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# Growth, structural and magnetic properties of Gadolinium oxide ($Gd_2O_3$) thin films

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## ABSTRACT
$Gd_{2O3}$ nanoparticles thin films obtain through the spin coating techniques deposited on substrate of p-Si(100) . The structural properties of the thin film were measured by X-ray diffraction technology (XRD) to know the crystal structure and the effect of the difference of different annealing temperatures on the crystal growth of these samples, and the scanning electron microscopy examinations (SEM) and the magnetic properties were studied using the Vibrating Sample Magnetometer(VSM) of Various annealing temperatures (500,600,700,800)℃. The results of the X-ray diffraction showed that all the prepared membranes have a multi-crystalline structure with different diffraction values and directions and the $Gd_2O_3$ thin film were of the cubic type and it was noted that by increasing the annealing temperature we get the best crystalline development and the results of SEM showed that the shape of the deposited nanoparticles was spherical, and we note an increase in the saturation magnetization (MS) about (0.1421 - 0.1811) emu / g with increased of annealing temperature.

## 1- Introduction
Rare earth elements are a group of seventeen chemical elements in the periodic table. After discovering its composition the same sediments that the lanthanides form and have similar chemical properties arising from their 4f electron. The oxides of the lanthanides group ($Ln_2O_3$) are very thermally stable, they are at high temperatures and are characterized by their extreme heat resistance[1-5].Gadolinium atomic number 64 and is a white, silver metal of a group of rare earth elements. Gadolinium oxide ($Gd_2O_3$) is that has chemical strength and thermal stability and low phonon energy [6]. In ambient conditions, these materials exist in three different polymorphic forms of A ,B, and C. The A phase has a hexagonal structure, the B phase has a monoclinic structure and the C phase has a cubic structure as With an increase in temperature ,phase transitions usually follow the sequence of C-B-A for most $RE_2O_3$ with medium-sized action's [7]. In addition reported the C-B and B-A phase transitions in $Gd_2O_3$ at the temperatures of about 1500K and 2443K,respectively [8]. The $Gd^{3+}$ ion has a $^8S_{7/2}$ ground state, it carries the characteristics of ferromagnetic materials at room temperature[9,10]. The Curie point is 293 k (20 ° C, or 68 ° F). Above this temperature the material becomes a diamagnetic [11,12]. Spin coating processes of colloidal suspensions are carried out in four different stages, the first occurs quickly and the second takes a long time. In the first stage, colloidal liquid is deposited as a drop on a fixed substrate. In the second stage, the substrate is accelerated to a specific rotational speed and the liquid diffuses to form the film approximately. In the third stage, the liquid spreads out and softens, and is controlled by centrifugal force and viscous shear force. In the fourth stage, the film becomes delicate with the effect of fumigation, which reduces the thickness of the film. The transition from the third to the fourth stage depends on the properties of the liquid used in the coating. For colloidal suspension, the thickness of thin films can decrease during this fourth stage with the same particle size by the influence of capillary forces that affect the aggregation of the particles[13]. In this work, we prepared $Gd_2O_3$ nanoparticles by the sol-gel technique, then obtained thin films at different temperatures of spin coating technique and studied their structural and magnetic properties.

## 2- Method of Experimental
### 2-1 Sol-Gel Processing of $Gd_2O_3$
A simple relating to procedure is carried out to grow quality $Gd_2O_3$. During the first step mixing 0.283 g of

$Gdd_2O_3$ powder for 1 h 99.9% party with 40 ml of distilled water followed by drop wise addition of dilute $HNO_3$ until reach a clear. The pH of the mixture is then adjusted to 13.1 by adding drops of KOH solution with constant stirring for three horse finally sol is obtained. The chemical reaction steps and flow chart diagram of hydrothermally synthesized $Gd_2O_3$ are described by following equations and fig(1)respectively .

