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A Study on Microstructural and Electrical Characteristics of Mg (10 Mol%) Doped $CaCO_3$ Ceramics Films

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Abstract

Mg (10 mol %) doped $CaCO_3$ ceramics were prepared by solid-state reaction method in the vacuum chamber at the annealing temperature of 1000°C for 1h. Analar grade of Magnesium Oxide, MgO and Calcium Carbonate, $CaCO_3$ were used with a desired stoichiometric composition to prepare $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramics. Structural properties of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramics were examined by XRD method. Sol-gel based $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si films were fabricated by thermal diffusion technique at 400°C for 1 h in vacuum chamber. The film was characterized by Scanning Electron Microscopy (SEM) and current-voltage (I-V) measurements. Microstructural and electrical characteristics of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si film were studied in this work.

Key words: $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si film, SEM, IV characteristic measurement.

Introduction

Ceramics is an artifact made of hard brittle material produced from nonmetallic minerals by firing at high temperatures. In addition to the well-known physical properties of ceramic materials in hardness, compressive strength, brittleness, there is the property of electric resistivity. Categories of advanced ceramic materials are employed in a wide variety of electric, optical, and magnetic applications. Advance ceramics also have important uses in electronics, aerospace, construction, and nuclear industries**.**

Carbonates are among the most widely distributed minerals in the Earth's crust. The crystal structure of many carbonate minerals reflects the trigonal symmetry of the carbonate ion, which is composed of a carbon atom centrally located in an equilateral triangle of oxygen atoms. This anion group usually occurs in combination with calcium, sodium, uranium, iron, aluminum, manganese, barium, zinc, copper, lead, or the rare-earth elements. This work deals with the growth mechanism and characterization of Mg10 (mol%) doped $CaCO_3$ film. Structural properties of the Mg(10 mol%) doped $CaCO_3$ sample were investigated by XRD measurement. Morphological features and I-V characteristics of the film were also investigated in this work.

Experimental Procedures

The raw materials of $CaCO_3$ and MgO were used as starting materials. The amount of high purity $CaCO_3$ (90 mol%) and MgO (10 mol%) powders were mixed in desired stoichiometric composition to prepare $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic. The mixture powder was ground by agate motor for 2 h to be homogeneous and to obtain uniform grain size.

$Ca_{0.90}Mg_{0.10}(CO_3)_2$ powders were then mixed with 2-methoxyethanol solution and HCl then heated up to 100°C for 2 h to prepare precursor solution. The solution was stirred by glass stick throughout the preparation of precursor solution to get the homogeneous and transparent solution (sol-gel). Then, the homogeneous precursor solution was obtained when the mixture was cooled down to room temperature. The n-Si (100) wafer was used as the substrate to perform p-n junction ceramic film. Prior to the deposition, the substrate was cleaned according to standard wafer cleaning procedure. n-Si (100) wafer dimension of (0.5

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cm x 0.5 cm) was used as the substrate. The substrate was cleaned through standard wafer cleaning process. n-type silicon substrate was etched in HF:H_2O (1:5) for 10 min, and immersed in deionized (DI) water for 10 minutes. And then, the substrate was immersed in acetone and methyl alcohol for 10 min and was rinsed in DI water for 10 min. Next, the substrate was dried at room temperature.

After the cleaning process, the precursor solution was coated onto substrate by spin-coating technique. Later, coated-layer was first dried at room temperature. The $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si material was deposited at 400^oC for 1 h by using CAHO SR-T903 Temperature Controller in vacuum chamber (160mmHg). The K-type thermocouple was used as the temperature sensor to read-out the real process temperature of the sample and substrate. After the deposition, the obtained $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si film was etched in the mixture of ethanol, DI-water and HCL (1:1:1) for 15 min to get the homogeneously surface of the dopant ceramic on n-Si substrate. The etched $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si film was dried at room temperature. Finally, $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si ceramic film were obtained.

XRD Measurement

A structural analysis of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic was performed by RIGAKU, MULTIFLEX X-ray diffractometer using Ni-filter with CuK_α radiation, λ = 1.54056 Å. This measurement was done at Universities' Research Centre (URC), Yangon University. The main reflections in the range $10^o < 2\theta < 70^o$were observed, and the collected data were used to refine the unit cell parameters of the crystal from the observed 2θvalues with JCPDS (Joint Committee on Powder Diffraction Standards). Lattice parameters of those crystals have been investigated.

