

**PROCESS FOR THE CONTROLLED
PREPARATION OF A COMPOSITE OF
ULTRAFINE MAGNETIC PARTICLES
HOMOGENEOUSLY DISPERSED IN A
DIELECTRIC MATRIX**

FIELD OF THE INVENTION

The present invention relates to a process for forming bulk materials having magnetic particles dispersed in a non-magnetic matrix.

BACKGROUND OF THE INVENTION

Composite "granular metals" have been prepared by co-evaporating an immiscible metal and a non-metal under properly controlled conditions onto a suitable substrate to form an intimate mixture of the two constituent phases where the particle size of the metallic phase is of the order of nanometers. These composite materials exhibit properties which are not only related to the properties of the constituent phases but are also strongly composition and particle-size dependent. However, the nature of the co-evaporation procedure makes it most suitable for the production of thin films of the composite material.

SUMMARY OF THE INVENTION

The present invention is directed to the synthesis of bulk materials with very fine dispersions of metallic phases in a dielectric matrix. Practical applications for such bulk materials include high density magnetic recording media which require magnetic particles of ultrafine diameter. Bulk materials with high electrical resistivity may be used as replacements for ferrites in microwave components such as filters and resonators. Magneto-optic devices, using Kerr and Faraday effects, e.g., high resolution information storage and retrieval media, may advantageously use such bulk materials. Also, bulk materials as described above may be used in levitation devices such as magnetic bearings and as anti-radar coatings.

It is an object of the present invention to provide bulk materials with very fine dispersions of metallic phases in dielectric matrices.

It is further an object of the present invention to provide a metallic phase in a dielectric matrix wherein the particle size of the metallic phase is of the order of nanometers.

It is further an object of the present invention to provide a process of producing finely divided magnetic particles in a bulk non-magnetic matrix.

It is further an object of the present invention to provide a process for producing finely divided magnetic particles having a controlled magnetic particle size.

It is further an object of the present invention to provide a process which allows control over the magnetic state of magnetic particles during the synthesis of the particles in a non-magnetic matrix.

The present invention provides a process wherein finely divided magnetic particles are generated within a bulk nonmagnetic matrix so that the magnetic particle size and the magnetic state of the particles are controlled during synthesis through low temperature polymerization and oxidation and reduction reactions.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 shows an x-ray diffraction pattern for an as-cured 11% Fe+silica gel nanocomposite;

FIG. 2 shows a TEM micrograph of the hydrogen-treated 11% Fe+silica gel nanocomposite;

FIG. 3 shows a Mössbauer spectra for a 25% Fe+silica gel nanocomposite after (a) curing and (b) treating in a hydrogen atmosphere;

FIG. 4 shows the magnetic susceptibility, χ , and $1/\chi$ vs. temperature, T, data for a 40% Fe+silica gel nanocomposite wherein the dot-dashed line is a least squares fit to the $1/\chi$ data above 90 K and follows the relationship $1/\chi = 6536 + 142.7 \cdot T$ and wherein the inset shows the composition dependence of the intercept temperature, T_n ;

FIG. 5 shows the magnetic moment per Fe atom and Curie constant, C, (inset) vs. Fe content in the cured Fe+silica gel nanocomposites;

FIG. 6 shows magnetization vs. Field data for the hydrogen-treated 25% Fe+silica gel nanocomposite at 10K, measured after cooling to this temperature in zero applied field and after cooling in a 9 kOe field;

FIG. 7 shows room temperature Mössbauer patterns for (a) sample 10A and (b) sample 11B;

FIG. 8 shows Mössbauer patterns measured for the 10% Fe sample 10A at (a) 45 K, (b) 20 K, and (c) 4.2 K;

FIG. 9 shows magnetization vs. applied field data for sample 10A measured as the field was cycled between +10 kOe and -10 kOe at room temperature (open symbols) and 10 K (filled symbols);

FIG. 10 shows magnetization, M, (open symbols) and reciprocal magnetization (filled symbols) vs. temperature data for sample 10A measured during cooling in a 2 kOe applied field;

FIG. 11 shows scanning electron micrographs of samples (a)10A and (b)11B taken at 50,000X;

FIG. 12 shows room temperature Mössbauer patterns measured for sample 10A following treatments in (a) Hydrogen and (b) ammonia atmospheres;

FIG. 13 shows magnetization vs. applied field data for sample 10A at 300 K following a treatment in hydrogen gas; and

FIG. 14 shows magnetization vs. temperature data at 100 Oe applied field H_0 for the H_2 -treated sample containing 10% Fe as it was (a) cooled in H_0 and (b) warmed in H_0 following cooling in zero field.

DESCRIPTION OF THE INVENTION

According to the present invention, a process for producing bulk quantities of an iron-silica gel composite with control over the particle size, form, and magnetic state of the iron is provided. The process involves low temperature polymerization of a silicon alkoxide, a ferric compound, e.g., ferric nitrate, and water, generally under the influence of a catalyst. The chemical and magnetic states of the iron in the resultant composite can be modified in situ by exposure to suitable oxidizing or reducing agents at temperatures under 400° C. At these low temperatures, the formation of undesirable and/or non-magnetic phases such as Fe_2SiO_4 (fayalite) can be avoided.

According to the present invention, many different silicon alkoxides may be employed. Tetramethylorthosilicate (TMOS), tetraethylorthosilicate (TEOS), tetrapropylorthosilicate and tetraisopropylorthosilicate or mixtures thereof are preferred, although other silicon alkoxides may be used depending upon the specific