***Acidification:*** **$Gd_2O_3 + 6HNO_3 \longrightarrow 2Gd(NO_3)_3 + 3\,H2O$ ---- (2-1)**

***Hydrolysis*:** **$Gd(NO_3)_3 + 3\,kOH \longrightarrow Gd(OH)_3 + 3\,kNO_3$ -----(2-2)**

***Dehydration*:** **$2\,Gd(OH)_3 \longrightarrow Gd_2O_3$ (Nano powder) + $3\,H_2O$ ----(2-3)**

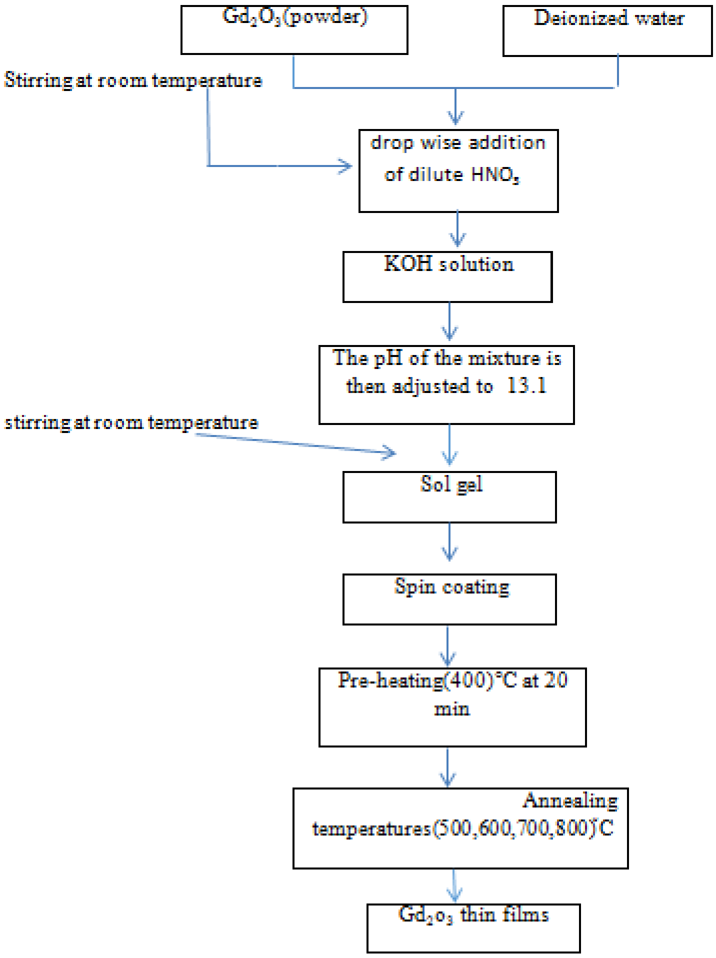


**Fig.1: Chart showing preparation of the sol-gel and thin film deposition**

**2-2 Film deposition**

The $Gd_2O_3$ nanomaterial was deposited on substrate of p- Si (100). Before deposition , substrate were cleaned with dilute HCL, acetone ,ethanol and distilled water. The films has deposited on substrate by spin coating at room temperature with rate of 3000 rpm for 30 s. After each coating the deposited films were dried at (400° C)at (20 min) the coating and drying procedures were four times, all the films were annealed for 1h at( 500,600, 700 and 800) ° C. The crystallographic information was revealed through x-ray diffraction type (SHIMADZU) that employs a Cu $K_\alpha$ source ($\lambda$=0.1543 nm).The crystallite size were calculate using Scherrer equation (1) [14]:

**$Cs = h\,\lambda / \beta\cos\Theta$ ---------- (1)**

Where Cs the crystallite size , h is the factor of microstructure (K =0.94), $\lambda$ is the wavelength of radiation, $\beta$ is the full-width at half-maximum (FWHM) and $\Theta$ is the diffraction angle. An examination VSM (CM120 Philips) was conducted to study the magnetic properties of samples prepared from $Gd_2O_3$ nanostructure. The saturation magnetization (M*S*) is essentially the limit value to which the curve tends within the high-field region, and is reached when all the magnetic moments in the material are aligned with the external field.

