

A COMPARATIVE STUDY OF PROPERTY MEASUREMENT FOR ZnO-THIN FILM GROWTH PROCESSES USING HYDROCHLORIC ACID (HCl) AND WATER (H₂O) SOLUTION-DEPENDENT ON NOVEL ELECTROSTATIC SPRAY DEPOSITION (ESD)

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This investigation has been carried out utilising the innovative ESD technique for six ZnO nano polycrystalline (NP) samples. The Debye-Scherrer model (DSD) is used to study the mechanical elastic properties (MEP) as lattice dislocation density (δ) and the lattice strain (ϵ %) contribution to the X-ray diffraction line broadening in nanocrystalline ZnO thin film. The average size of the crystallites was determined to be approximately 48 nm. When studying the phenomenon of line broadening, it is critical to take into account MEP anisotropy. This is necessary in order to effectively distinguish between the influences of strain and particle size. The novel ESD technique promotes ZnO growth mechanisms by adding small amounts of HCl into spray solutions on conductive In₂O₃:Sn (ITiO)-coated alkali-free glass substrates while changing the water (H₂O) ratio. XRD pattern characterisation revealed the preferred orientation of

the (002) planes in ZnO thin films. This study represents a significant advancement in the easy understanding of the ZnO growth mechanism and the changes in MEP enabled by ESD-deposited thin film in cost-effective industrial and commercial applications of electronics.

Keywords: ESD, Debye–Scherrer method, lattice dislocation density, lattice strain, ZnO growth mechanism.

1. INTRODUCTION

Zinc oxide (ZnO) nanostructure semiconductors have attracted considerable attention in research due to their potential applications in diverse fields such as solar cells [1], light-emitting diodes [2], photocatalysis [3], and so on. However, novel semiconductor materials frequently use strain as a design parameter to increase electron mobility [4]. This phenomenon is now used in a number of applications, including strained metal-oxide semiconductor field-effect transistors (MOSFETs) [5] and piezoelectric resistors [6]. The incorporation of lattice defects intricately links the functionality of semiconductor materials and devices [7]. Their appearance and ability to move around make things less effective and more likely to break after a while of use [8]. Commonly used techniques for creating ZnO thin films include dry methods such as sputtering [9] and pulsed laser deposition [10], as well as wet methods such as chemical bath deposition [11], mist chemical vapor deposition [12], and spray pyrolysis [13].

Additionally, the ESD method is not only easy to use, but it also has important industrial benefits, such as the ability to cover large areas because it is simple and straightforward to understand. This technique allows for non-vacuum deposition, has a low cost, and provides convenient control over the composition ratio and dop-

ing [14]. These advantages should lead to the development of inexpensive and simple electronic devices that can transmit visible light without obstruction using the ESD technique. Recently, there has been a dearth of research projects and papers that specifically examine the growth mechanism of oxide-based semiconductors using the ESD approach [14] and concentrate on the chosen solution. Characterisation using the ESD approach is essential for comparing the results with other techniques.

This study aims at investigating the effects of sequential additions of 0.005M and 0.015M HCl, using the ESD method, on the growth process of ZnO, taking into account any changes in mechanical behaviour. The ESD method is used on six different ITiO glass substrates at three different temperatures: 300 °C, 400 °C, and 500 °C, by varying the H₂O ratio in the precursor solutions. In this case, ZnCl₂ is used as a starting material and dissolved in CH₃CH₂OH at a concentration of 0.1 M. The experimental sections mention the detailed preparation method for spray solutions. Furthermore, nanoscale-level studies are the focus for how behaviour is changing and affected by ESD with ZnO growth processes. This study represents the initial phase in the development of affordable and uncomplicated devices that are transparent to visible light.

