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MECHANICAL PROPERTIES OF BORON-DOPED-ZINC OXIDE THIN FILMS USING SPRAY PYROLYSIS TECHNIQUE

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Keywords

Mechanical, Properties, Boron, Doped, Zinc, Oxide, Pyrolysis.

Abstract

This study aims to establish the mechanical properties of boron-doped Zinc Oxide (ZnO) thin film. Mechanical properties are important for engineering applications and are a focus of engineering materials. Using the spray pyrolysis process, a thin coating of boron-doped (ZnO) is created on a soda lime glass substrate at a variable percentage. An iron-constantan thermocouple was used to monitor the substrate's temperature while the film was being deposited at different temperatures. Metallurgical microscopy was used to determine the mechanical characteristics of the doped and undoped samples, including hardness, impact, and tensile strength. The results indicated that when zinc oxide is doped, variations in impact and hardness values are detected, and maximum stress increases with dopant concentration. However, when zinc oxide is undoped, stress and hardness increase with temperature.

SPREY PİROLİZ TEKNİĞİYLE BOR KATKILI ÇİNKO OKSİT İNCE FİLMLERİN MEKANİK ÖZELLİKLERİ

Anahtar Kelimeler

Mekanik, Özellikler, Bor, Dopingli, Çinko, Oksit, Piroliz.

Öz

Bu çalışma, bor katkılı Çinko Oksit (ZnO) ince filmin mekanik özelliklerini belirlemeyi amaçlamaktadır. Mekanik özellikler mühendislik uygulamaları için önemlidir ve mühendislik malzemelerinin odak noktasıdır. Sprey piroliz işlemi kullanılarak, soda kireç camı alt tabaka üzerinde değişken bir oranda ince bir bor katkılı (ZnO) kaplama oluşturulmuştur. Film farklı sıcaklıklarda biriktirilirken alt tabakanın sıcaklığını izlemek için bir demir-konstantan termokupl kullanılmıştır. Katkılı ve katkısız numunelerin sertlik, darbe ve gerilme mukavemeti gibi mekanik özelliklerini belirlemek için metalurjik mikroskopi kullanılmıştır. Sonuçlar, çinko oksit katkılandığında, darbe ve sertlik değerlerinde değişimler tespit edildiğini ve maksimum gerilimin katkı konsantrasyonu ile arttığını göstermiştir. Bununla birlikte, çinko oksit katkısız olduğunda, gerilme ve sertlik sıcaklıkla artmaktadır.

Cite

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Highlights (At least 3 and maxium 4 sentences)

- Effect of dopant solution on ZnO thin films was studied
- Boron was proposed as a suitable dopant solution
- Additon of the dopant solution resulted in an improved grain size
- The improved grain size enhanced the mechanical characteristics of the ZnO thin films

Purpose and Scope

The purpose of carrying out a study on Boron-doped Zinc Oxide thin fillms is to characterise thin films and determine the effect of dopant solution on their mechanical properties. The scope is to investigate if boron will improve the mechanical properties of ZnO thin films.

Design/methodology/approach

To determine the effect of dopant solution on ZnO thin films, a metallurgical microscope was used and the mechanical properties were determined by using x-ray diffractometer. The dopant solution, and temperature was varied respectively.

Findings

The study findings reveal that dopant solution can be added to ZnO thin films in order to improve the properties, but the addition should be at an appropriate proportion. The dopant solution increases the size of thin films and improves the mecanical properties.

Social Implications

Thin films have potential applications in heterojunction solar cell devices, flexible electronics, and other optoelectronic devices. Doping of thin films improve their characteristics.

Originality

The novelty of this study is the varied app;ication of dopant solution on ZnO thin films by using spray pyrolysis technique and increased temperature. The research will aid solar techniques for homes, business and industries. This study can assist in enhancing researchers understanding on behavior of thin films and contibute to the development of innovative materials.

