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SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE γ-Fe_2O_3 BY COMBUSTION METHOD

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ABSTRACT

The γ-Fe_2O_3 was prepared using combustion method with iron acetylacetonate and urea as starting materials. The prepared γ -Fe_2O_3 has nanocrystalline particles at 350°C. Proszki computer programme was used to determine the structure of the prepared γ -Fe_2O_3 from the XRD pattern and revealed that γ -Fe_2O_3 was tetragonal having unit cell parameters; a = 8.33942 A^0; c = 25.01156A^0. Electron microscopic studies gave the surface morphology of the prepared γ-Fe_2O_3. The particles of γ-Fe_2O_3 were less than 300nm from the scanning electron microscopy while the transmission electron microscopy revealed near-acircular particles of γ -Fe_2O_3 of mean particle size of 200nm. The magnetic properties of this important oxide measured reveal that γ -Fe_2O_3 was ferromagnetic.

Keywords: γ -Fe_2O_3, urea, nanocrystalline, combustion, ferromagnetic.

INTRODUCTION

Nanocrystalline materials have increasingly attracted interest because of their distinctive properties and applications. These materials provide materials scientists and engineers a unique opportunity to obtain materials having properties that are otherwise unachievable with equilibrium materials (Lau et al, 1996). Ferrite-based magnetic materials like γ -Fe_2O_3 and Fe_3O_4 are also very important materials for practical applications. They are effectively utilized as magnetic core materials with low iron loss for optomagnetic devices, bubble memory devices, vertical recording magnetic materials etc. Furthermore, Revindranathan and Patil (1986) reported that γ -Fe_2O_3 is the most widely used recording materials for magnetic tapes and disks.

Takahasi et al (1991) prepared γ -Fe_2O_3 using the sol-gel method involving iron nitrate dissolved in ethylene glycol as the starting material. The preparation of γ -Fe_2O_3 via combustion method from iron nitrate and malonic acid dihydrazide in the molar ratio 1.0:0.94 was also reported by Suresh and Patil (1993). However, it is interesting to note that a particular material may have different electrical, magnetic and structural properties depending upon the method of preparation, starting materials and temperature (Li et al, 1994).

Combustion synthesis, also called self propagating high-temperature synthesis, provides a simple and rapid means of preparing inorganic materials, many of which are technologically important (Mahesh et al, 1992). This method involves a highly exothermic reaction between an oxidizer (such as a mixture of metal nitrates) and a fuel (such as urea, glycine, hydrazine and their derivatives) which causes spontaneous combustion. In this way, the firing temperature and the time required to obtain the products are reduced. The main aim of this type of combustion reaction is the continuous formation of a well-crystallized single-phase fine powders utilizing both external heating and thermal energy released during combustion. A noteworthy feature of this method is that it enables one to produce inorganic oxide materials in the form of sub-micrometre to nanoscale ceramic powders.

This paper reports for the first time the preparation of nanocrystalline γ -Fe_2O_3 using iron acetylacetonate and urea as starting materials via the combustion method.

EXPERIMENTAL

Equimolar amounts of urea and iron acetylacetonate were mixed together in a

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beaker. The mixture when rapidly heated at 350^{0}C, initially boils then froths and ignites to yield fine-particle of γ -Fe_2O_3 in less than 5 minutes. The prepared γ -Fe_2O_3 was characterized by X-ray diffraction, scanning electron microscopy (SEM), electron diffraction (ED), transmission electron microscopy (TEM) and magnetic measurements. A Seifert 300 X-ray powder diffractometer with Cu-K α radiation was employed for X-ray diffraction studies. A Leica S440i SEM was used for microstructure studies. TEM and ED images were recorded with a JEOL 3010 microscope. The saturation magnetization of γ -Fe_2O_3 at room temperature was measured using the vibrating sample magnetometer (VSM, EG & G Princeton Applied Research Model 155). The BET surface area measurement was done by nitrogen adsorption employing a Micromeritics Accusorb 2100E instrument.

