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Fabrication of carbon layer coated FE-nanoparticles using an electron beam irradiation

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HIGHLIGHTS

- Carbon layer coated Fe-nanoparticles were successfully synthesized.
- The size of iron nanoparticles is controlled by electron beam irradiation dose.
- The encapsulation is confirmed between the carbon layer and iron nanoparticle.

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ABSTRACT

A novel synthesis of carbon encapsulated Fe nanoparticles was developed in this study. Fe chloride (III) and polyacrylonitrile (PAN) were used as precursors. The crosslinking of PAN molecules and the nucleation of Fe nanoparticles were controlled by the electron beam irradiation dose. Stabilization and carbonization processes were carried out using a vacuum furnace at 275 °C and 1000 °C, respectively. Micro structures were evaluated by X-ray diffraction (XRD) and transmission electron microscopy (TEM). Fe nanoparticles were formed with diameters of 100 nm, and the Fe nanoparticles were encapsulated by carbon layers. As the electron beam irradiation dose increased, it was observed that the particle sizes decreased.

1. Introduction

Magnetic nanomaterials have attracted interest for applications in many industrial fields, such as magnetic sensors (Koh and Josephson, 2009), magnetic carriers (Tada et al., 2007), biomedical applications (Roca et al., 2009) and many electric devices (Wang and Li, 2008). Among the ferromagnetic materials, Fe nanoparticles have received extensive attention because of their high saturation magnetization (Sun et al., 2000). However, metallic nanomaterials have problems with oxidation, aggregation, and flammable properties. Capsulation is a key technology to prepare stable nanomaterials. Polymer (Ditsch et al., 2005), silica (Tada et al., 2007), and carbon (Schiffmann et al., 1999) materials have been reported as capsulation materials for metallic nanoparticles. Among these, carbon shells are a good candidate because of their electrical properties and excellent stability in physically harsh environments. The polyacrylonitrile (PAN) chosen in this study is usually used as a carbon precursor because of its good mechanical properties and stability. In our previous works, the fabrication of carbon nanomaterials was investigated using PAN (D.Y. Kim et al., 2012), PAN/lignin (Seo et al., 2011) and PAN/TiO_2 (Jeun et al., 2011) along with electron beam irradiation. Electron beam irradiation is useful to stabilize PAN materials by molecular crosslinking, cyclization, and oxidation. In addition, it has been reported that electron beam irradiation can be used to fabricate metallic nanomaterials with size control (S.E. Kim et al., 2012).

In this paper, we applied the electron beam irradiation technique for the generation of Fe nanoparticles and the stabilization of PAN simultaneously.

2. Experimental details

The experiment was involved material preparation, electron beam irradiation, stabilization and carbonization process, as shown in Scheme 1. The steps are explained in detail below.

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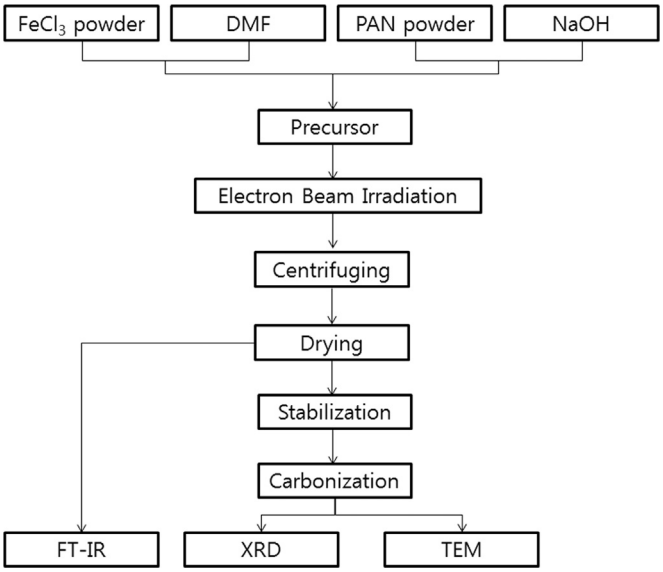

Scheme 1. Schematic diagrams for the experimental procedure.

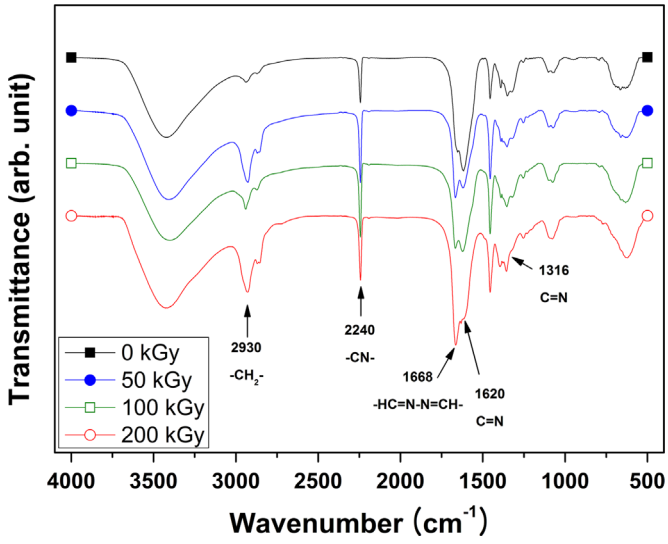

Fig. 1. FT-IR spectra of the PAN/Fe composites as a function of the electron beam irradiation dose.

