International Journal of Current Engineering and Technology
E-ISSN 2277 – 4106, P-ISSN 2347 – 5161
Available at http://inpressco.com/category/ijcet

Research Article

Synthesis of α-Fe_2O_3 Nanostructure Films Prepared by Laser Assisted Spray Pyrolysis

Bahaa T. Chiad[†], Zainab S. Sadeq[†*], Dunia K. Mahdi[†], and Fatin H. Mohammed[‡]

[†]Physics Department, Baghdad University, College of Science, Baghdad, Iraq
[‡]Physics Department, Al Mustansiriya University, College of Science, Baghdad, Iraq

Accepted 03 Sept 2015, Available online 17 Sept 2015, **Vol.5, No.5 (Oct 2015)**

Abstract

This work describes the influence of the concentration of precursor solution for prepared Nanocrystalline α-Fe_2O_3 via laser assisted spray pyrolysis. The XRD studies show that the formation of pure α-Fe_2O_3 without any impurity phases. The SEM studies show that the decrease in precursor concentration can effectively decreases the particle sizes.

Keywords: *Laser Assisted Spray pyrolysis, Nanostructures α-Fe_2O_3*

1. Introduction

As one of the most important transition metal oxides with a band gap of 2.2 eV, hematite (αFe_2O_3) have received extensive attention due to its good intrinsic physical and chemical properties, such as its low cost, stability under ambient conditions, environmentally friendly properties (Wu *et al*, 2006) Due to these properties, αFe_2O_3 nanostructures can have wide applications in many fields including magnetic recording materials, catalysis, optical devices, gas sensors, photochemical and pigments (Wei *et al*, 2006), (Tang *et al*, 2006) , (Huo *et al* 2000), (Cao *et al*, 2010), (Faust *et al*, 1989).

2. Experimental

Fe_2O_3 nanoparticle coatings were synthesized by the laser assisted spray pyrolysis method. Laser Assisted Spray Pyrolysis is a new technique that one can use to get finer and more concentrated droplets of precursor compared to regular spray pyrolysis. In the Laser Assisted Spray Pyrolysis (LASP), the droplets interact with a continuous wave (CW) CO_2 beam as they come out of the nozzle.

The precursor of high purity was prepared by dissolving of Iron (III) chloride anhydrous $FeCl_3$ (Sigma-Aldrich Labor chemikallen Gmbh) using double distilled water as a solvent, different concentration of the aqueous solution of Iron (III) chloride was used (in order to arrive the perfect solution use the Magnetic stirrer).

*Corresponding author: **Zainab S. Sadeq**

After preparing the aqueous solution of $FeCl_3$ locate in the ultrasonic nebulizer with frequency 1.7MHZ to sprayed on the glass substrate (4oo ± 5) °C, it was kept constant during the deposition time (5min). A 3W CW CO_2 laser with wavelength 10.6 μm, sulfur hexafluoride (SF_6) is used as carrier gas for aerosol transport. Other preparative parameters were spray rate: 5 cc/min; nozzle to substrate distance: 33 cm; and nozzle diameter: 0.05 cm, kept constant for all experiments.

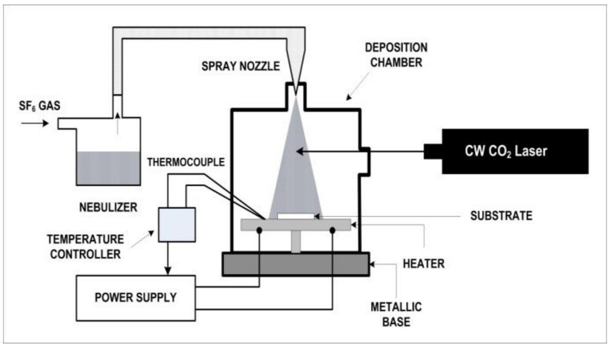

Fig. 1 Schematic diagram of the experimental setup

Note that. The deposition was carried out inside of deposition chamber, it made from stainless steel. Figure (1) shows the schematic diagram of the experimental apparatus of Laser Assisted Spray Pyrolysis (LASP) technique. The glass substrates are placed on the hot plate at distance 14 cm from the nozzle of the ultrasonic nebulizer with deposition time (5 min) to get good layers of Fe_2o_3 thin films with various thicknesses dependent on concentration of procurer solution of $FeCl_3$. Table (1) presented the optimized parameters used in this work.

