CVC 법에 의해 제조된 Fe 나노분말의 미세조직 분석 Microstructural characterization of Fe nanoparticles produced by CVC process

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1. Introduction.

In recent years, much attention has been paid to synthesize and investigate nanoparticles, because of their wide range of potential applications. Since the properties of these nanoparticles are basically determined by their mean size, size distribution, external shape, internal structure and chemical composition, the characteristics of powders must be controlled during the production of the nanoparticles so that they are suitable to specific applications.

In this paper, we synthesized iron nanoparticles by Chemical Vapor Condensation (CVC) by the pyrolysis of organometallic precursor of Fe(CO)₅. The effect of processing parameters and annealing process on the microstructure and size of Fe nanoparticles were investigated.

2. Experimental Details

The basic setup for CVC is similar to that described in literature elsewhere. The flow of carrier gas entraining precursor (iron pentacarbonyl (Fe(CO)₅) vapor passed through the heated tubular furnace to the work chamber. To collect nanoparticles, the mixing gas of Ar contained a trace O_2 flowed for 10h with pressure of 1kg/cm² for a passivation process, which slowly oxidized nanoparticles. The morphologies and particles size distribution were determined by transmission electron microscopy using JEOL JEM-2000FXII equipment. The lattice parameter determination and the phase analysis of samples were carried out on RIGAKU Geigerflex diffractometer with monochromatic CuK_{α} radiation.

3. Results and Discissions.

Iron nanoparticles was successfully synthesized by CVC process using iron pentcarbonyl as a precursor under Ar or He atmosphere. The spherical nanoparticles of the mean diameter of 6-25nm comprise of the metal core and oxide shell. Average particle size increases and size distribution becomes wider and more asymmetric with increasing the decomposition temperature. Particles produced by CVC have larger size if argon as carrier gas was used.

For the particles, which were prepared at the decomposition temperature of $4\dot{0}0^{\circ}$ C, the particle size distribution is more close to normal than lognormal ones. It means that absorption growth mechanism predominates at lowest decomposition temperatures; particles growth is going on by separate atoms absorption. The increase of decomposition temperature leads to coalescence growth mechanism predominates, the distribution becomes lognormal and the mean particle size increases.

The increase of lattice parameter of metallic core with the decreasing particle size can be explained by the epitaxial growth of oxide shells. The drastic decreasing of lattice parameter of BCC phase during low temperature annealing is observed.