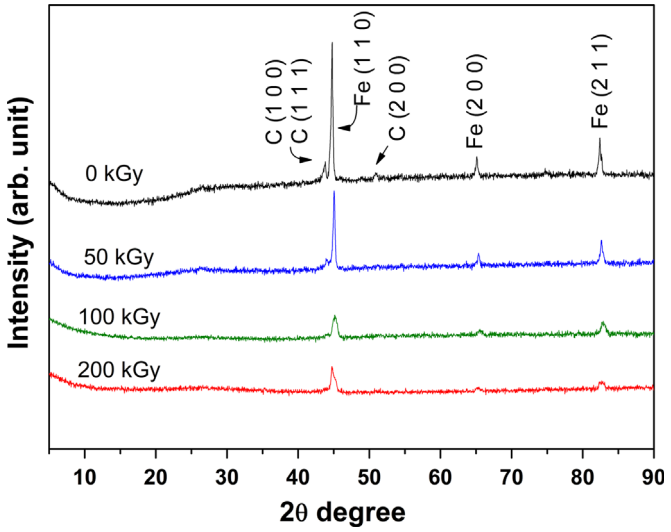

Fig. 2. XRD spectra of carbon encapsulated Fe nanoparticles as a function of the electron beam irradiation dose after the carbonization process.

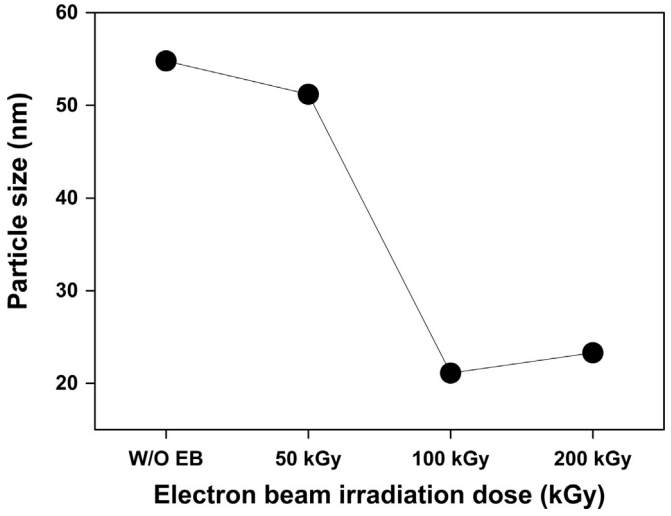

Fig. 3. Simulated particle size of nanoparticles according to the main peaks in the XRD spectra according to the Scherrer equation.

2.1. Materials

Iron chloride ($FeCl_3$, Aldrich) and polyacrylonitrile (PAN, Mw=150,000, Adrich) were used as precursors. PAN (2 g) was dispersed in NaOH solution (40 mL, 1 N, 99%, Showa) using magnetic stirring. $FeCl_3$ powders (2 g) were dissolved in dimethyformamide (DMF, anhydrous, 99.8%, Aldrich) solution (36 g). The two solutions were mixed using magnetic stirring in a beaker and divided into four glass bottles with the same weight to prepare the precursor. All of the materials were used without further purification.

2.2. Electron beam irradiation

Electron beam irradiations were performed with a beam energy of 10 MeV and a beam current of 0.5 mA at Korea Atomic Energy Research Institute (KAERI). Each sample was irradiated by 50, 100, and 200 kGy a rate of 1 kGy/scan. After irradiation, all samples were centrifuged at 13,000 rpm for 10 min and washed with DMF.

2.3. Stabilization and carbonization

The stabilization process was carried out using a vacuum furnace at 275 °C for 1 h. The background pressure of the furnace tube was less than 5×10^{-4} Torr, and a gas mixture of O_2 (100 sccm) and N_2 (400 sccm) was injected to create a working pressure of 10 Torr. The carbonization process was also performed using a vacuum furnace at 1000 °C for 1 h at a working pressure of 10 Torr with only a N_2 gas atmosphere.

2.4. Characterization

Fourier transform infrared (FT-IR) spectrometry (Brucker Tensor 37) was used to analyze the chemical structure of samples that underwent different electron beam pretreatments. The dried powders were mixed with KBr and analyzed in the range of 500–4000 cm^{-1}. The microstructure of the carbonized samples was evaluated by X-ray diffraction (XRD) measurements (PANalytical X'pert Powder) with Cu Ka (λ=1.5418 Å) and field-emission transmission electron microscopy (FE-TEM, JEOL JEM-2100F).