Table 1. Typical Deposition Parameters and Conditions for ESD

S. no.	Parameter	Value
1.	Distance between the nozzle aperture and the hot plate	3.5 cm
2.	Flow rate	2.0 ml/h
3.	Deposition time	5 min
4.	Applied voltage	8 kV

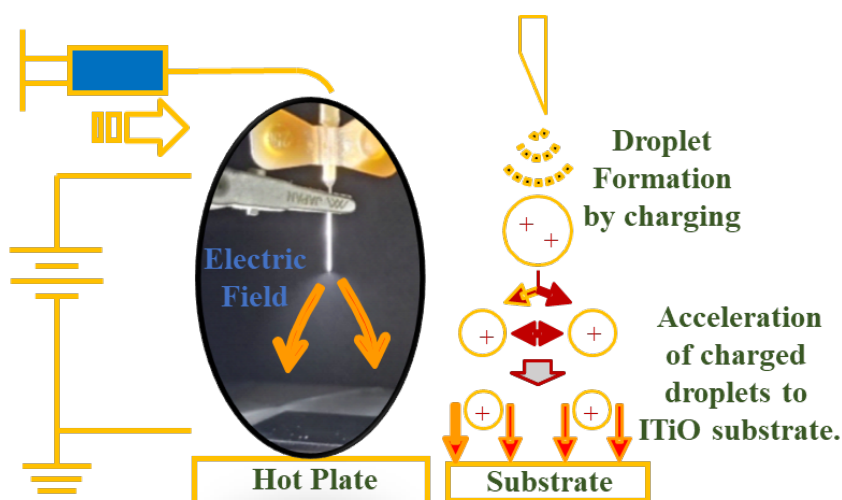


Fig. 1. F. I. Abbas, *Latvian Journal of Physics and Technical Sciences*: Schematic diagram of ESD.

2. EXPERIMENTAL METHODS

The zinc sources used in this study were ZnCl_2 (98% Assay, $\text{ZnCl}_2=136.32$), Lot. LEH7398, Mfg. Date. 2021.09, FUJIFILM Wako Pure Chemical Corporation). The solutes were ethanol ($\text{CH}_3\text{CH}_2\text{OH}$ (99.5), Cat. No. 14033-70, FW: 46.07, KANTO CHEMICAL CO, INC), and deionized water (H_2O). ZnO films were deposited using ESD on a conductive $\text{In}_2\text{O}_3:\text{Sn}$ (ITiO) coated alkali-free glass substrate. The experimental diagram of ESD is shown in Fig. 1. 20 ml of six (6) different spray solutions were prepared with changing the H_2O

ration, and HCl concentrations for the ESD spray technique.

To prepare the ESD solution with 0% of H_2O and a concentration of 0.1 M, separately, 20 ml of $\text{CH}_3\text{CH}_2\text{OH}$ was measured and placed in two glass beakers. The density of $\text{CH}_3\text{CH}_2\text{OH}$ was 0.78945 g/cm^3 . Then 0.2726 g ZnCl_2 was added with the $\text{CH}_3\text{CH}_2\text{OH}$ in the glass beaker. Then we introduced distinct solutions of HCl at concentrations of 0.005 M and 0.015 M into the ESD solutions, respectively. To make the solution homogeneous, it was heated up

to 20 minutes in the magnetic stirrer with hot plate (ADVANTEC SR/350) (Fig-1 (c)). The temperature was considered to be 150 °C for the magnetic stirrer with hot plate. Similarly, two separate glass beakers were used to prepare the second type ESD spray solutions, which consisted of 80 % CH₃CH₂OH (16 ml) and 20 % H₂O (4 ml), respectively. Next, the same amount of ZnCl₂ was added to the separate glass beakers along with 16 ml of CH₃CH₂OH. The same procedures were applied for mixing the 0.005 M and 0.015 M HCl concentrations, and the homogeneous spray solution was prepared. For the third type of ESD spray solution, 50 % CH₃CH₂OH (10 ml of CH₃CH₂OH) and, 50 % H₂O (10 ml of the same procedures were applied for the mixing of the 0.005 M and 0.015 M HCl concentrations, and the homogeneous spray solution was prepared, respectively.