Scanning Electron Microscopic (SEM) Measurement

In the present work, surface morphology and diffusion layer thickness of $Ca_{0.90}Mg_{0.10}(CO_3)$/n-Si thin film was investigated by JEOL JSM-5610LV Scanning Electron Microscope(SEM) with the accelerating voltage of 15 kV and the beam current of 50mA.

I-V Characteristic Measurement

The light sensitive effect of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film has been investigated in the dark condition (0 lux) and four different illumination conditions using (60 W) electric bulb light source. The light intensity sensor of Si-photodiode (LIGHTMETER 2330LX, SEW) was placed near the sample with parallel position to record constant intensity of light source throughout the measurement. Current-voltage (I-V) characteristics of the film in the dark and in illumination conditions were also investigated between the bias voltage of -5V and +5V with the step voltage of 0.2 V.

Results and Discussion

XRD Study

The powder X-ray diffraction pattern of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ is shown in Fig1. Some of the recorded XRD data of diffraction angle (2θ), atomic spacing (d), intensity (I) and miller indices (hkl) are tabulated in Table (1). As shown in Figure (1), the observed XRD pattern and the obtained XRD data of the crystals are found to agree with the standard data of JCPDS (Joint Committee on Powder Diffraction Standards).

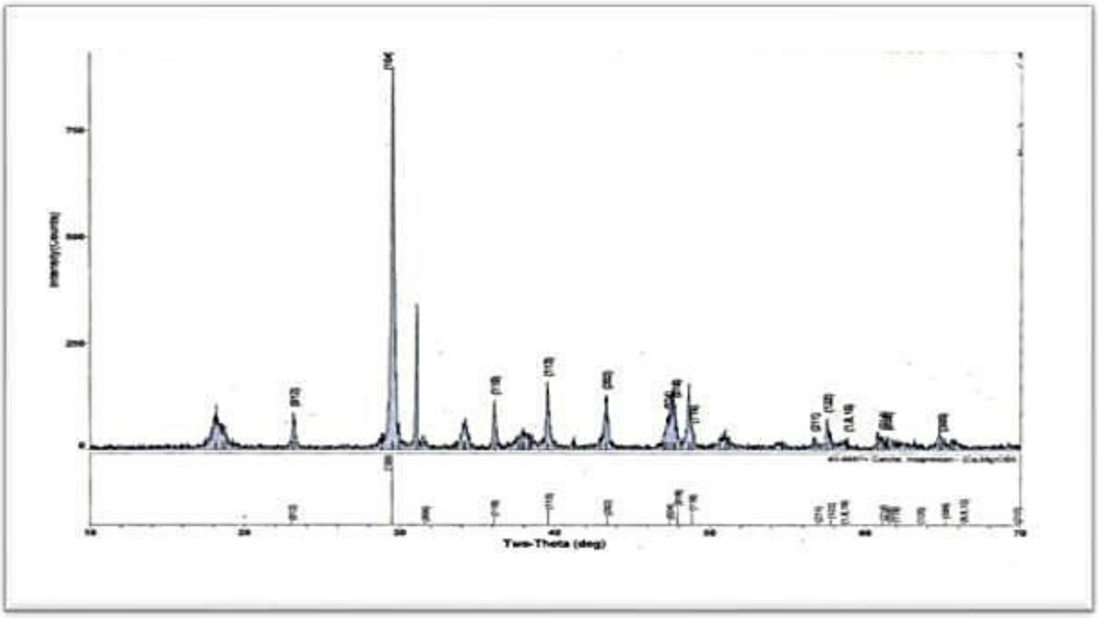


Figure (1)XRD pattern of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic

Table (1) XRD data of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic

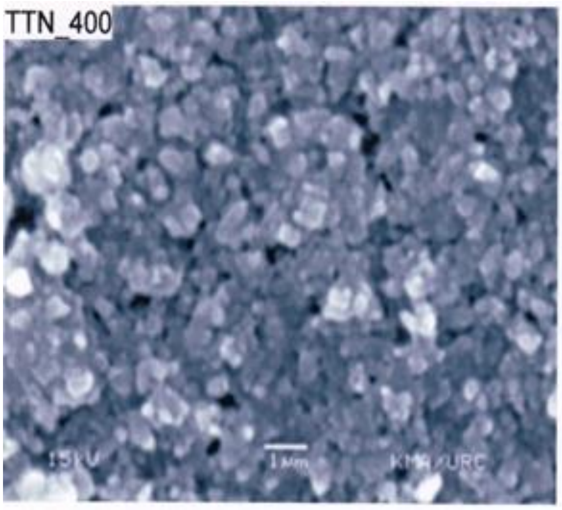
Line No	2θ (°)	(*hkl*)	*d* (Å)	I (%)	FWHM (°)
1	18.139	(101)	4.8865	8.20	0.501
2	18.559	(121)	4.7770	5.40	0.479
3	18.762	(012)	4.7256	4.90	0.638
4	23.158	(012)	3.8377	9.20	0.194
5	28.820	(104)	3.0953	3.70	0.194
6	29.520	(104)	3.0234	100.00	0.236