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1. Introduction

Due to its unique physical and chemical properties, zinc oxide (ZnO), a chemical molecule that is naturally occurring in the mineral zincite, has a broad band gap semiconducting substance with numerous applications (Choi et al., 2001, Mohammed Nahhas, 2018). With a large excitation binding energy of 60 meV, it is one of the semiconductor materials that optoelectronic applications are looking at the most. Single crystals, powders, thin films, and nanostructures are some of its structural forms. According to Asif et al. (2018) and Naqvi et al. (2018), it is a significant metal-oxide based semiconductor with good, non-toxic, and processable features appropriate for opto-electronics, sensor/transducer, data storage, batteries, personal care goods, and bio-chemical sensor applications. In addition, it can be utilised as a photo detector, solar cell, light-emitting diode, and solid-state lighting source (Kim et al., 2007, Berginski et al., 2007). Ecologically friendly and plentiful (Tumbul et al., 2018), its broad and straight band gap of around ~3.37 eV is one of its physical properties (Willander et al., 2009). It can be utilized in photodetectors, solar cells, and light-emitting diode displays (Kim et al., 2007). ZnO possesses significant thermal conductivity, which is advantageous in additive manufacturing applications like tyre production (Özgür et al., 2006; Paulthangam et al., 2022). Additionally, its temperature allows for the creation of photoelectronic and highly optically efficient ZnO-based devices. Furthermore, ZnO thin-film applications for short wavelength optoelectronic devices are stress-free due to their large bandgap (Znaidi, 2010). ZnO provides appropriate electrical characteristics when combined with other materials (Khan et al., 2013). Similarly, its extremely piezoelectric nature results from its non-central configuration, also referred to as its wurtzite structure. This is a crucial attribute when building electromechanically coupled sensors and transducers (Desai and Haque, 2007).

Doping thin film with an appropriate dopant can improve the mechanical properties of this somewhat soft material, which has a low Mohs hardness of approximately 4.5 (Huzni et al., 2021, Badreddine et al., 2020). Thus, ZnO can be produced using the following methods: Sol gel (Berestok et al., 2012), chemical spray pyrolysis (Animasahun, 2019), RF thermal-plasma evaporation (Khun et al., 2021), and electron beam evaporation (Dwivedi et al., 2013).

According to Hong et al. (2011), a thin film is a layer of material that ranges in thickness from nanometer to micrometre. One may say that substrates are coated with a thin layer, and typical appliances have thin films—that is, nanostructures on sturdy substrates—(Cordill et al., 2022, Xiao and Dorey, 2008). Deposition, the controlled synthesis of materials like thin films, is a crucial stage in a number of applications (Jilani et al., 2017). A common illustration is the home mirror, which often has a small layer of metal applied to the rear to provide a reflective interface (Ştefanov et al., 2020). Because ZnO is a good material for solar cells, UV light emitters, gas sensors, surface acoustic wave devices, transparent high-power electronics, etc., it has been receiving a lot of attention (Pearton and Ren, 2014). A substrate is a surface that is covered in layers of material. It is a supporting substance that gives thin films mechanical support, sufficient adhesion, and an atomic arrangement template. Nonetheless, the thin films that are formed on a substrate have a significant impact on the features of the film (Phillips, 2013). Substrates include things like glassware, quarts, semiconductor wafers, ceramics, and stainless steel. Surfaces of substrates need to be clean, polished, and free of holes, chips, and fractures that could result from machining or polishing (Yilbas, 2014). Substrates are a crucial component in thin-film device design and construction (Catania et al., 2022). In order to perform spray pyrolysis, a chemical solution must be atomized into tiny droplets and then applied to a heated substrate by means of a gas that forms thin films. Spray pyrolysis is a low-cost, straightforward process that uses basic equipment, can produce films of any composition, and doesn't require expensive or complex chemicals (Tahir et al., 2020).