ESD spray was performed for 5 minutes at a solution flow rate of 2.0 ml/h on the substrate surface, and voltage was applied between nozzle and substrate of 8 kV during spraying. Followed by ESD temperature ranged from 300 °C, 400 °C, and 500 °C

in air for 5 minutes. For the simplification and understanding the ESD technique, the parameters are listed in Table.1 The crystallographic orientations of the ZnO films were evaluated using X-ray diffraction (XRD; Rigaku Ultima IV, Rigaku Smart-Lab). MEP of ZnO thin films was investigated using DSD, which was correlated to the basic crystal growth mechanism [15]–[22], by the novel ESD method. The equations involved in this estimation are given below (1)–(3) [15], [20].

$$D = \frac{0.9 \lambda}{\beta \cos \theta} X, \quad (1)$$

$$\delta = \frac{I}{D^2}, \quad (2)$$

$$\varepsilon = \frac{\beta_G \cos \theta}{4 \sin \theta}, \quad (3)$$

where D, λ , θ , β are crystallite, lambda as the wavelength (1.54 Å), theta as angle (diffraction angle), beta as angle (in radian), respectively.

3. RESULTS AND DISCUSSION

3.1. The XRD Analysis

The XRD experiment was conducted to analyse the structural properties of the ZnO thin film growth mechanism utilising the ESD technique at temperatures of 300 °C, 400 °C, and 500 °C, respectively. Six distinct spray solutions were prepared by dissolving ZnCl₂ in CH₃CH₂OH at a concentration of 0.1 M. These solutions were used to investigate the impact of 0.005M and 0.015M HCl on the growth mechanism of ZnO thin films using the ESD method. The H₂O ratio was

varied during the experiments. The XRD pattern of the ZnO thin films on an In₂O₃:Sn (ITiO)-coated alkali-free glass substrate is shown in Fig. 2 (a–c), and 2(d–f), for the 0.005M and 0.015M HCl, with varying H₂O ratios of 0 %, 20 %, and 50 %, respectively. Figures 2(a) and 2(d) are for 0.005M HCl and 0.015M HCl mixing without the H₂O in the spray solution, respectively. Figure 2 (a) exhibits strong peaks at the (002), and (102) planes. This peak observation

clearly confirms the ZnO thin film growth, without the H₂O mixing into the solution at a concentration of 0.005M HCl. Figure 2(d) also observed a pattern at (002) plane for the ESD spray solution with a 0.015M HCl concentration. Figure 2(d) shows that the peak strength at 500 °C is stronger in the (002) plane compared to the 0.005M HCl solution.

Figures 2 (b) and 2(e) showed the results for 0.005M and 0.015M HCl in the spray solution with a 20 % H₂O ratio, respectively. The peak intensities reveal the appearance of the (002), and (102) planes for the 0.005M HCl added ESD solution at 500 °C ESD performing temperature. However, for the 0.015M HCl solution peak is only seen at (100) plane, which is very weak at 300 °C, 400 °C, and 500 °C, respectively. In addition, the peak at (002) plane is only for 400 °C ESD temperature. In Fig. 2(b), the 0.005 M HCl solution has a stronger peak intensity in the (002) plane at 500 °C than the 0.005 M HCl solution shown in Fig. 2(a). Surprisingly, in Fig. 2(e) for the high-concentration HCl solution of 0.015 M, the peak for the (002) plane at 500 °C has not appeared. This may be due to the complex formation effect in the spray solution caused by H₂O and the high concentration of acid at that temperature [23]. The results for 0.005M and 0.015M HCl in the spray solution with a 50 % H₂O ratio are shown in Figs. 2(c) and 2(f) in sequential order. For 0.005M HCl case, the peak intensities indicate the presence of the (100), (002), (101), and (102) planes where the strong peak appears at (002) plane for 500 °C, ESD performing temperature and weak peak at (102) plane. On the other hand, (100), (102) plane for 400 °C is slightly stronger compared to 20% H₂O ratio ESD sample at 400 °C. For 300 °C, very weak peak intensities were found at (100) and (102) planes. Compared to Figs. 2(a) and 2(b), Figure 2(c) shows a