The diffraction line at 29.52° of (104) plane is found to be the strongest one (I = 100%) and some of the diffraction lines (extra peaks) are not assigned with the comparison of standard data library due to the dopant effects. According to powder XRD pattern, $Ca_{0.90}Mg_{0.10}(CO_3)_2$ belongs to hexagonal structure at room temperature and the lattice parameters are evaluated by

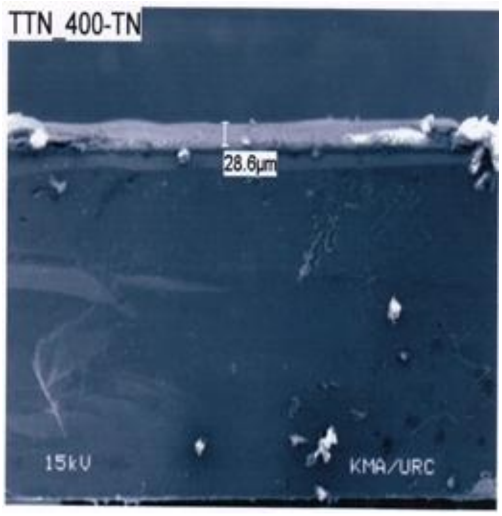
the equation of $\frac{1}{d^2}=\frac{4}{3}\left[\frac{h^2+hk+k^2}{a^2}\right]+\frac{l^2}{c^2}$ and the lattice parameters are obtained as $a = b =$ 25.3906Å, and c = 13.8508 Å respectively. The observed XRD data is calculated using the Scherrer formula, $t = \frac{0.9\lambda}{B\cos\theta}$, where "t" is the crystallites size (nm), "λ" is the wavelength of incident X-ray (nm), "θ" is the diffraction angle of the peak under consideration at FWHM (°) and "B" is the observed FWHM (radians). In this work, we used the diffraction line 2θ is 29.52°, Miller index (hkl) is (104) and FWHM is 0.202° to calculate the crystallite size of the sample because this line is the strongest in intensity among the observed XRD patterns. The crystallite size of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic is obtained as 34.81 nm. It is consistent with the crystallite size of prepared samples.

Microstructural Analysis

Figure (2) shows the SEM image of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film. As shown in the figures, the grain shape of the sample is circular and rectangular random-fashion and the grain sizes are 0.20 µm – 2.20 µm. It was observed that there was a grain growth in diffusion layer crack-free**.** Also**,** the diffusion layer thickness of the sample is shown in Fig 3. It is found that (1) the layer boundary of the sample and substrate are mostly homogeneous and (2) the diffusion layer thickness of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin films is 28.60 µm. It is indicated that the sample of $Ca_{0.90}Mg_{0.10}(CO_3)_2$ is successfully deposited on n-Si substrate by using thermal diffusion technique.



Figure(2) SEM micrograph (surface morphology) $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film.