According to Jana et al. (2011), boron doping entails utilising boric acid as the boron source. When cavities are discernible in a thin layer, the concentration of boron will rise. By doping ZnO with boron at different temperatures, one may ascertain the semiconducting characteristics of thin films (Dash et al., 2018). Due to its high electron mobility, luminescence, direct band gap energy, and suitable temperature, doping ZnO results in modifications to its optical, electrical, electronic, mechanical, magnetic, and structural properties, which in turn lead to enhancements in various applications (Sharma et al., 2022). Therefore, the modification of ZnO properties by addition of impurity/dopant is of interest. According to Skorenko et al. (2016) and Chiba et al. (2014), ZnO exhibits enhanced electrical and chemical stability together with a high surface-to-volume ratio. In this work, spray pyrolysis is used to dope ZnO thin films with boron. The low temperature need, cost-effectiveness, and non-toxicity of the chemical spray pyrolysis process make it a viable option.

2. Material and Method

2.1. Materials

Substrate, zinc acetate dehydrate, tri-methylborate, methanol, and acetic acid are among the materials used in this investigation. Soda lime glass substrate is the substrate that is used. One precursor solution that was utilised was zinc acetate dehydrate. Tri-methylborate is employed as a dopant solution. Acetate and methanol are combined in a 3:1 ratio to act as a solvent. To create a transparent and clear solution, acetic acid, also a dopant solution, was added.

2.2. Equipment used

Substrate cutter, beaker, grinding machine, metallurgical microscope, Charpy impact tester, Brinell hardness tester, and X-ray diffractometer are among the tools utilised.

2.3. Sample Preparation

2.3.1. Chemical Composition

The required mass, m, of the Zinc Acetate was determined by using Equation (1) derived from basic equations of number of moles.

$$m = C \times V \times M \quad (1)$$

Where $C\ is\ the\ molar\ concentraion\ mol/dm^3$

$V\ is\ the\ molar\ volume\ in\ \ dm^3$

$M\ is\ molar\ mass\ in\ g/mol$

In order to obtain molar concentration of Zinc Acetate of $0.2\ mol/dm^3$ by dissolving a calculated mass of sample in $0.1\ dm^3$ $(100\ ml)$ of solvent, the molar mass of Zinc Acetate is $219\ g/mol$ and by substitution, the required mass becomes,

$$m = 0.2 \times 0.1 \times 219$$

$$m = \ 4.38\ g$$

2.3.2. Preparation of the solution

$4.38\ g$ of Zinc Acetate is weighed and dissolved in $100\ ml$ solvent of Acetone and Methanol. The ratio for the solution is 3:1, that is, 3% of Methanol and 1% of Acetone, the respective volume becomes,

$$\tfrac{3}{4} \times 100\ ml\ =\ 75\ ml\ of\ methanol$$

$$\frac{1}{4}\ \times\ 100\ =\ 25\ ml\ of\ methanol$$

After adding three drops of acetic acid to the mixture, a transparent and clear solution was produced. To obtain a 2% dopant of tri-methylborate, 2 ml of the 100 ml solution was withdrawn and replaced with 2 ml of tri-methylborate. After that, the solution was shaken in the beaker using a magnetic stirrer that was powered by electricity for nearly two hours. For the 3% and 4% solutions, this procedure was repeated.

2.3.3. Chemical Spray Pyrolysis Setup

The iron-constantan thermocouple wire, hot plate, flow metre, air atomizer nozzle, air compressor, receptacle, and temperature controller assembled in a flame chamber make up the chemical spray pyrolysis system components. The film was applied by spraying certain prepared precursors—0%, 2%, 3%, and 4% doped, respectively—onto a heated substrate that was set on a hot plate using an air brush atomizer with a 0.2 mm spray nozzle and compressed air at a pressure of roughly 10^5 Pa as the carrier gas. A temperature controller was utilised to monitor the temperature of the substrate through the use of an iron-constantan thermocouple. Three specific temperatures of (300±5) °C, (350±5) °C, and (375±5) °C were used to deposit the film. To achieve optimal deposition in the spray system, it was necessary to clean the system components and place them in a manner that would guarantee experimental uniformity. To rid the tube and nozzle of any other solutions sprayed earlier, at least 3 ml of solvent were sprayed. To reduce contamination, it is usually best to use the same solvent that was used to prepare the precursor. To ensure experimental consistency and minimise the deposition area, a substrate reference point was established on the system base. This approach avoids the need for additional stress during the spraying of system components. As a result, the substrate's distance from the spraying nozzle's tip was maintained at 27 cm. For this investigation, a stepwise multi-pass spraying technique was used. A maximum volume of 2 millilitres of the precursor was applied via spraying in each of the procedural stages. This is required to have a thin film that is largely uniform, free of pinholes and cracks.

