RA06

Synthesis of Fe Fine Particles Encapsulated in Carbon Cages using Surface Wave Plasma

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The Arc discharge technique has been developed to synthesize fullerenes and carbon nanotubes. The carbon nanocapsules filled with LaC_2 were discovered during investigation of fullerenes. The carbon nanocapsules are typically 10-200 nm in diameter and their graphite shells keep the LaC_2 particles away from degradation.[1] Various metals have been encapsulated in carbon nanocapsules. Magnetic fine particles encaged in carbon nanocapsules, in particular, have attracted a great deal of attention from scientific and practical interest.[2] In this study, Fe fine particles encapsulated in carbon cages were prepared by surface wave plasma technique. Fe filled carbon cages were characterized by TEM, SEM and EDX.

A Schematic illustration of the reactor is shown in figure 1. A surface wave plasma was produced by a microwave (2.45 GHz, 500 W) discharge. CH_4 / Ar mixture gas was introduced into the reactor. The total pressure was 0.6 - 1.5 kPa. Fe encapsulated in carbon cages was supplied by a DC arc discharge evaporation of pure Fe electrodes. The arcdischarge current was varied from 0.5 to 3.0 A. After the process, the Fe anode was consumed by the arc discharge evaporation. Fe particles were deposited on a ceramic plate located under the Fe electrodes.

The typical size of Fe particles, which was 100 nm - 10μm in diameter, was approximately one order of magnitude larger than that synthesized by the arc discharge. A typical TEM image of Fe fine particle is shown in Fig. 2. From the selected area electron diffraction measurements, it was confirmed that the outer shell was composed of graphite layers and the inner particle was bcc Fe single crystal.

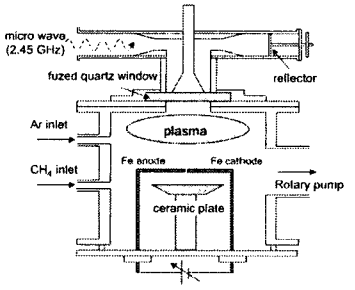

Fig. 1. Illustration of the reactor.

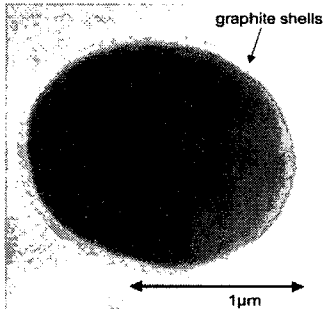

Fig. 2. TEM image of Fe particle.

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RA07

Phase Control and Characterization of Iron and Iron-Oxide Nanocrystals Synthesized by Pulsed Wire Evaporation Method

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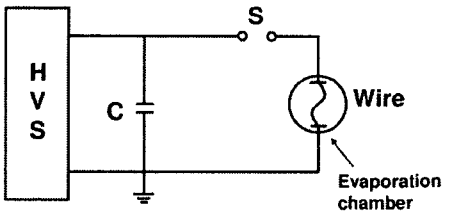
Nanosize powders have been of scientific and technological interest because of their specific properties that are not found in bulk materials. The electromagnetic and chemical reactivity, along with the optical properties from a large specific surface area allow the nanosize powders to be new material that bears a great potential for many important applications. In this work, Nanosize iron-metal and -oxide powders were synthesized by the pulsed wire evaporation (PWE) method, one of the gas condensation processes, which is known as one-step synthetic technique with high efficiency and high production rate compared with other wet processes involving several treatment steps [1].

The experimental setup for evaporating the wires to produce nanosize powders is shownin Fig. 1. The synthetic apparatus consists of four main components, the high voltage dc power supply, the capacitor bank, the high voltage gap switch, and the evaporation chamber. Pure iron wire (>99.9 %) with a diameter of 0.47 mm was used as a starting material and the feeding length of the wire into the reaction chamber was 88 mm. When a pulsed high voltage of 26 kV is driven through a thin wire, a non-equilibrium over-heating, which is induced in the wire, can evaporate the wire into plasma within several micro-seconds. The high-temperature plasma is cooled by an interaction with an ambient gas and condensed into small-size particles. The nanosize iron-metal powder was synthesized by evaporating a thin iron wire in argon inert gas and then an oxide passivation layer was added to the fresh powder by a slow oxygen filling with a flow rate of 3 mL/min for 30 min. X-ray diffraction (XRD) and transmission electron microscopy (TEM) measurements indicate that the spherical iron nanoparticles are about 55 nm in diameter and the thickness of the surface passivation layer is between 2 and 3 nm.

In the case of the iron-oxide powders, an argon-oxygen mixed ambient gas was supplied to the reaction chamber during the evaporation process, in which the total pressure of the mixed gas was about 1.3 bar. The phase analysis of the produced iron-oxide powders was systemically investigated using Mössbauer spectra, where the phase of Fe_2O_3 and Fe_3O_4 showed a strong dependence on the oxygen concentration in the mixed gas. The results suggest that classified nanosize iron-metal and -oxide powders can be obtained simply by controlling a well-designed ambient gas condition during the PWE process.



HVS : High Voltage DC Source
S : Switch
C : Capacitor

Fig. 1. Experimental setup for a pulsed wire evaporation.

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