US005316699A

United States Patent [19]

Ritter et al.

[11] Patent Number: 5,316,699

[45] Date of Patent: May 31, 1994

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

[75] Inventors: **Joseph J. Ritter**, Mt. Airy; **Robert D. Shull**, Boyds, both of Md.

[73] Assignee: **The United States of America as repesented by the Secretary of Commerce**, Washington, D.C.

[21] Appl. No.: **501,981**

[22] Filed: **Mar. 28, 1990**

[51] **Int. Cl.**[5] **G02B 5/20**; B01J 13/00; C08L 83/00

[52] **U.S. Cl.** **252/584**; 252/582; 252/587; 252/588; 252/315.6; 524/429; 524/431; 524/588

[58] **Field of Search** 252/582, 584, 587, 588, 252/315.01, 315.6; 524/429, 431, 588

[56] **References Cited**

U.S. PATENT DOCUMENTS

2,496,265	2/1950	Bilisoly	428/553
3,188,217	6/1965	Elmer et al.	252/584
3,261,802	7/1966	Bobear	524/431
3,284,369	11/1966	Bergna et al.	252/454
3,474,039	10/1969	Newing	252/584
3,551,352	12/1970	Carr et al.	252/439
3,791,808	2/1974	Thomas	252/584
4,113,658	9/1978	Geus	252/454
4,529,749	7/1985	Favre et al.	524/588
4,678,717	7/1987	Nickola et al.	428/553
4,764,499	8/1988	Vanderspurt et al.	502/258
4,959,247	9/1990	Moser et al.	427/126.5

OTHER PUBLICATIONS

B. Abeles, Appl. Solid State Sci. 6, 1 (1976).

R. A. Roy and R. Roy, Mat. Res. Bull. 19, 169 (1984).

E. J. A. Pope and J. D. Mackenzie, J. Non-Crys. Sol. 87, 185 (1986).

Shull et al., *Multicomponent Ultrafine Microstructures* (edited by L. E. McCandlish et al., MRS Symposium Proc., vol. 132 (1989) p. 179.

J. B. Chan et al., J. Mater. Res., 4, (1989), 693.

Schmitt et al., Chemical Engineering at Supercritical Fluid Conditions, Paulaites et al., eds., Ann Arbor Science Publishers, Ann Arbor, MI (1983) p. 445.

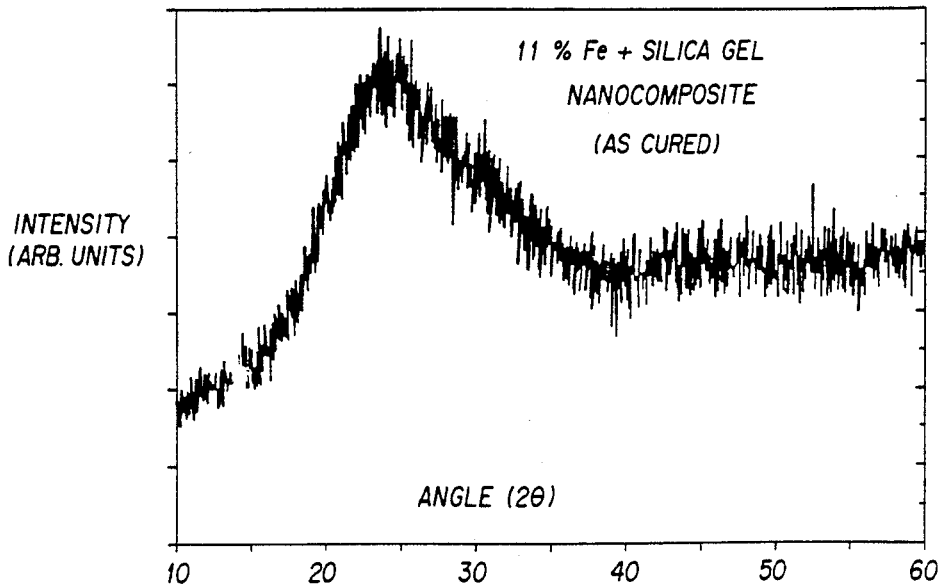
Kein et al., Ultrastructure Processing of Ceramics Glasses and Composites, Hench et al., eds., John Wiley and Sons, N.Y., p. 88, (1984).

Larry L. Hench, Science of Ceramic Chemical Processing, Hench et al., eds., John Wiley and Sons, N.Y., p. 52, (1986).

Primary Examiner—Philip Tucker

Attorney, Agent, or Firm—Oliff & Berridge

[57] **ABSTRACT**

A chemical process for producing bulk quantities of an iron-silica gel composite in which particle size, form, and magnetic state of the iron can be selected. The process involves polymerizing an ethanolic solution of tetraethylorthosilicate, ferric nitrate and water at low temperature under the influence of an HF catalyst. The chemical and magnetic states of the iron in the resultant composite are modified in situ by exposure to suitable oxidizing or reducing agents at temperatures under 400° C. Iron-containing particles of less than 200 Å diameter, homogeneously dispersed in silica matrices may be prepared in paramagnetic, superparamagnetic, ferrimagnetic and ferromagnetic states.

17 Claims, 12 Drawing Sheets