## 3- Results and Discussion

### 3-1 Structural Properties

the XRD patterns of Gd2O3 films fig (2) deposited on substrate p-Si (1 00) heat-treated at temperature

(500–800)C. For films annealed at higher temperature intense and typical diffraction peaks of cubic $Gd_2O_3$ with spatial group Ia3 (global card (00-012-0797))[15] appear. The crystallite size of $Gd_2O_3$ in films was estimated following the equation(1). Oriented growth of (431) face has been observed. When the annealing temperature was increased to 700℃, the films transformed from (2 2 2) orientation dominated to (4 0 0) orientation dominated. The transformation of orientation has been also observed in the electron diffraction patterns exhibit mainly rings associated to (2 2 2), (4 0 0), (4 4 0) and (6 2 2) planes of $Gd_2O_3$ cubic phase, which were already observed by XRD experiment on a multiplayer waveguide heat treated in the same conditions[16,17]. The annealing can induce the nucleation of small grains (defect free). If the annealing is long enough/high enough temperature continued growth of these new grains (defect free ) may eventually become larger than the original grain size. So one can expect annealing to induce a grain size reduction (initially, incomplete recrystallization) and then an ultimately (possibly) larger grain size[18,19]. And the appearance of vertices indicates that the installation of samples deposited with different annealing temperature is a multi-crystalline structure because there is more than one peak . A small displacement of some peaks was also observed, and this may be due to mechanical stress [20].

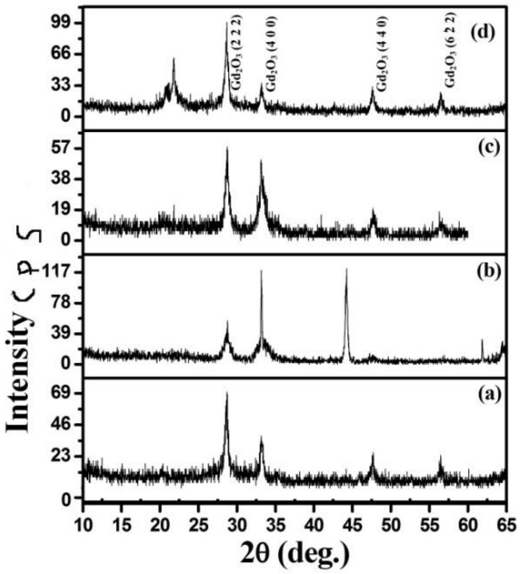


**Fig 2: XRD patterns of the Gd2O3 thin films deposited on substrate of p- Si (100) a-(500℃ ),b- (600℃) , c-(700℃) and d-(800℃)**

**3-2 scanning electron microscopy (SEM)**

The morphologies of $Gd_2O_3$ thin film annealed at different temperature (500,600,700,800)°C investigated in fig.(3), this figure confirms the existence of $Gd_2O_3$ nanoparticles and approximate spherical [21] and grain size of the particles investigated in fig.(3). Finely grain size of the particles was increased with increasing the annealing temperature.

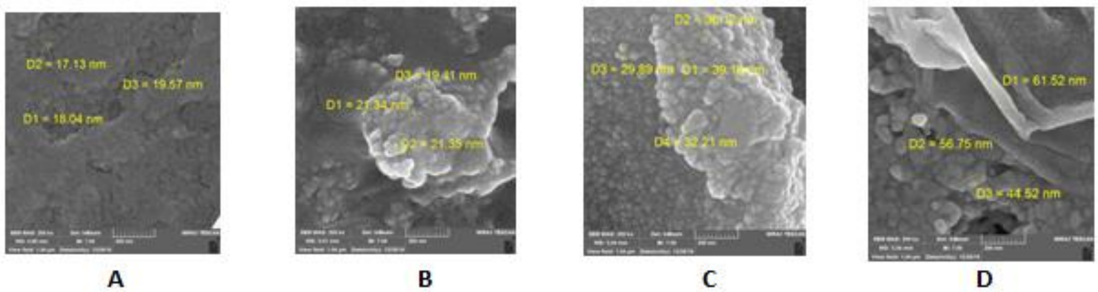


**Fig. 3: SEM micrographs of $Gd_20_3$ thin films deposited on p-Si(100) substrate and annealing for 1h with different annealing temperatures: A (500 °C), B(600 °C) , C(700°C ) , D (800°C )**

**3-3 Magnetic properties**

The vibrating sample magnetometer (VSM) was used to study the $Gd_2O_3$ nanoparticles deposited on p-Si(100) substrate and annealing for 1h at different temperatures (500,600,700 and 800)°C. The hysteresis loops shown in fig.(4) the values of specific saturation magnetization (Ms) increased with increasing annealing temperature as shown in fig.(5). Can be explained the magnetic parameters (specific saturation magnetization (Ms)) as a function of heat treatment may be due to the growth of the grains with increasing annealing temperature. [22]