2.4. Mechanical properties

Mechanical properties examined in this study include hardness, impact and tensile properties.

2.4.1. Hardness test
After grinding, samples were completely cleaned and polished to provide a better indentation. In addition, a Brinell Hardness testing equipment was used to measure the samples' hardness, and the results were documented in accordance with ASTM E10.

2.4.2. Impact test
An impact testing machine operating in Charpy mode was used to test the impact toughness of the produced samples. As per the ASTM E23 test standard, test samples were created. Every sample's impact strength was computed and noted.

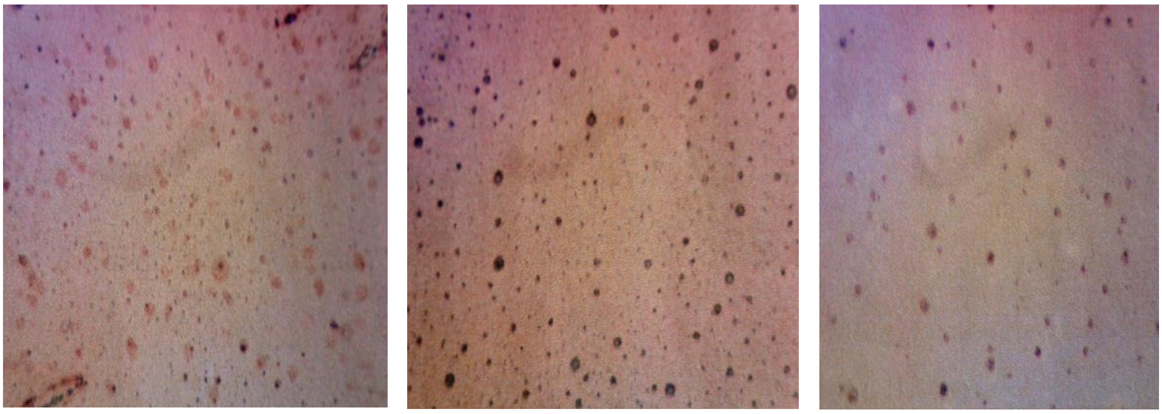
2.4.3. Tensile test
Tensile test was conducted on samples in order to reveal their tensile properties, by using X ray diffractometer according to ASTM D638.

3. Result and Discussion

3.1. Undoped ZnO film

3.1.1. Metallurgical Analysis

ZnO films were placed on a substrate made of soda lime glass, and metallurgical microscopy observations were made. Figure 1 illustrates how the grain size is affected by undoped ZnO. The ZnO films are devoid of dopant solution, and metallurgical microscopy shows that the grain size of the ZnO film does not grow when temperature is raised for A1, A2, and A3 from 300 to 350 and 375°C, respectively.



(a) A1 (b) A2 (c) A3

Figure 1: Metallurgical microscope image of undoped ZnO thin films

3.1.2. X-Ray Diffractometry Analysis

3.1.2.1. Mechanical Properties

The mechanical characteristics of undoped ZnO thin films produced by spraying at higher temperatures were ascertained. Table 1 displays the impact and hardness values of the undoped ZnO derived from the X-ray diffractometry investigation. The hardness and impact values are 35.17 and 19.48 at 300°C, 36.72 and 19.50 at 350°C, and 37.39 and 19.58 at 375°C. Temperature affects both the impact and hardness ratings.

