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THE IRON-GOLD MAGNETIC NANOPARTICLES: PREPARATION, CHARACTERIZATION AND MAGNETIC PROPERTIES

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Abstract. Superparamagnetic iron-gold nanoparticles were prepared by a reverse micelle method. The ferromagnetic iron formed a core of the nanoparticles while the diamagnetic gold a shell on the surface of the core. The presence of a gold coating inhibits oxidation and allows the particles to be stable. The Mössbauer spectra confirmed the superparamagnetic behaviour and indicated that the Fe and metallic Au exist as composite nanoparticles, not as separate particles. The obtained hyperfine parameters also confirmed the absence of iron oxides (Fe_3O_4, Fe_2O_3). Magnetic measurements in the temperature range of 4.2– 280K show that the particles are superparamagnetic with a blocking temperature T_B of 24K. At the temperature 280K (above T_B) no coercivity H_C is observed in the magnetization curve, while at 4.2K (below T_B) coercivity H_C is observed to be 150 Oe.

Nanosized magnetic particles have physical and chemical properties that are characteristic of neither the atom nor the bulk counterparts. Quantum size effect and the large surface area of magnetic nanoparticles dramatically change some of the magnetic properties and exhibit superparamagnetic phenomena and quantum tunnelling of magnetization, because each particle can be considered as a single magnetic domain [1]. Due to their unique properties, superparamagnetic nanoparticles have been widely used in the fields of biotechnology and ferrofluid technology [2,3]. In the present work, the nanoparticles of iron coated by gold were prepared by chemical method and their structural and magnetic characteristics were analyzed.

All chemicals were purchased from Aldrich and Sigma and used without further purification. Nanoparticles of were prepared in reverse micelles [4]. All reverse micelle solutions were prepared using cetyltrimethylammonium bromide (CTAB) as the surfactant with octane as the oil phase. 1-butanol was used as co-surfactant, increasing the polarity of the surfactant and helping to stabilize the micelle solutions [5]. Aqueous reactants of $FeSO_4$, $NaBH_4$, and $HAuCl_4$ were used to form the reverse micelle. The size of the nanoparticles is

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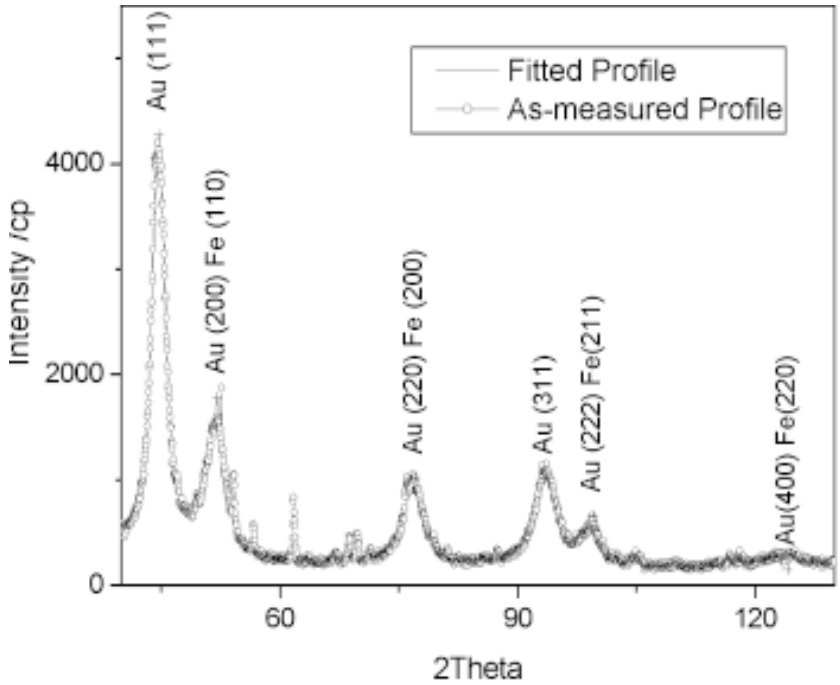

Fig. 1. X-ray diffraction pattern of the Fe@Au nanoparticles.