Figure(3) SEM micrograph (diffusion of the layer thickness) of the(top view of the film) $Ca_{0.90}Mg_{0.10}(CO_3)_2$ /n-Si thin film (side view of the film)

I-V Characteristics of the Film

I-V characteristic curves of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under dark (0 lux), 390 lux, 1000 lux, 1500 lux and 2000 lux using (60W) electric bulb light source are shown in Figures (4, a - e). As shown in observed I-V characteristic curves, the output current is exponentially increased with increasing bias voltages and these curves are rectifications of diode action of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film device.

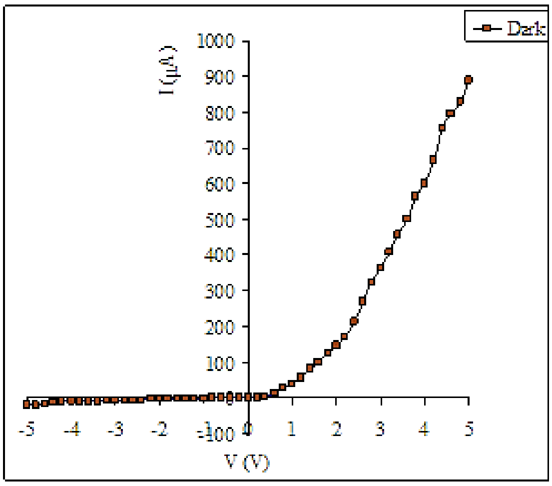


Figure (4-a).I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film in dark condition

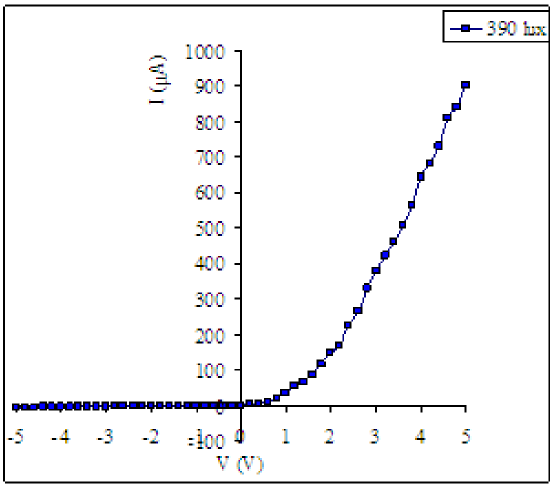


Figure (4-b). I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under 390 lux (ambient) illumination condition

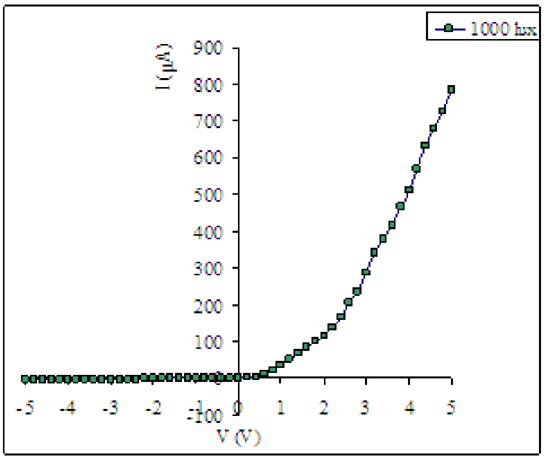


Figure (4-c).I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under 1000 lux (ambient) illumination condition

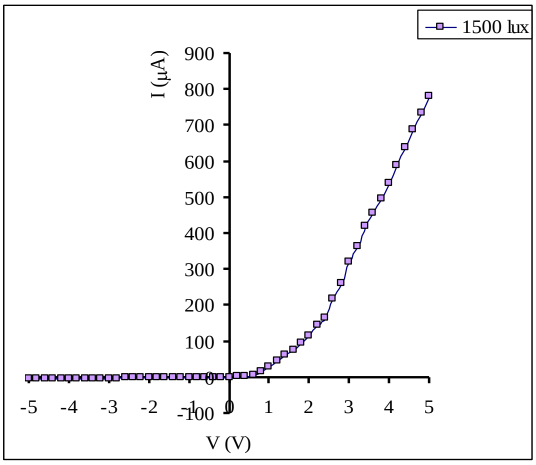


Figure (4-d).I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under 1500 lux (ambient) illumination condition

