increased from 5 nm to 65 nm. The coercivity  $H_c$  firstly increased with increasing grain size. The maximum coercivity  $H_c$  reached up

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