ポリオールプロセスを用いた鉄ナノ粒子の合成

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Synthesis of Fe Nanoparticles through Polyol Process

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The synthesis of pure transition metal nanoparticles has been difficult to achieve due to their highly oxidative nature. The synthesis of metal nanoparticles through aqueous chemical processing techniques often leads to large amount of oxide impurities. However, Co and Ni particles have been successfully synthesized using the polyol process [1]. Here, hydroxyl ions have been found to play a major role in enhancing the reaction rate and Co nanoparticles with hcp-, fcc- and ε-Co structures have been reported [2]. In the case of Fe, synthesis of sub micron size particles has been achieved [3] through the disproportionation of $Fe(OH)_2$ in the presence of highly alkaline conditions as shown below

$$FeCl_2.4H_2O+2NaOH \rightarrow Fe(OH)_2+2NaCl+4H_2O$$

$$4Fe(OH)_2 \rightarrow Fe + Fe_3O_4 + 4H_2O$$

As expected from the above equation, equal amount of Fe_3O_4 and Fe are synthesized and the yield was reported to be low. Since then, the synthesis of Fe nanoparticles in polyols has not been attempted. In this paper, we describe about the synthesis of Fe nanoparticles in a polyol medium like EG, PG, TMEG, TEG and Tri EG in the presence of NaOH using $FeCl_2.4H_2O$ as the metal precursor. By varying the reaction conditions, Fe nanoparticles was synthesized with higher yield with the particle sizes around 20-50 nm as seen from the TEM micrograph shown in Figure 1 (a). The XRD pattern of the particles synthesized in a polyol medium showed the presence of both magnetite and Fe with average crystallite sizes of 10 and 20 nm, respectively. However, on the contrary to what is proposed by the disproportionation equation, we observe only very small amount of magnetite and varying the synthesis conditions could control its relative peak intensity. Thus, we believe that the reaction mechanism is quite different to disproportionation as proposed by previous researchers. The influence of synthesis parameters such as salt concentration, hydroxide to metal mole ratio, reaction temperature, etc. on the purity, yield and property of the particle will be reported. And the reaction mechanism will also be discussed.

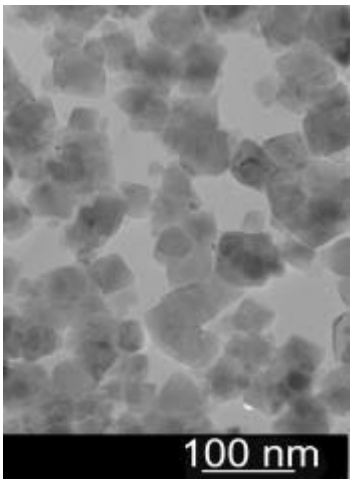

Fig.1(a) The TEM photograph of the Fe nanoparticles.

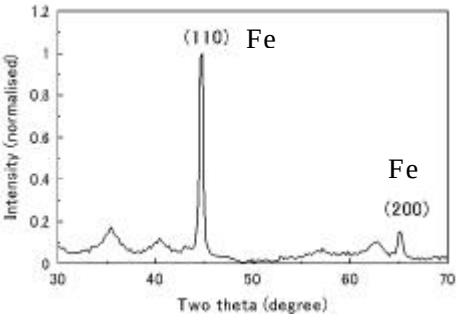

Fig.1(b) The XRD pattern of the nanoparticles synthesized in polyol.

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