Table 1. Mechanical properties of 0% solution

Sample	Temperature (°C)	Hardness Value	Impact Value
A1	300	35.17	19.48
A2	350	36.72	19.50
A3	375	37.39	19.58

3.1.2.2. Tensile Properties
The tensile properties of undoped ZnO film are determined by use of x-ray diffraction patterns. The tensile properties of the undoped ZnO under study show that the maximum tensile stress increases with temperature, as seen in Figure 2, which

plots tensile stress (MPa) versus tensile strain (mm/mm). At 300°C, the maximum tensile strain and stress are 0.00357 (mm/mm) and 0.22096 MPa, respectively. The percentage elongation is 3.57%, the percentage reduction is 0.00359%, and the Young's modulus is 6.189 MPa. At 350°C, the maximum tensile stress and strain are 0.01489 (mm/mm) and 0.43988 MPa, respectively. 29.54 MPa is the Young's modulus, 1.489% is the percentage of elongation, and 0.0003592 is the percentage of reduction. At 375°C, the maximum tensile stress and strain are 0.000637 (mm/mm) and 0.77804 MPa, respectively. The percentage reduction is 0.003593%, the percentage elongation is 0.448, and the Young's modulus is 189.71 MPa. Although the tensile strain fluctuates with temperature, the tensile stress increases.

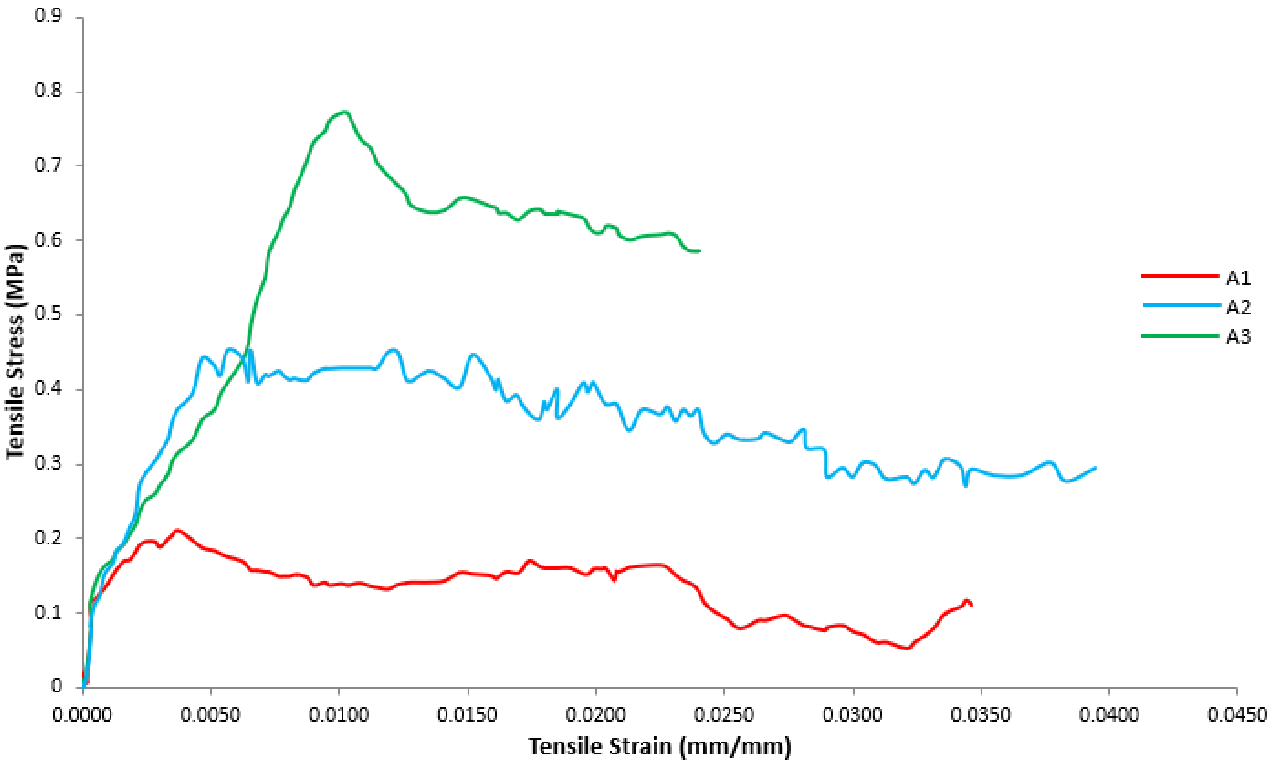
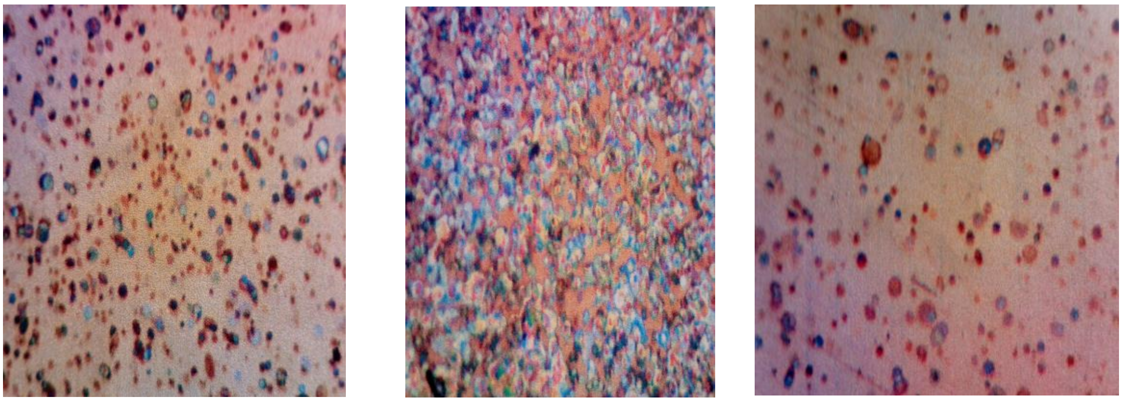