RESULTS AND DISCUSSION

The formation of γ -Fe_2O_3 (by the method described) is confirmed by the characteristic powder XRD pattern (Fig. 1). The XRD pattern shows considerable line broadening, indicating the fine-particle nature of the γ -Fe_2O_3. Fig. 1 is also consistent with JCPDS file number 33-669. Proszki computer program reveals that the prepared γ-Fe_2O_3 is tetragonal having unit cell parameters (a = 8.33942 A^0;c= 25.01156 A^0). The SEM photograph (Fig. 2) reveals that the particles of the prepared γ -Fe_2O_3 are less than about 300nm. However, the TEM

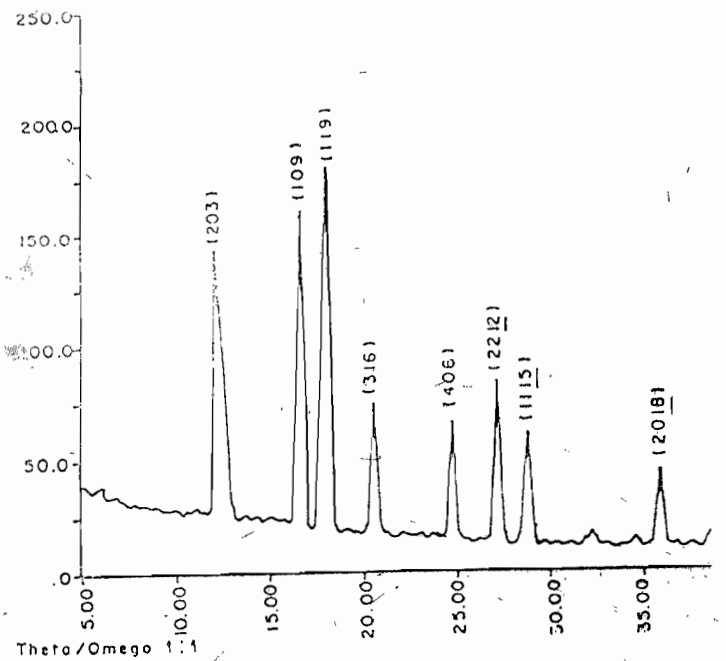

Fig. 1 The XRD pattern of γ -Fe_2O_3 obtained by combustion method.

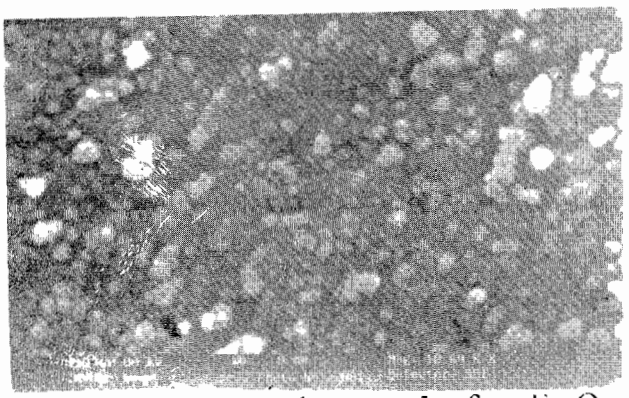

Fig. 2 The SEM photograph of γ -Fe_2O_3 obtained by combustion method.

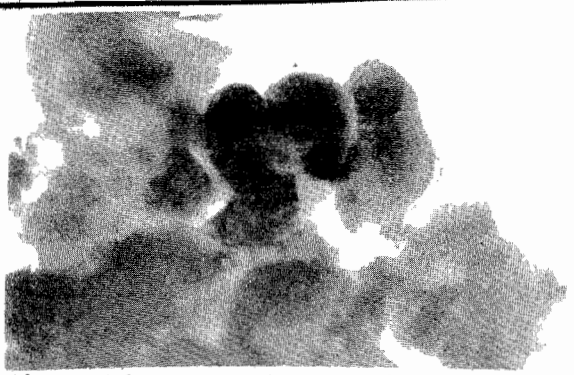

Fig. 3 The TEM photograph of γ -Fe_2O_3 obtained by combustion method.

