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Preparation of Au and Au-coated Fe nanopowder by microemulsion

Yong-Jin Kim[a,*], Ji-Hun Yu[a], Jung-Ho Ahn[b]

[a] *Powder Materials Research Center, Korea Institute of Machinery and Materials, 66 Sangnam, Changwon 641-010, Republic of Korea*
[b] *Department of Materials Engineering, Andong National University, 388 Songchung, Andong 760-749, Republic of Korea*

Abstract

Au and Au-coated Fe nanopowders were fabricated by reverse micelle process. The *R* ([water]/[surfactant]) value in the system was varied to control powder characteristics. The synthesized Au powders had spherical shape with uniform size of around 10 nm and the powder size increased with increasing of *R*-value. Au-coated Fe nanopowder showed that Fe core of 5 nm in size was coated by Au shell of 2 nm in thickness. The coated nanopowder presented superparamagnetic behavior having very low coercivity of 31 Oe at room temperature.

Keywords: Nanostructured materials; Chemical synthesis; Microstructure; Magnetic measurement; Reverse micelle

1. Introduction

Nano-sized gold and iron powders have attracted much attention due to their potential applications in biotechnologies [1,2]. Gold nanopowders have high thermal and electrical conductivities, and also have high corrosion and oxidation resistance even in very fine particle size region. Iron nanopowders, however, are easily oxidized when the powders are exposed to the air. To prevent the oxidation and corrosion of iron powders, gold can be a good candidate as protective material to coat on the iron nanopowder surface.

In the present work, gold and gold-coated iron nanopowders were synthesized using microemulsion process [3,4]. Using water-in-oil microemulsions called reverse micelles, the monodispersed Au and Au-coated Fe nanopowders have been fabricated. The characteristics of the powders were discussed with microstructures and magnetic properties.

2. Experimental procedures

2.1. Synthesis of Au nanopowder

The microemulsion systems used in this study were CTAB/1-butanol/octane/aqueous solution. CTAB (cetyltrimethylammonium bromide) and 1-butanol were the surfactant and the co-surfactant, respectively. Octane was the oil matrix and aqueous solution droplet contained Au salt (0.056 M $HAuCl_4$). The other microemulsion with the reduction agent (0.32 M $NaBH_4$) mixed with Au microemulsion and Au salt was decomposed to form Au nanopowder by reduction agent. Then the solid product was separated by the centrifuging and washed several times with a mixture of chloroform and methanol. The washed particles were dried in vacuum at 70 °C for 2 h. The molar ratio of water to surfactant, *R*, was changed from 3 to 25 to vary the particle size.

2.2. Synthesis of Au coated Fe nanopowder

Iron nanopowder was fabricated using 0.056 M $FeSO_4$ under high pure Ar atmosphere by the microemulsion process as shown above. Two aqueous solutions with gold salt (0.056 M $HAuCl_4$) and reduction agent (0.32 M $NaBH_4$) were added into the fabricated Fe particle solution. After decomposition of Au salt, the final solid product was obtained by the above separation, washing and drying processes. These powders were characterized by using TEM, EDS, XRD and VSM.

3. Results and discussion

Fig. 1 shows the TEM micrograph of Au nanopowder synthesized using $R = 5$. It shows the well-arrayed Au nanopowders having spherical shape. In this condition ($R = 5$), the synthesized powder had very narrow size distribution ranging from 5 to 7 nm and the average size of 5.8 nm. The diffraction pattern (Fig. 1b) shows a typical ring pattern corresponding to metallic Au phase. The effect of *R*-value on the powder size was investigated and the result is shown in Fig. 2. The mean size of Au nanopowder increased from 4 to 9 nm with increasing *R*-value from 3 to

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[*] Corresponding author. Tel.: +82 55 280 3527; fax: +82 55 280 3399.
E-mail address: yjkim@kmail.kimm.re.kr (Y.-J. Kim).

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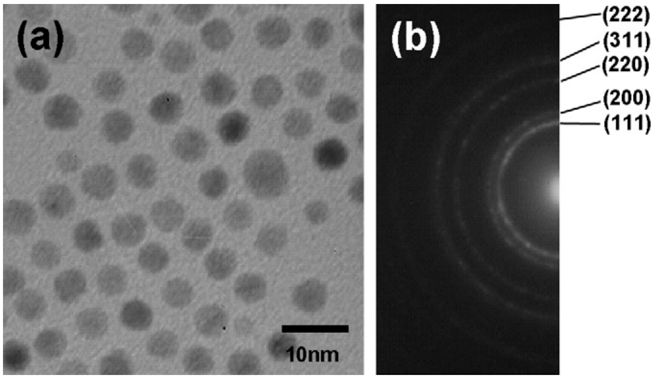

Fig. 1. TEM micrographs of Au nanopowder at the synthesis condition of $R = 5$: (a) bright field image and (b) selected area diffraction pattern.

