Chemical Synthesis of MSe_2 (M = Ni, Fe) Particles by Triethylene Glycol Solution Process

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Chemicals: Nickel(Ⅱ) nitrate ($Ni(NO_3)_2 \cdot 6H_2O$, 98.0%), Iron(Ⅱ) chloride ($FeCl_2 \cdot 4H_2O$, 99.7%), selenium powder (Se, 99%), triethylene glycol ($C_6H_{14}O_4$, TEG 99%), triethylenetetramine ($C_6H_{18}N_4$, TETA 70%), polyvinylpyrrolidone (PVP, Mr =10000), absolute ethanol (CH_3CH_2OH, 99.7%), and high-purity nitrogen gas. All chemicals were used as received.

Preparation of $NiSe_2$ particles: In a typical procedure, 0.25mmol $Ni(NO_3)_2 \cdot 6H_2O$ was dissolved into 10ml of TEG in a beaker under magnetic stirring at room temperature for 1h until a clear nickel precursor solution was obtained. 0.5mmol Se powder and 0.1g PVP were added into 40ml of TEG in a 100ml three-necked round-bottom flask as Se precursor solution. The three-necked flask was attached to a reflux condenser and heated under nitrogen stream and magnetic stirring from room temperature to temperature of 250℃. Then, the nickel precursor solution was quickly injected into the Se precursor solution in the three-necked flask with vigorous stirring. The reaction solution was refluxed at temperature of 230℃ for 45min, and then cooled to room temperature by water-bath quenching. The products were abstracted by high speed centrifugation in centrifuge tubes and then washed ultrasonically with excess amounts of absolute ethanol followed by high speed centrifugation for five times to purify products. In investigation to synthetic temperature, injection temperature was set as 190℃, 210℃, 230℃, 250℃, respectively, and the corresponding refluxing temperature was below 20℃ than its injection temperature, while the other processing parameters used were unchanged as above condition.

Preparation of $FeSe_2$ particles: The synthesis process was followed as above for $NiSe_2$ preparation, except that 0.25mmol $FeCl_2 \cdot 4H_2O$ was substituted for 0.25mmol $Ni(NO_3)_2 \cdot 6H_2O$ and an additional 0.05ml TETA was added into the Se precursor solution just before Fe precursor solution was injected. Also, synthetic temperature was set at 190℃, 210℃, 230℃, 250℃, respectively, and the corresponding refluxing temperature was below 20℃ than its injection temperature, while the other processing parameters used were unchanged as typical condition.

Characterization: X-ray diffraction (XRD) was detected by Rigaku D/Max 2500V/PC X-ray powder diffractometer (Japan) with CuKα radiation at 40kV and 200mA and a scan rate of 8°/min from 2θ = 10° to 90°. Morphological and structural inspections were performed by FEI Tecnai G2 F20 field-emission transmission electron microscope with SAED attachment(Netherlands) and Hitachi s-4800 field emission scanning electron microscope with EDX attachment(Japan). Raman spectrum was carried out on RENISHAW inVia reflex Raman spectroscopy(England). Optical absorption was recorded at wavelength range from 300 to 1200nm by Hitachi U-4100 UV/vis/NIR spectrophotometer(Japan).