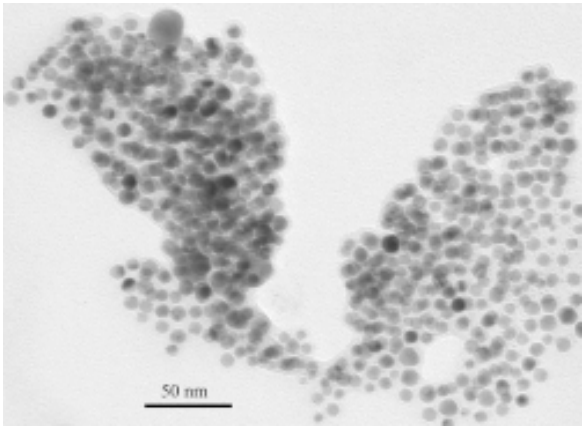

Fig. 2. The TEM micrograph of the Fe@Au nanoparticles.

determined by the water to surfactant molar ratio. Here, the molar ratio of water to surfactant was $\omega = [H_2O/CTAB] = 8$. The structure of the nanoparticles was studied by X-ray diffraction (XRD) using a Philips PW1050 diffractometer with Fe filtered CoK_α radiation (λ=0.17902 nm). The microstructure and the size of nanoparticles were examined by transmission electron microscope TEM (Philips CM-20). The elemental analysis was measured by a Ge detector NORAN EDS, attached to the CM-20 microscope. The temperature and the field dependence of the magnetization (from 4.2K up to 300K) were measured using a Vibration sample magnetometer (VSM). Mössbauer spectra were collected at room temperature, with a conventional constant acceleration spectrometer in transmission geometry with a $_{57}$Co source diffused into a Rh matrix.

The X-ray diffraction pattern taken from Fe@Au nanoparticles is shown in Fig. 1. All the peaks are corresponding to the fcc metallic gold diffraction. The XRD pattern of α- iron is hidden under the pattern of gold due to the overlapping of their diffraction peaks at 2θ=52.16°, 76.9°, 99.6°, 124.5°. No diffraction patterns of iron oxides were observed in the XRD spectrum indicating that the iron nanoparticles were well protected by the gold shell. The diffraction studies confirm a high degree of crystallinity and uniformity in the particles.

The elemental analysis measurements show, that the iron-gold nanoparticles are composed by 68% of metallic Au and 32% of metallic Fe. Morphology of the nanoparticles was determined using TEM. Representative micrograph is presented in Fig. 2. The particles of Fe@Au are isolated without aggregation and clearly spherical with average size 10 nm.

The Mössbauer spectra confirmed the superparamagnetic behaviour of the nanoparticles

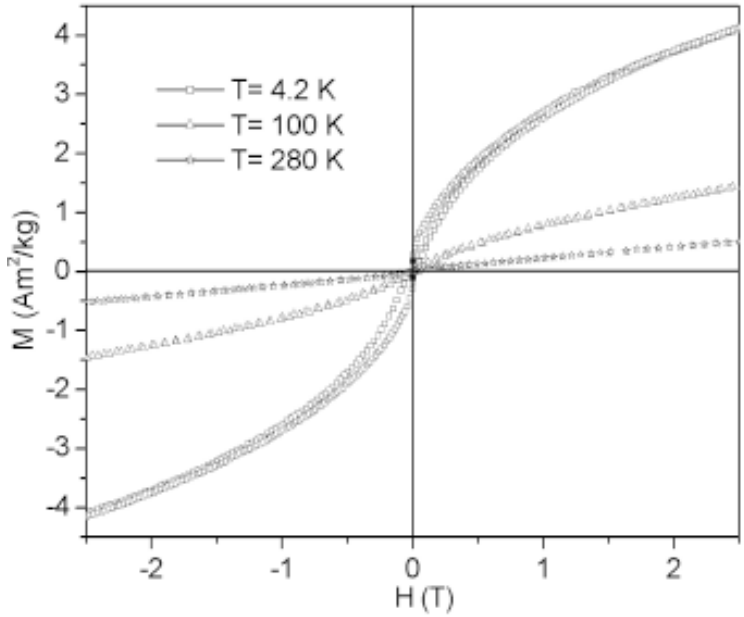

Fig. 3. The field dependence of the magnetization of Fe@Au nanoparticles measured at significant temperatures.

