別紙様式８

研　究　主　論　文　抄　録

論文題目

Synthesis of Fe and Sn, In system nanoparticles by pulsed plasma in liquid method
（液体中パルスプラズマ法による Fe と Sn、In 系ナノ粒子の合成）

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主論文要旨

The purpose of this study in this dissertation was to synthesize Fe, magnetite (Fe_3O_4), $Au@Fe_3O_4$, FePt, In, Sn and oxide (In_2O_3, SnO_2) nanoparticles using a pulsed plasma in liquid method and characterize their structure, morphology and properties. The pulsed plasma in liquid, in which nanoparticles are formed from repetitional- pulsed plasma, has the potential to meet the increasing demand for the direct preparation of well-dispersed nanostructures and offers a simple, non-toxic and low-cost alternative to conventional techniques. Brief conclusions of each chapter are described below.

In the ***Chapter 1***, the general introduction to Nanotechnology, importance and main properties of magnetic Fe-system nanoparticles, Sn- and In- system nanoparticles and their applications, pulsed plasma in liquid method, purpose of the work and summary of the dissertation were given.

In the ***Chapter 2*****,** pure α-Fe nanoparticles with ≤10 nm in size were synthesized using pulsed plasma in liquid method. This was the first time that pure metallic nanoparticles were prepared by arc discharge method using water-toluene interface as a medium. Several experiments made evident, that toluene-water ratio in emulsion influences the purity and size of Fe nanoparticles. The purity of the nanoparticles gradually increased from 48 to 98%, while particle size decreased from 21 to 9.5 nm with smaller toluene volume fraction (from 40 to 5%) in the microemulsions. Finally, toluene:water with 95:5 (%) ratio was found to be the most appropriate medium for pure Fe nanoparticle formation. Lattice parameters of the obtained Fe samples calculated from XRD found to be larger (a=0.2927 nm) than those previously reported Fe ($a_{(BCC\text{-}Fe)}$ =0.2866 nm). HRTEM showed spherical-shaped Fe nanoparticles with partial aggregation. Vibrating sample magnetometer indicated superparamagnetic properties of particles with high-saturation magnetization (M_s=125 emu g^{-1}) at room temperature.

In the ***Chapter 3*****,** spherical ferromagnetic Fe_3O_4 (magnetite) nanoparticles with an average diameter of 19 nm were synthesized by pulsed plasma in liquid at a voltage of 200 V, a current of 6

A, a frequency of 60 Hz, and a single discharge duration of 10 μs. Water with different concentrations of 1-hexadecylpyridinium bromide (CPyB) was applied as a liquid, and several experiments made evident that the surfactant concentration affects the phase compositions of the produced materials. The purity of magnetite phase in the sample increased (from 65 to 98%) with increasing CPyB concentration (from 0.10 to 0.84 g) in 200 ml of water. The crystal structure of magnetite nanoparticles with the Fd3m space group and a lattice parameter of a=0.8393 nm was evident from X-ray diffraction results.

Ferromagnetic Au-Fe_3O_4 nanoparticles with coercivity (Hc) of 210 Oe were synthesized from Fe electrodes submerged in aqueous solution of $HAuCl_4$ using pulsed plasma. Cytotoxicity of spherical Au-Fe_3O_4 samples, with average size of 18 nm for Au and 14 nm for Fe_3O_4, was evaluated using a mammalian endothelial cell line, HeLa cells. Both Fe_3O_4 and Au-Fe_3O_4 nanoparticles showed low cytotoxic effects on the cancer cells (cell viability 85-92%), demonstrating their biomedical applicable possibilities.

In the ***Chapter 4***, ferromagnetic FePt nanoparticles were successfully synthesized from FePt (50:50) alloy rods using the pulsed plasma in ethanol. A1 phase of as synthesized FePt nanoparticles which has chemically disordered face-centered-cubic structure has been transformed into the ordered face-centered-tetragonal $L1_0$ –FePt after annealing at 400^{o}C for 1h. Along with phase change, the size and magnetic properties of FePt nanoparticles were changed. The average diameter of fcc –FePt was about 3 nm and the mean diameter of fct-$L1_0$ type FePt nanoparticles was 6 nm. VSM and SQUID analysis indicated the ferromagnetic behavior of A1 and $L1_0$-type FePt nanoparticles with coercivity of 750 and 1100 Oe, respectively.

In the ***Chapter 5***, synthesis and characterization of tin (Sn), indium (In), tin dioxide (SnO_2) and indium oxide (In_2O_3) nanoparticles produced from Sn-Sn and In-In electrode using the pulsed plasma in water were reported. The tetragonal Sn and In nanoparticles showed good crystallinity, with a spherical shape and an average diameter of 3-4 and 4.5-6.0 nm, respectively. Thermogravimetrical analysis revealed insignificant weight loss from the particles (2.4%), indicating their thermal stability. DSC measurements revealed a decrease in the melting temperature of the synthesised nanoparticles to 226.5°C (for Sn) and 152.7°C (for In), lower than that of the bulk material by 2.7%. SnO_2 and In_2O_3 nanoparticles were successfully obtained via further annealing (800^{o}C and 400^{o}C) of the as-synthesized Sn and In nanoparticles. Structural and morphological analysis indicated the full oxidation of Sn and In and size growth of oxide nanoparticles up to 15-25 nm. Despite the lower sample concentration of SnO_2 catalyst (0.1 g/L) in dye solution, the full degradation of methylene blue and methyl orange completed in 90 and 180 min, respectively. This shows a good photocatalytic activity of SnO_2 nanoparticles obtained by us.

In the ***Chapter 6***, general conclusion, awards, main papers of the presented work and acknowledgements were summarized.