Fig. 4 The ED pattern of γ -Fe_2O_3 obtained by combustion method.

(Fig. 3) which shows the near-acircular particles of the prepared γ -Fe_2O_3 gives the mean particle size as 200nm. The crystalline nature of the γ -Fe_2O_3 is confirmed from the ED pattern (Fig. 4) which exhibits white spots. Amorphous materials do not exhibit any white spot. The BET surface area measurement by nitrogen adsorption reveals that the surface area of the prepared γ -Fe_2O_3 is 10m^2/g. Fig. 5 shows that the γ -Fe_2O_3 is ferromagnetic as revealed from the plot of magnetization versus field where a hysteresis loop is observed. The magnetic moment is obtained as 0.0027emu and the coercivity as 660.1 Oer while the saturation magnetization at room temperature is 0.0031 emu The

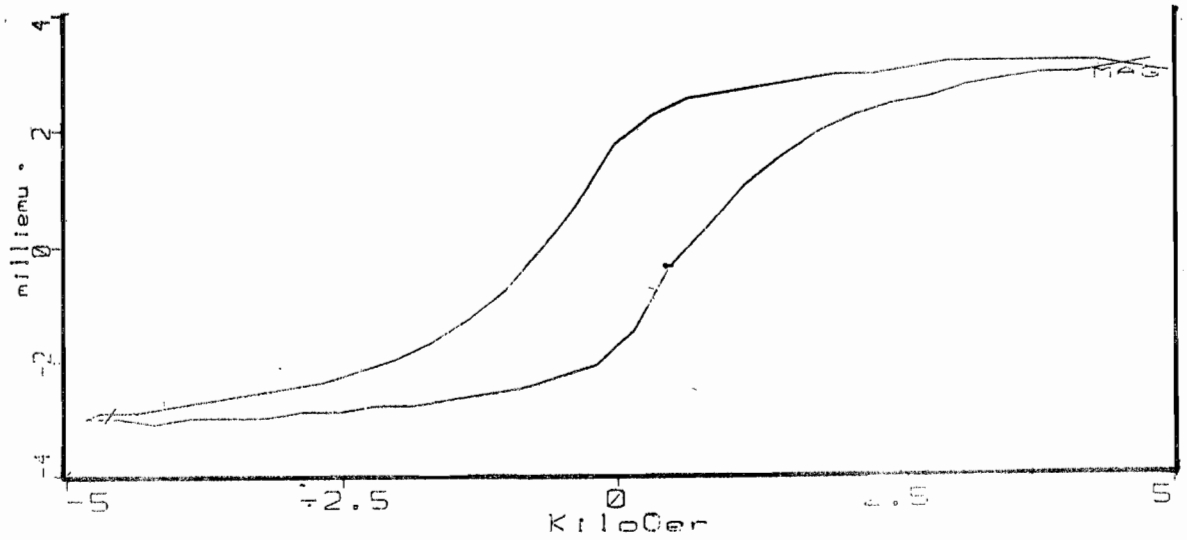

Fig. 5 The plot of magnetization versus field for γ -Fe_2O_3.

combustion synthesis of nanoscale magnetic iron oxide particles is a facile and useful method and is not only rapid but also safe. The combustion method has all the advantages of the wet chemical methods such as atomic level doping of desired dopants, e.g. Co^{2+} ions, which is known to improve coercivity (Suresh and Patil, 1993).

CONCLUSION

The γ -Fe_2O_3 has been synthesized from iron acetylacetonate and urea as starting materials via the combustion method for the first time. The synthesized γ -Fe_2O_3 is a tetragonal ferromagnetic nanoscale material. The coercivity of the prepared γ -Fe_2O_3 is substantial.

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