Table 1 Optimized spray parameters used for the preparation of α-Fe_2O_3 films

Spray mode	Ultrasonic nebulizer
Ultrasonic frequency	1.7(MHz)
Laser	CO_2 laser with10.6 µm wavelength
Carrier gas	SF6
Solution flow rate	3.5 L/min
Substrate-Nozzel distance	10 cm
Solvent	Distilled water
Precursor	Ferric chloride
Concentration	(0.1, 0.15,0.2,0.25)mol/L
Deposition temperature	400◦C
Substrate	Glass

3. Results and Discussion

3.1 Structural properties

Figure (2) shows the X-ray pattern of the sample prepared at 400°C for 5min at concentration 0.2M of aqueous solution of $FeCl_3$.

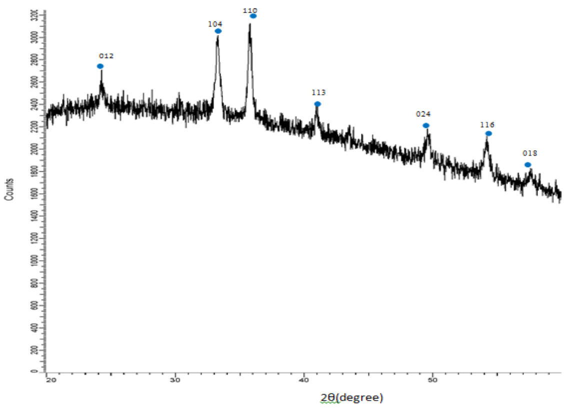

Fig.2 X-ray diffraction pattern of Fe_2O_3 nanograined sample prepared by laser assisted spray pyrolysis

The pattern exhibits the characteristic XRD pattern of hematite (α-Fe_2O_3) in accordance with data from the ASTM (American Society of Testing Materials) cards. The Scans were performed over 2θ = 20-60˚ for each sample. Figure (2) indicate crystalline peaks of α-Fe_2O_3 (012), (104), (110), (112), (024), (116) and (118). The crystallite size was calculated using the Scherer equation, and was found to be 30nm for this film. Thus, the laser heating is affecting on the structural of the film leading to the film of pure nano-crystalline structure of α-Fe_2O_3 without any impurity phases. Our results agree with the results of R. Suresh *et al* (Suresh *et al*, 2009).

We know that, with reducing the concentration of the precursor, the percentage of the compound present in a droplet also decreases. The samples prepared as a function of precursor concentrations of (0.1, 0.15, 0.2, and 0.25) M have been analyzed by SEM to study the relation between concentration and particle size in the LASP process. To investigate the initial growth of the particles precursor droplets were deposited for 5min on glass substrate, the substrate was heated to 400 ºC. The boiling point of de ionized water is 100ºC. Therefore, if the laser heating is sufficient to heat the carrier gas SF6 above this temperature, complete evaporation of the solvent can be expected. The morphology of the hematite and its results are presented in figures (3)-(6) at various concentration (0.1, 0.15, 0.2, 0.25) M respectively .Table (2) illustrates the effect of precursor concentration of the solution on the size and on the shape of Fe_2O_3 which prepared by LASP technique. We observed from the results of the SEM studied that the reducing in precursor concentration lead to get the smaller particle sizes. Our results of affecting of laser beam via LASP technique agree with results of G. S. Dedigamuwa (Dedigamuwa, 2005).

Table 2 Illustrates the effect of precursor concentrations on size and shape of Fe_2O_3 particles which prepared LASP techniques

Concentration of precursor solution (M)	Particle size (nm) by using LASP	Shape of particles by LASP
0.1	13-38	Cubic +spherical
0.15	17-48	Cubic +spherical
0.2	20-56	Cubic +spherical
0.25	26-64	Cubic+ spherical

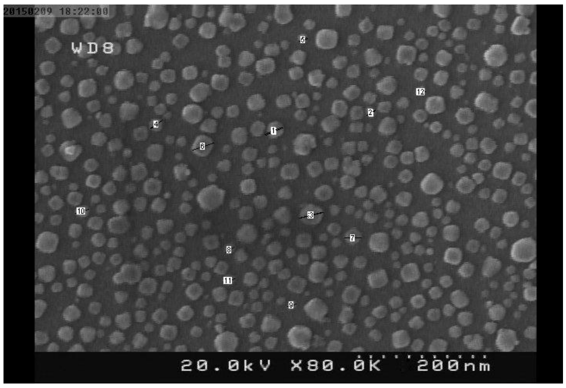

Fig.3 SEM image of Fe_2O_3 samples obtained at precursor concentration of 0.1M by LASP.