higher peak intensity in the (002) plane at 500 °C for the 0.005 M HCl solution. This behaviour occurs gradually as the H₂O ratio increases in the spray solution with a low-concentration 0.005M HCl. The peak intensity in the (100) plane for the 0.015M HCl solution for both the 50 % and 20 % H₂O ratios has shown very small change. These behaviours can be understood to analyse the results with basic crystal theory [20], [21]. In the following section, it will be discussed elaborately.

Figure 2 (a–c) displays a sequence of successive and gradually increasing peak intensities, which correlate to the formation of ZnO thin films. These peaks were observed at a diffraction angle of around 34.80° for samples containing 0.005 M of HCl, using the ESD method at a temperature of 500 °C. Peaks at the (100) and (102) planes showed low intensities in the range of 300 °C–400 °C, but the appearance of the (002) plane in the observation confirmed the fabrication of ZnO thin films with a hexagonal wurtzite structure [15]. However, Fig. 2 (d–f) for the 0.015M HCl mixing results clearly shows the peaks for the (002) plane. The 20 % H₂O ratio samples exhibit sudden drops in peak intensity. The peaks in the (002) plane are once again prominent for the 0.015 M HCl mixing samples with a 50 % H₂O ratio. Also, the peak intensity for the (100) plane is a little higher for the samples mixed with 0.015 M HCl than for the samples mixed with 0.005 M HCl from the ESD at the temperature ranges that were studied. However, the (002) plane consistently shows rising peak intensity results for the 0.005M of HCl mixing samples, indicating the gradual growth of the ZnO thin film. The XRD results in Fig. 2(a–c) showed that all of the thin films that were deposited had a clear orientation along the c-axis (002) plane and a slightly stronger peak along the (102) plane corresponding

to the ZnO thin film growth. Increasing the temperatures as well as the film thickness of ESD-produced films results in a sharper and more intense (002) plane of diffraction peak [14], accompanied by a decrease in the full width at half maximum (FWHM). Accord-

ing to the fundamental theory, the growth of ZnO thin films is determined by the c-axis orientation [20], [21]. This theory asserts that the (002) plane of ZnO possesses the lowest surface energy, which plays a crucial role in determining the crystal structure.