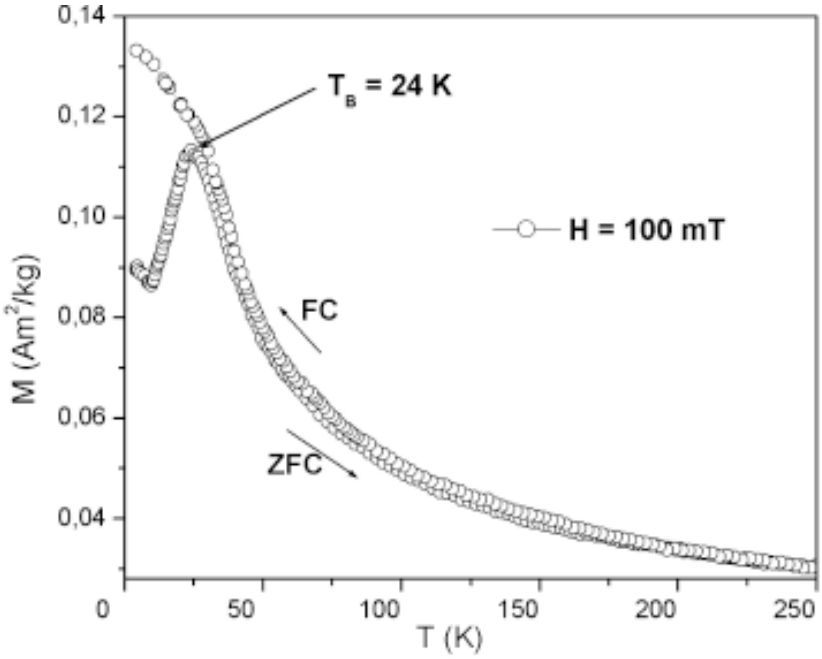

Fig. 4. The temperature dependence of the magnetization (ZFC and FC plots) of Fe@Au nanoparticles measured in the field 100 mT.

[6]. The major component in Mössbauer spectra measured at temperature 300K is a doublet with high value of quadrupole splitting. The spectra were fitted by hyperfine field distribution method. The obtained hyperfine parameters also confirmed the absence of iron oxides (Fe_3O_4, Fe_2O_3).

To check the superparamagnetic behaviour of iron-gold nanoparticles, we have performed magnetization measurements (by VSM) as functions of both temperature *T* and applied field *H*. Fig. 3 shows the variation of magnetization *M* as a function of external magnetic field *H* in the range 0–6 T

measured at temperatures ranging from 4.2 to 280K. The magnetization versus field plot *M(H)* measured at the temperature 280K (Fig. 3) shows that hysteresis disappeared, indicating the absence of a long-range magnetic dipole-dipole interaction among the superparamagnetic iron-gold nanoparticles. When the temperature decreases to 4.2K the magnetization of the sample increases with a symmetric hysteresis loop (coercivity H_C ~150 Oe), showing a transition from superparamagnetic to ferromagnetic behaviour (fee Fig. 3).

The zero-field-cooled (ZFC) and field-cooled (FC) magnetization curves measured in the DC field of 100 mT are shown in Fig. 4. The ZFC curve exhibit a relatively narrow maximum with the peak value at the blocking temperature T_B=24K. This indicates that below blocking temperate (T_B), the magnetic moment of each particle is blocked along its easy magnetization axis, whereas above T_B the particles are characterized by superparamagnetic behaviour. The shape of the ZFC curve also suggests a relatively narrow size distribution of the Fe@Au nanoparticles [4].

We have synthesized the superparamagnetic spherical Fe@Au nanoparticles with size 10 nm and with the core-shell structure. The superparamagnetism was confirmed by magnetic measurements at various temperatures (4.2–280K) and by Mössbauer spectra measurements.

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