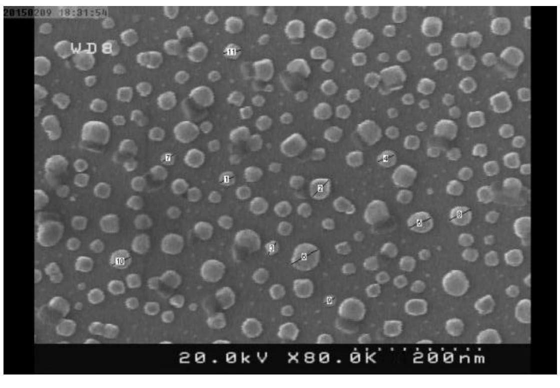

Fig.4 SEM image of Fe_2O_3 samples obtained at precursor concentration of 0.15M by LASP.

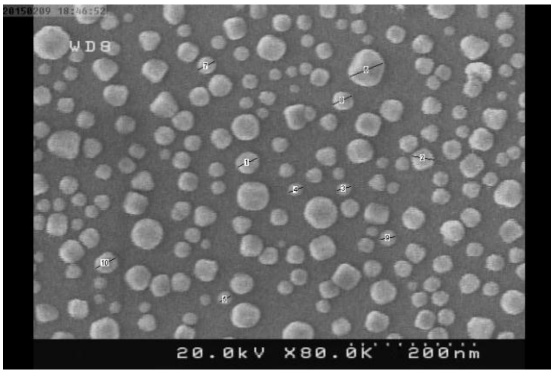

Fig.5 SEM image of Fe_2O_3 samples obtained at precursor concentration of 0.2M by LASP.

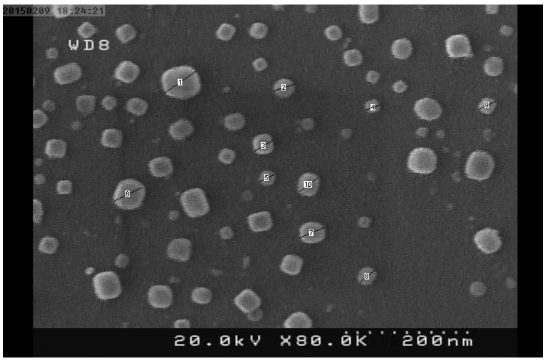

Fig.6 SEM image of Fe_2O_3 samples obtained at precursor concentration of 0.25M by LASP.

3.2 Optical Energy Gap Properties of Fe_2O_3 films

In general, the concentration of precursor solution also affects the other properties of the film such as direct band gap. The optical band gap (E_g) was derived assuming a direct transition between the edge of the valence and conduction band. The plot of $(\alpha hv)^2$ as a function of the energy of incident radiation has been shown in Figures (7) - (10). The energy of band gap is obtained from intercept of the extra plotting linear part of the curve with the energy axis. The direct band gap of the α -Fe_2O_3 films slightly decrease from (2.83 - 2.65) eV with increases of precursor solution concentrations for the prepared films as shown in the above figures. These result agreement with that of A. Barranon (Barranon, 2009).

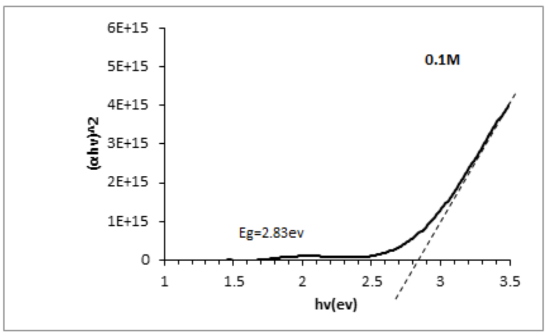

Fig.7 Variation of $(\alpha hv)^2$ with photon energy (hv) of Fe_2O_3 films for 0.1 M of concentration of precursor for the samples which prepared by LASP.

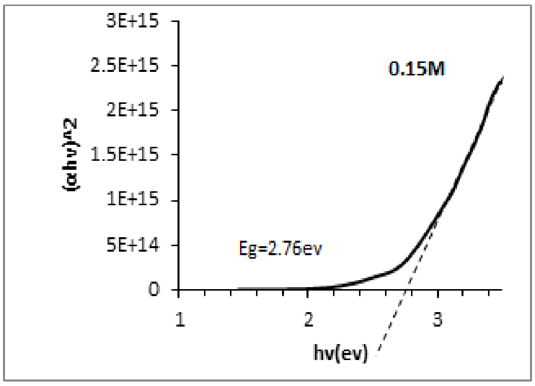

Fig.8 Variation of $(\alpha hv)^2$ with photon energy (hv) of Fe_2O_3 films for 0.15 M of concentration of precursor for the samples which prepared by LASP.