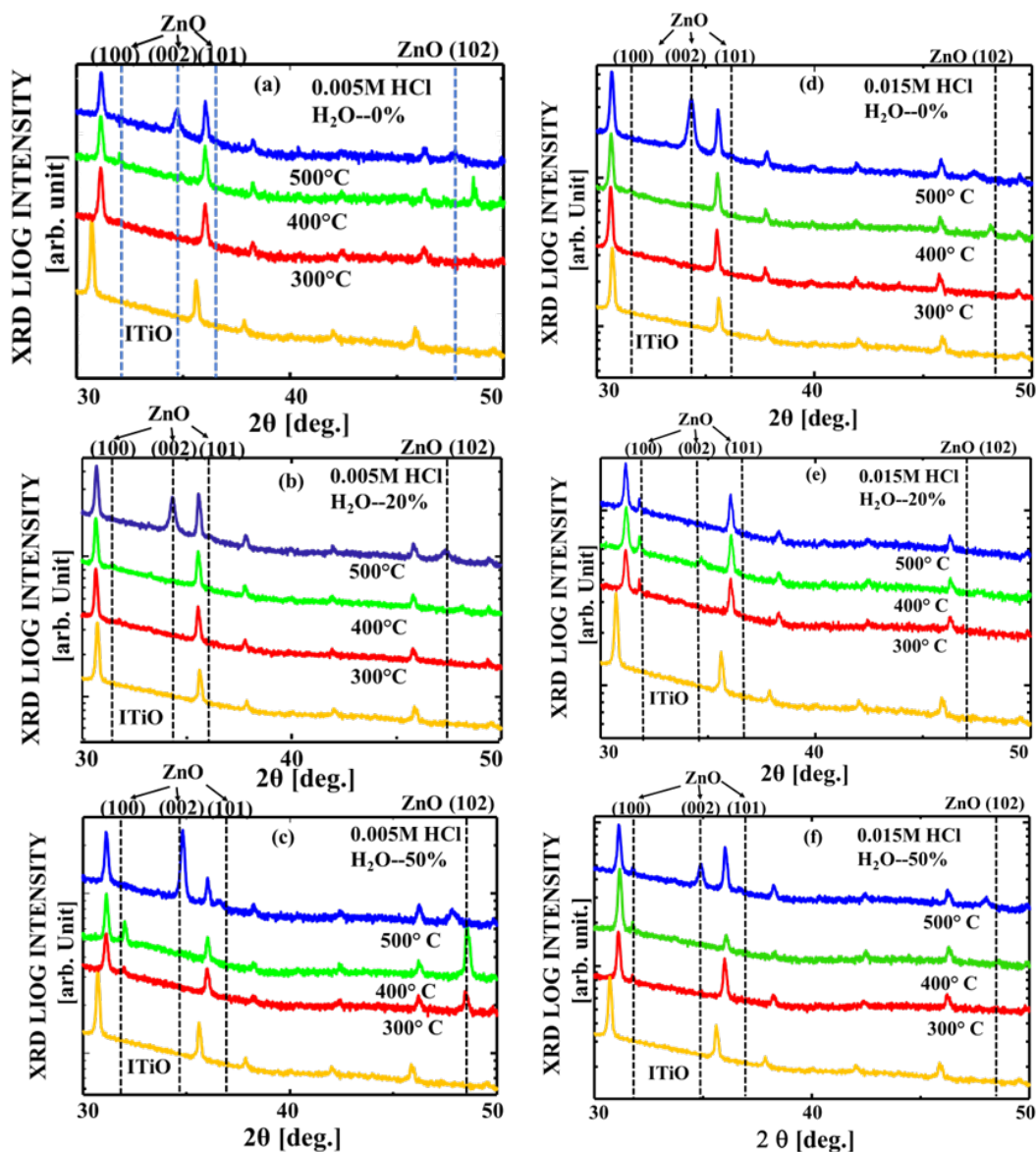


Fig. 2. F. I. Abbas, *Latvian Journal of Physics and Technical Sciences*: XRD patterns of ZnO film according to (a–c) 0.005M HCl, and (d–f) 0.015M HCl weight ratio of H_2O /solvent in the solution deposited by ESD at 300 °C, 400 °C and 500 °C, respectively.

3.2. ESD TEMPERATURE EFFECT ON MEP

A key factor in determining the MEP of thin film materials is δ ($\times 10^{18} \text{ m}^2$), which is the principal method of plastic deformation in these materials. The investigation of the correlation between dislocation behaviour and mechanical reaction has been an important focus of extensive research ever since Taylor initially proposed the concept of dislocation in 1934 [24]. Dislocations play a critical role in determining the mechanical characteristics of materials by influencing the microstructure development. Figures 3 (a–c) and 3 (d–f) show a more detailed look at the ZnO nanoparticle growth mechanism from the mechanical point of view, changing the concentration of the ESD process.

Figures 3(a, b) and 3(d, e) show the results of δ ($\times 10^{18} \text{ m}^2$) for 0.005 M HCl and 0.015 M HCl of the ESD spray solution for 0 % and 20 % H_2O ratio, respectively. These figures show a constantly decreasing trend for δ ($\times 10^{18} \text{ m}^2$) over the entire temperature range of 300 °C to 500 °C. On the other hand, Figs. 3(c) and 3(f) represent the result for δ ($\times 10^{18} \text{ m}^2$) for a 50 % H_2O ratio in the ESD spray technique. In Fig. 3(c), the δ ($\times 10^{18} \text{ m}^2$) increases from 300 °C to 400 °C, and then it decreases from 400 °C to 500 °C. Comparing, in Fig. 3(f), the δ ($\times 10^{18} \text{ m}^2$) decreases from 300 °C to 400 °C, and then it starts increasing from 400 °C to 500 °C.