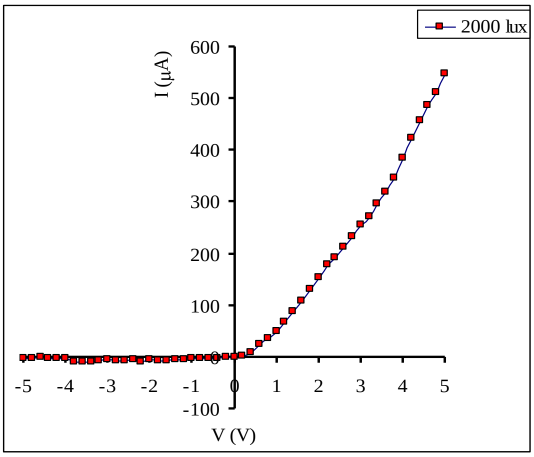


Figure (4-e).I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under 2000 lux (ambient) illumination condition

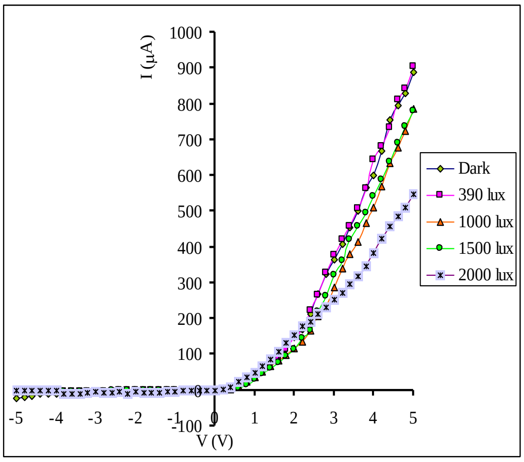


Figure (4-f).Comparison of the I-V characteristic curves of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film in dark and different illumination conditions

Furthermore, a comparison of the output currents as a function of (-5 V - +5 V) bias voltages in different illumination conditions is shown in Figure (4-f). Maximum output currents of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film at +5 V in different illumination conditions are listed in table 2. The maximum output current of the film is obtained as 905 µA in 390 lux (ambient) condition.

Table (2) The maximum output currents of $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film under different illuminations

Sr No	**Illumination (lux)**	**I_{max} (µA)**
1	0 (dark)	890
2	390 (ambient)	905
3	1000	785
4	1500	781
5	2000	549

Conclusion

Mg (10 mol%) doped Calcium Carbonate, $CaCO_3$ ceramic was prepared at 1000°C for 1 hour by using solid state reaction method in vacuum chamber (160 mmHg). The as-prepared $Ca_{0.90}Mg_{0.10}(CO_3)_2$ ceramic was characterized by XRD. In addition, $Ca_{0.90}Mg_{0.10}(CO_3)_2$ was deposited on n-Si substrate using thermal diffusion method at 400°C for 1 h in vacuum chamber. $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film was characterized by SEM and I-V measurements. XRD technique is employed to examine crystal structure and phase assignment of $Ca_{0.90}$ $Mg_{0.10}(CO_3)_2$ ceramic. In this study, most of the diffracted peaks are well-matched with the standard JCPDS. The strongest peak is found to be (104) reflection, which indicates the dominated plane of the polycrystalline phase. From this study, $Ca_{0.90}Mg_{0.10}(CO_3)_2$ belongs to hexagonal structure and the lattice parameters are obtained as a = b = 25.3906 Å, and c = 13.8508 Å respectively. The crystallite size of the sample is obtained as 34.81 nm. It is consistent with the crystallite size of prepared sample. From SEM micrograph of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$/n-Si thin film, the grain shape of the sample is found to be a non-uniform circular shape with the sizes of 0.20 µm – 2.20 µm. The diffusion layer of the $Ca_{0.90}Mg_{0.10}(CO_3)_2$ on n-Si substrate is found to be homogeneous and the thickness is obtained as 28.60µm. From the I-V characteristic curves of the $Ca_{0.90}$ $Mg_{0.10}(CO_3)_2$/n-Si thin film, the film exhibited as a junction diode. According to the experimental results, the sample can be considered as the p-n junction diode and light dependent resistor (LDR).

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