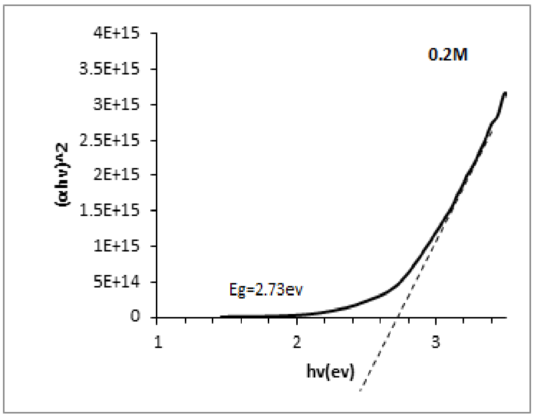

Fig.9 Variation of $(\alpha hv)^2$ with photon energy (hv) of Fe_2O_3 films for 0.2 M of concentration of precursor for the samples which prepared by LASP.

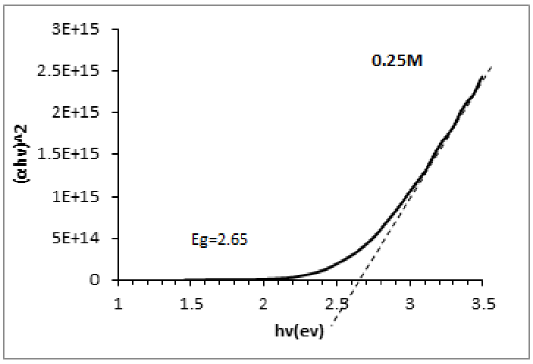

Fig.10 Variation of $(\alpha hv)^2$ with photon energy (hv) of Fe_2O_3 films for 0.25 M of concentration of precursor for the samples which prepared by LASP.

Conclusions

Hematite films (α-Fe_2O_3) were deposited by laser assisted spray pyrolysis at substrate temperature (400° C) with various concentration of precursor solution. Structural analysis of the films showed that: the films had pure nano-crystallite size and according to SEM analysis ,the particle size decreases with decreases the concentration of precursor solution from 26nm to nm13. The films exhibited the presence direct band gaps had a value of (2.83-2.65) eV dependent on precursor concentration.

References

Q. Wei, Z. Li, Z. Zhang and Q. Zhou, (2009), Facile Synthesis of α-Fe2O3 Nanostructured Films with Controlled Morphology, *Materials Transactions* 50, 1351 -1354.

B. Tang, G. L. Wang, L. H. Zhuo, J. C. Ge and L. J. Cui, (2006), Facile Route to α -FeOOH and α -Fe_2O_3 Nanorods and Magnetic Property of α -Fe_2O_3 Nanorods", *Inorg. Chem.* 45, 5196- 5200.

L. H. Huo, W. Li, L. H. Lu, H. N. Cui, S. Q. Xi, J. Wang, B. Zhao, Y. C. Shen and Z. H. Lu, (2000), Preparation, structure, and properties of three-dimensional ordered alpha-Fe_2O_3 nanoparticulate film, *Chem. Mater*. 12, 790-794.

S. Cao, Y.J. Zhu, G.F. Cheng and Y.H. Huang, (2010), Preparation and photocatalytic property of α-Fe_2O_3 hollow core/shell hierarchical nanostructures, *Journal of Physics and Chemistry of Solids* 71, 1680-1683.

B. C. Faust, M. R. Hoffmann and D. W. Bahnemann, (1989), Photocatalytic oxidation of sulfur dioxide in aqueous suspensions of **α-** Fe_2O_3., *J. Phys. Chem*. 93, 6371.

R. Suresh, L. Vijayalakshmi, A. Stephenand and V. Narayanan, (2009), Hydrothermal Synthesis and Characterization of Cobalt Doped α-Fe_2O_3", International Conference on Advanced Nanomaterials and Nanotechnology, Conference Proceedings, 1276, 362-367.

G. S. Dedigamuwa, (2005), Formation of nanocoatings by laser-assisted spray pyrolysis and laser ablatio n on 2d gold nanotemplates", Master thesis submitted to the Department of Physics, College of Arts and Sciences, University of South Florida.

A. Barranon, (2009), Research in Nanotechnology Developments, Study of the properties of iron oxide nanostructures, chapter 17, 183-194.