From the physical point of view, in Fig. 3. (a, b), 0 % and 20 % H_2O ratio mixed sample shows a consecutive and gradually decreasing nature of δ ($\times 10^{18} \text{ m}^2$) for the 0.005 M HCl of the ESD spray sample. This indicates the stabilisation mode of the thin film growth during the ESD process in the micro-level state. Similar behaviour is also observed for the 0 % and 20 % H_2O ratio ESD sample for 0.015 M HCl. However, for the 50 % H_2O ratio (Fig. 3. (c)), rapid

increasing and then then decreasing nature were found, which indicated the non-isotropic behaviour of the 0.005 M HCl. Perhaps, this non-isotropic nature is related to the complex formation of the small acid and large H_2O ratio of the ESD sample.

Importantly, the 50 % H_2O ratio with the 0.015 M HCl showed decreasing results from 300 °C to 400 °C and then increasing results found from 400 °C to 500 °C, which was similar to the isotropic in nature. Comparing the results with a 50 % H_2O ratio to 0.005 M HCl, this indicates that low concentrations of HCl mixed ESD spray samples are showing non-isotropic behaviour and high concentrations of HCl samples are showing isotropic behaviour. This dynamic behaviour is very essential for the growth of ZnO thin film on the ITIO substrate surface.

The study of crystal lattice strain, ϵ (%) is of utmost significance in the field of semiconductor materials and devices. When designing new semiconductor materials, strain is often used as a design feature to make it easier for electrons to move around [24]. Bardeen and Shockley initially emphasised the significant influence of mechanical phenomena in the field of semiconductor physics [25]. Figure 4 (a–c) presents a detailed analysis of the efficiency ϵ (%), of the ZnO nanoparticles in the ZnO thin film investigated in this study. The analysis focuses on the 0.005M concentration with different H_2O ratios of 0 %, 20 %, and 50 % in the ESD. Figure 4(a, b) consistently showed an increasing trend for ϵ (%), across the entire temperature range from 300 °C to 500 °C. Besides, Fig. 4(c) represents the result of ϵ (%), for a 50 % H_2O ratio in the ESD spray technique. Here, in the 300 °C to 400 °C temperature range, ϵ (%), starts to decrease, then from 400 °C to 500 °C it increases. Remarkably, the behaviour

of this sample is highly dynamic despite a small quantity of HCl being mixed. The dynamic nature of this process facilitates the advancement of ZnO thin film technology through the innovative ESD approach.

Furthermore, Fig. 4 (d–f) demonstrates the responses of ESD spray solution samples with varying H₂O ratios when mixed with 0.015 M HCl. In Fig. 4 (d–f), very small change in the ε (%), behaviour was found. This behaviour may be regarded as stiff features for the highly concentrated acid solution [26]. It is possible that the inflex-

ible strategy for distorting the crystal lattice suggests that the large amount of HCl in the sample does not facilitate ZnO formation through the ESD mechanism. The limited quantity of HCl may be deemed appropriate for facilitating the growth mechanism of zinc oxide thin film by altering the H₂O ratio in the ESD technology. Further research is required to understand the mechanism of thin film growth and its fundamentally changing nature, using the convenient and innovative ESD technique for the development of semiconductor studies.