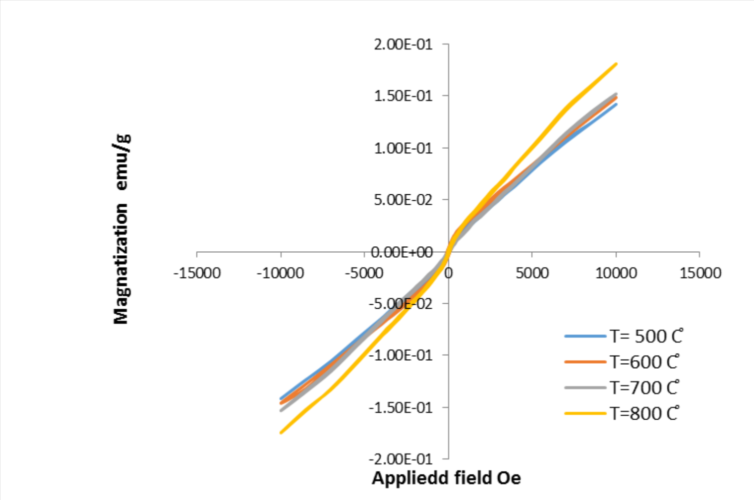


**Fig. 4. Hysteresis loops of $Gd_2O_3$ nanoparticles obtained at different annealing temperatures**

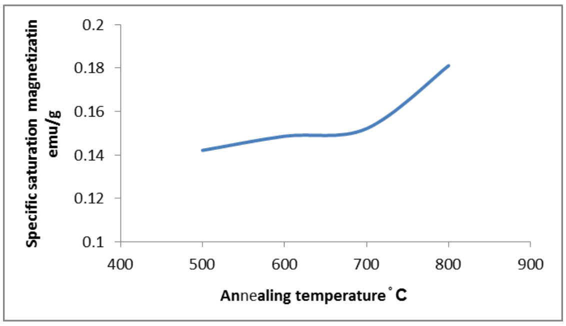


**Fig 5 .The specific saturation magnetization of $Gd_2O_3$ for different annealing temperature**

## Conclusion

The crystalline development was greatly affected by the annealing temperatures.

The x-ray analysis for prepared $Gd_2O_3$ thin films shows that the structures are cube and the crystallite size increases with increasing the annealing temperature.

The scanning electron microscope(SEM) analyses of $Gd_2O_3$ nano-particle shows approximate spherical as well as the specific saturation magnetization (Ms) increased with increasing annealing temperature **.**

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# الخصائص النمائية والتركيبية والمغناطيسية لأغشية أكسيد الجادولينيوم (Gd2O3)

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**الملخص**

تم الحصول على طبقة رقيقة من الأغشية النانوية $Gd_2o_3$ من خلال تقنية الطلاء الدوراني. وتم قياس الخواص التركيبية للغشاء الرقيق بتقنة حيود الأشعة السينية(XRD ) لمعرفة التركيب البلوري وتأثير اختلاف درجات حرارة التلدين المختلفة على النمو البلوري لتلك النماذج، وفحوصات المجهر الإلكتروني الماسح(SEM) وتم دراسة الخواص المغناطيسية بأستخدام مقياس المغنطيسية لعينة اهتزازية (VSM) في درجات حرارة التلدين المختلفة (800،700،600،500) سيليزية. وبينت نتائج حيود الاشعة السينية بأن جميع الاغشية المحضرة ذات تركيب متعدد التبلور ذات قيم حيود واتجاهات مختلفة وكانت اغشية $Gd_2O_3$ من النوع المكعب ولوحظ انه بزيادة درجة حرارة التلدين نحصل على افضل أنماء بلوري وبينت نتائج SEM ان شكل الحبيبات النانوية المرسبة كانت بشكل كروي ،ونلاحظ زيادة مغنطة التشبع (MS) بحوالي (0.1811 − 0.1421) emu / g مع زيادة درجة حرارة التلدين.