

Synthesis and polymer coating of nano iron powder by chemical vapor condensation

Dong-Won Lee*, Jung-Hyun Bae, Byoung-Kee Kim
Korea Institute of Machinery and Materials (KIMM)

1. Introduction : The synthesis of metallic iron nanoparticles has been actively tried by chemical vapor condensation with the interest of possibility for application such as high density of recording medias or magnetic fluids because of its superior magnetic properties to commercialised nanostructured magnetite (Fe_3O_4) powders. The main draw-backs in this process are 1) low productivity as especially chilling inside chamber and 2) surface oxidation of iron powders by passivation treatment, which negatively has affected the inherent magnetic properties of pure iron. In this study therefore the iron nano-powders generated into chamber were not chilled, and for self-assembled monolayer coating some of them was soaked directly into chemical solution installed inside the chamber.

2. Experiment : Ironpentacarbonyl, $\text{Fe}(\text{CO})_5$ was vaporized in the bubbler with the temperature of 160°C and transferred by the carrier gas of argon into hot-wall reactor tube with inner dia., 50mm and length, 400mm with different temperature ranges from 400°C to 1000°C . Synthesized iron nano powders were then generated directly into empty chamber with room temperature. The iron nanopowders were slowly piled in bottom and wall of chamber and then collected after air passivation. Polymer-coating of iron nanopowders was processed by means of installation of 2mM octadecylsulfate ($\text{CH}_3(\text{CH}_2)_{17}\text{OSO}_3$) solution inside the chamber. In produced all powders, the TEM microstructures, X-ray diffraction patterns, chemical element analysis and magnetic properties of VSM technique were investigated.

3. Results and Discussion : The mean particle size of produced iron nanopowders were increased from 8nm to 96nm with increase of temperature of reaction tube. On the other hand, the oxide shell thicknesses were also slightly increased but with the small tendency compared with increasing of core size. At 1000°C , approximately half amount of non-magnetic γ -Fe phase was mixed with α -Fe because of rapid cooling effect from γ -Fe stable region therefore it has disturbed the ferromagnetic properties of α -Fe. In the 8nm of means particles size, slightly decreased coercivity was probably due to the superparamagnetism revealed in some amount of particles smaller than 8nm. By polymer coating, oxide content in product was greatly decreased, the surface was protected with C-H chains and it resulted in big decrease of coercivity because of absence of Fe_3O_4 phase, which has higher coercivity and magnetic anisotropy than those of pure α -Fe.

Table 1. Properties of nano iron powders produced by chemical vapor condensation

Reaction Temp. ($^\circ\text{C}$)	Fe-Core (nm)	O (wt.%)	C (wt.%)	S (wt.%)	Fe_3O_4 Shell thickness (nm) (Calculated from oxygen Content)	Coercivity (Oe)	Max. Mag. (emu/g)
400	8	14.3	0.12	-	2.5	745	125
600	17	13.6	0.15	-	3.4	998	147
600 (Polymer-coated)	16	4.7	16.1	2.20	0.8	195	142
800	68	3.8	0.25	-	3.6	103	205
1000	96	2.0	0.23	-	3.8	95	76