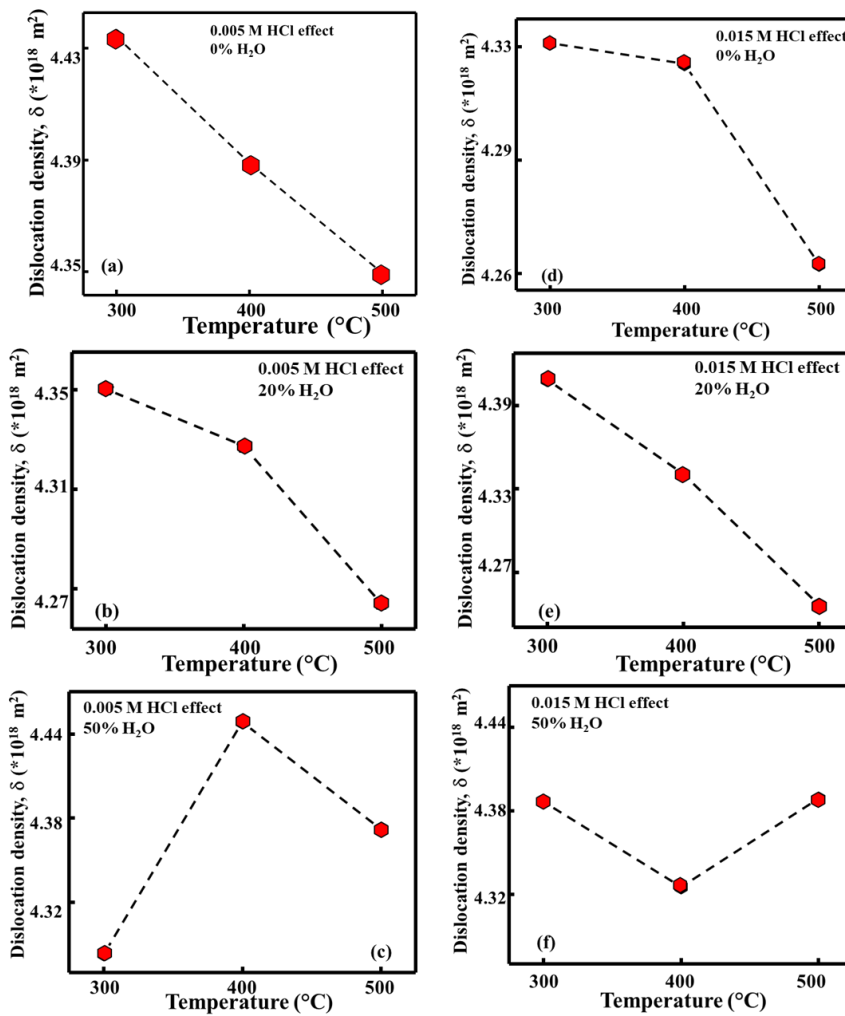


Fig. 3. F. I. Abbas, *Latvian Journal of Physics and Technical Sciences*: Dislocation density, δ ($\times 10^{18} \text{ m}^{-2}$) of ZnO film according to (a–c) 0.005M HCl, and (d–f) 0.015M HCl weight ratio of H₂O/solvent in the solution deposited by ESD at 300 $^{\circ}\text{C}$, 400 $^{\circ}\text{C}$ and 500 $^{\circ}\text{C}$, respectively.

