

The results shown in FIG. 10 indicate that small negative deviations from Curie-Weiss behavior do occur around 60 K, and the possibility of an antiferromagnetic transition is suggested by the intercept at  $\sim -13$  K of the extrapolated high temperature behavior.

FIG. 11 shows scanning electron micrographs of samples (a) 10A and (b) 11B taken at 50,000x showing the interconnected network of pores (dark) existing throughout the matrix (light) of these materials.

Series A samples containing 25 and 30% Fe possessed identical room temperature Mössbauer spectra to that shown for specimen 10A in FIG. 7a, indicating a small particle size for the Fe-containing regions in these samples. However, larger particle sizes were found to be present in specimen 18A as it possessed a spectra similar to that shown in FIG. 7b. For series B samples, 11B and 18B possessed room temperature spectra containing a magnetically ordered component while that measured for 25B only contained the large central doublet. Consequently, the Fe and H<sub>2</sub>O contents of the preparation solutions are not the only parameters affecting the particle size of the iron in these nanocomposites. A correlation, however, was observed between the morphology of the samples and their high temperature Mössbauer patterns. All the materials prepared here possessed an interconnected network of pores throughout the matrix as shown in FIG. 11. However, those materials which possessed large ( $\geq 45$  nm) pore diameters as shown in FIG. 11b for sample 11B also possessed Mössbauer spectra with a magnetically ordered component visible at room temperature. If the Fe-containing regions are localized in the pore areas, one might expect their sizes to be proportional to the amount of cation (Fe) in the pores during the drying process and, consequently, to the pore volume. Note from FIG. 11 that even for those materials with large pore size (and therefore possessing "large" iron particle sizes), the particle sizes of the Fe-regions are still too small ( $\leq 20$  nm) to be imaged in the conventional SEM.

Evidence was found for localizing the Fe-containing regions in the pore areas by subjecting separate pieces of the dried nanocomposite 10A to treatments in 1 atmosphere of hydrogen gas (378 C. for 20 hours) and 1 atmosphere of NH<sub>3</sub> gas (460 C. for 1 hour following the previous hydrogen treatment). Due to the interconnection of the pore areas, any Fe-regions located in these areas would be likely to change their form during these treatments. The Mössbauer spectra for these altered samples, displayed in FIG. 12, indeed show distinct changes in the form of the iron in these nanocomposites. The hydrogen treatment resulted in a pattern with a slightly broadened strong central doublet with an admixture of a less intense peak having a greater isomer shift; the NH<sub>3</sub> treated sample resulted in a material primarily possessing a much larger isomer shift of 1.4 mm/sec. At room temperature both of these altered materials possessed increased magnetization values (by factors of 2 and 3 respectively for the NH<sub>3</sub> and H<sub>2</sub> treated specimens) and changed their magnetic state from paramagnetic to superparamagnetic (see FIG. 13). Even though the Mössbauer pattern for the H<sub>2</sub>-treated sample was not remarkably different from the untreated sample 10A (FIG. 7a), its magnetic state is quite different. In fact, FIG. 14 shows the H<sub>2</sub>-treated 10% Fe sample possesses thermomagnetic history effects at temperatures below about 30 K. These effects combined with the observation of a displaced hysteresis loop

along the field axis at low temperatures are characteristic of spin glass magnetic behavior. This magnetic behavior results when the magnetic spins on the Fe become frozen in their high temperature orientation when cooled to below their freezing temperature (which in this case is  $\sim 30$  K).

FIG. 13 shows magnetization vs. applied field data for sample 10A at 300 K following a treatment in hydrogen gas. The room temperature data measured for the NH<sub>3</sub>-treated material looked similar, but with a maximum magnetization at 9 kOe of 0.2 emu/g.

FIG. 14 shows magnetization vs. temperature data at 100 Oe applied field for the H<sub>2</sub>-treated sample containing 10% Fe measured as it was either (a) cooled in the measuring field (open symbols) or (b) warmed in the measuring field following a cool to the lowest temperature in zero field (filled symbols). Arrows indicate the direction of measurement.

In another aspect of the present invention, a magneto-optical device is provided by forming a flat optical reflector from the material described above. Polarized light is reflected from the reflector surface. Upon application of a magnetic field to the reflector, the polarization of the light may be changed (Kerr effect). Generally, any of the bulk materials produced in accordance with the present invention may be cast into a monolith having the shape of an optical flat. Once cured, the material forms an amber colored glass. Upon reduction with an appropriate chemical agent, the iron in the material is brought into a ferromagnetic state. The reduced material is highly reflective of visible light, particularly when the iron loadings approach 30%. Due to the controlled polarization of the reflected light, applications for high resolution information storage and retrieval are possible.

Alternatively, information storage and retrieval using these materials in the form of an optical window, may be accomplished by using magnetically controlled polarization changes in a transmitted beam of electromagnetic radiation, e.g., light, x-rays, gamma rays, microwaves or radiofrequency waves (Faraday effect).

This class of nanocomposites has an additional potential for use as selective filters for electromagnetic radiation. For example, the as-cured 10% Fe/SiO<sub>2</sub> material is amber in color and transparent to visible light. Upon chemical reduction or oxidation the material becomes respectively, either black or orange and is somewhat opaque to visible radiation. This controlled change in both color and in transmissivity is a function of the concentration, ultrafine particle size and chemical state of the iron.

It is to be understood that the present invention is not limited to the specific embodiments described herein. It will be appreciated by those skilled in the art that additions, modifications, substitutions and deletions may be made without departing from the scope of the invention defined in the appended claims.

What is claimed is:

1. A process for synthesizing a bulk material having magnetic particles dispersed in a non-magnetic matrix, comprising the steps of:

- a) polymerizing silicon alkoxide in solution with at least one iron compound to form a gel;
- b) curing said gel to form a porous hydrolyzed silicon product which contains iron;
- c) subsequently contacting the porous hydrolyzed silicon product with an oxidizing agent at a temper-