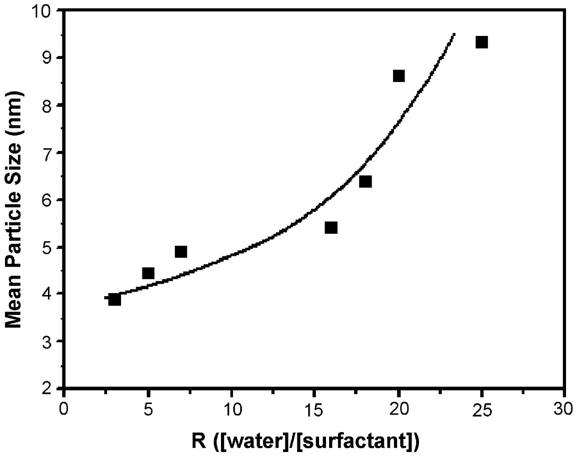

Fig. 2. Variation of mean size of Au nanopowder with R-value.

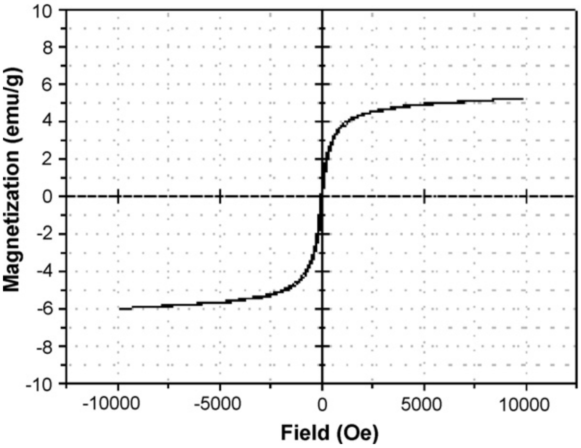

Fig. 4. Magnetic property of Au coated Fe nanopowder synthesized at $R = 20$.

25. It has been well known that the particle size increases with the increasing of R-value due to the larger droplet size at higher R-value. Therefore, this implies that the R-value is one of important factor to determine the particle size in the microemulsion system.

Fig. 3 shows the TEM micrographs of Au-coated Fe nanopowder synthesized at $R = 20$ using a mixture of 0.056 M $FeSO_4$ and 0.056 M $HAuCl_4$ aqueous solutions. It shows the particle size of core-shell structured powders was 9 nm on average representing well agreement with the result of Fig. 2. According to XRD result (not shown here), only metallic Au and Fe peaks were observed without any oxide peak.

The magnetic property of Au-coated Fe nanopowder measured by VSM is shown in Fig. 4. The saturation magnetization (M_s) and coercivity (H_c) are 6 emu/g and 31.3 Oe, respectively. Considering that Fe composition in Au-coated Fe nanopowder measured by EDS on TEM was only 10 wt%, this lower M_s value is quite reasonable. The superparamagnetic behavior of Au-coated Fe nanopowder having low coercivity demonstrates a potential for biomedical applications.

4. Conclusions

Well-dispersed Au and Au-coated Fe nanopowder with narrow size distribution have been successfully synthesized by reverse micelle process. The particle size increased with increasing R-value. The Fe core size and Au thickness of the Au-coated Fe nanopowder were 5 and 2 nm on average, respectively, and had very low M_s and H_c values exhibiting superparamagnetism.

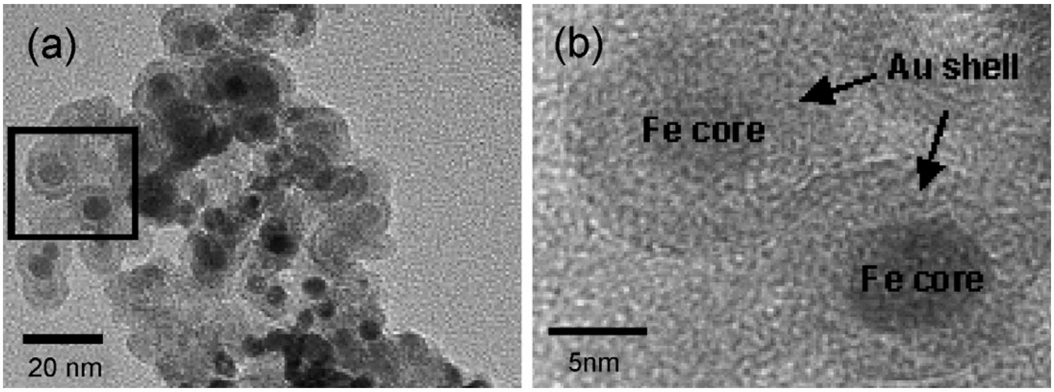

Fig. 3. TEM micrographs of Au coated Fe nanopowder synthesized at $R = 20$.

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