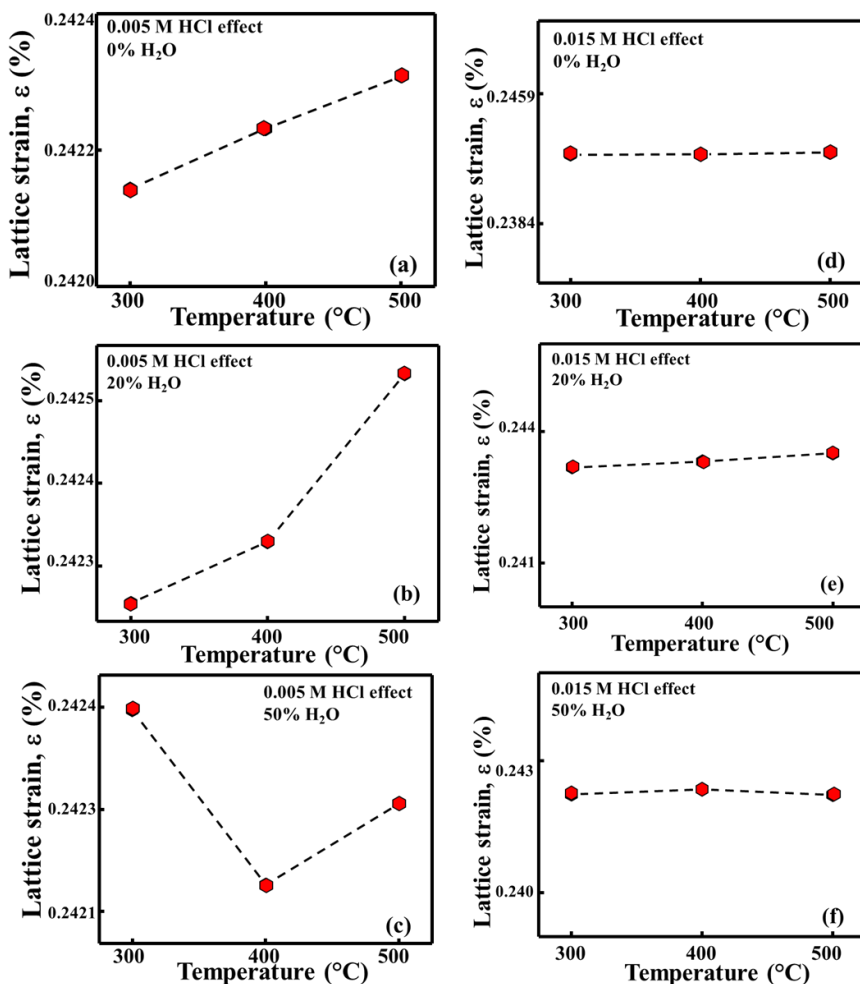


Fig. 4. F. I. Abbas, *Latvian Journal of Physics and Technical Sciences*:
Lattice strain, ϵ (%) of ZnO film according to (a – c) 0.005M HCl,
and (d – f) 0.015M HCl weight ratio of H₂O/solvent in the solution deposited
by ESD at 300 °C, 400 °C and 500 °C, respectively.

Finally, this study investigated the HCl acid mixed solution-dependent growth mechanism of ZnO semiconductor thin film by varying the H₂O ratio solution through the novel ESD method on a conductive In₂O₃:Sn (ITiO)-coated alkali-free glass substrate from the temperature range of 300 °C, 400 °C, 500 °C. Compared to other dry processes [9], [10], and wet processes [11]–[13], crystal quality is not very high, and XRD analysis shows that adding a little HCl (0.005 M)

makes the growth process enhancement of ZnO thin films made with the novel ESD technology quantitatively good. In the area of a defect (dislocation) is elastically distorted (strained). Elastic fields cause the diffraction peaks to broaden. This phenomenon is sometimes referred to as microstrain and is contingent upon the presence of non-uniform lattice distortions. Here, it is also found that there is a possible correlation with the growth mechanism and the MEP by ESD.

Still, the results obtained from this study of ZnO thin film growth mechanism were quantitatively good in agreement to consider the cost effectiveness. This method can also be used for other purposes of semiconductor thin films growth mechanism study. Further

investigation on other microstructural properties is also needed for clear visualisation on ZnO growth mechanism by the novel ESD technique. For our upcoming study, we will further explore the structural characteristics of this research.

4. CONCLUSIONS

This study found that ESD can develop the oxide-based semiconductor thin film growth technique cost-effectively. The possible correlation between the growth mechanism of semiconductor thin films and their MEP can be easily realised. Importantly, future studies on thin-film semiconductor growth mechanisms may apply using the ESD method.

Altogether, these findings indicate that employing ESD for depositing the ZnO film is advantageous for creating stacked thin film devices. This study is important as it is the primary stage in developing cost-effective and uncomplicated devices that are transparent to visible light using the ESD method for semiconductor oxide-based materials.

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