Synthesis of Nano-crystalline Nickel by solvothermal Reduction Process

Yuanzhu Mi, Dingsheng Yuan, Yingliang Liu* Jingxian Zhang

Tel: +862085221813; Fax: 862085221697; E-mail: tliuyl@jnu.edu.cn

Department of Chemistry, Jinan University, Guangzhou 510632, *P.R. China*

Transition metallic materials such as ferro-magnetic metal Ni,Fe,Co have been extensively studied because of their various applictions, such as catalysis, solar energy absorption, permanent magnets, magnetic fluids and magnetic recording media. Nickel nano-structures have thus attracted much attention recently because of their potential applications in magnetic sensors and memory devices [1-3]. Different Ni nanostucture have been successfully synthesized by various methods.

Herein we report the preparetion of fcc and hcp phase nickel through reduction of nickel chloride by KBH₄ in the absolute enthylenediamine. Their magnetic properties have been measured.

Fig.1 is the XRD pattern for the as-prepared Ni sample synthesized in enthylenediamine at different temperature. Figure 1a is shown x-ray diffraction pattern of Ni at 300 °C. The peaks are assigned to from the (010), (002), (011), (012), (110), (103), (112), (201) planes of hexaganol close-packed (hcp) nickel, respectively. Which are in agreement with the value in the JCPDS card (No45-1027). From the pattern, no characteristic peaks presenting face centered cubic (fcc) nickel have been observed. Fig.1b is the XRD pattern for the sample at 250 °C. The peaks from the pattern can be indexed to hcp and fcc nickel, which indicates that both hcp and fcc phases of nickel coexist in the samples at 250 °C. The nickel synthesized at 200 °C is only fcc phase (as shown in Fig.1c).

Figure 2 is shown the transmission electron microscopy (TEM) images of Ni synthesized in enthylenediamine at 300°C. Figure 2A is a high-resolution lectron microscopy (HREM) image. Microstructure of nickel shows hexagonal. Figure 2B shows a typical selected-area electron diffration (SAED) pattern that has recorded from the nanocrystal Ni. It can be indexed to be the [011] zone axis of the hcp nickel. These pattern spots demonstrate the single crystallinity of this Ni nanocrystals.

The magnetic properties of nano-sized Ni have been measured through VSM 7407 magnetometer. Figure 3 shows hysteresis loops of different crystal phase Ni at room temperature. The saturation magnetization (Ms) and coercivity(Hc) of fcc Ni are 39.86emu/g and 143.930e, respectively. These of hcp Ni are 7.35emu/g and 94.320e, respectively. Thus, nano-sized Ni materials with the different structure possess the different magnetic propertieds.

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Fig 1 XRD pattern of Ni synthesized in enthylenediamine at different temperature. (a)200°C,(b)250°C,(c)300 °C.(*)hcp phase nickel;(■) fcc phase nickel.Peaks represent fcc phase nickel (marked in parentheses)



Fig 2 TEM images and the electron diffraction pattern of nickel nanocrsytals synthesized at 300℃



Fig 3 Hysteresis loops of different crystal phase Ni at room temperature. (A)fcc, (B)fcc and hcp, (C)hcp