Figure 2: Graph of tensile stress against tensile strain of A1, A2, A3 at 0% dopant composition

3.2. 2% boron-doped ZnO film

3.2.1. Metallurgical Analysis

ZnO films were placed on a substrate made of soda lime glass, and metallurgical microscopy observations were made. Figure 3 illustrates how the grain size of the ZnO films rises with temperature when deposited in a 2% dopant solution at 300, 350, and 375°C. This is in agreement with literature (Saadeldin et al., 2019, Temiz et al., 2020).



(a) B1 (b) B2 (c) B3

Figure 3: Metallurgical microscope image of ZnO films for 2% dopant solution

3.2.2. X-ray Diffraction Analysis

3.2.2.1. Mechanical Properties

The mechanical characteristics of 2% boron-doped ZnO thin films produced by spraying technique under varied temperature conditions were ascertained. Table 2 displays the undoped ZnO's impact and hardness values. The values of hardness and impact are 35.17 and 19.48 J at 300°C, 36.72 and 19.50 J at 350°C, and 37.39 and 19.58 J at 375°C. In contrast to the undoped ZnO thin film, the hardness values for the 2% boron-doped ZnO thin film drop with temperature, which is inconsistent with previous research (Senol et al., 2019). However, the impact and hardness values increase with temperature.

Table 2. Mechanical properties of 2% solution

Sample	Temperature (°C)	Hardness (BHN)	Impact (J)
B1	300	23.30	19.86
B2	350	24.08	19.86
B3	375	23.62	19.93

3.2.2.2. Tensile Properties

The x-ray diffraction pattern of a 2% boron-doped ZnO thin film is used to further determine the tensile properties of doped ZnO films. Figure 4 shows a graph of tensile stress (MPa) against tensile strain (mm/mm) at 2% concentration. The mean maximum stress is 0.96006 MPa, the mean strain is 0.10912 (mm/mm), the percentage elongation is 10.918%, and the percentage reduction is 0.027833%. Young's modulus is 31.659 MPa. The observed doped ZnO's tensile characteristics show that when strain increases, the mean maximum tensile stress does as well.

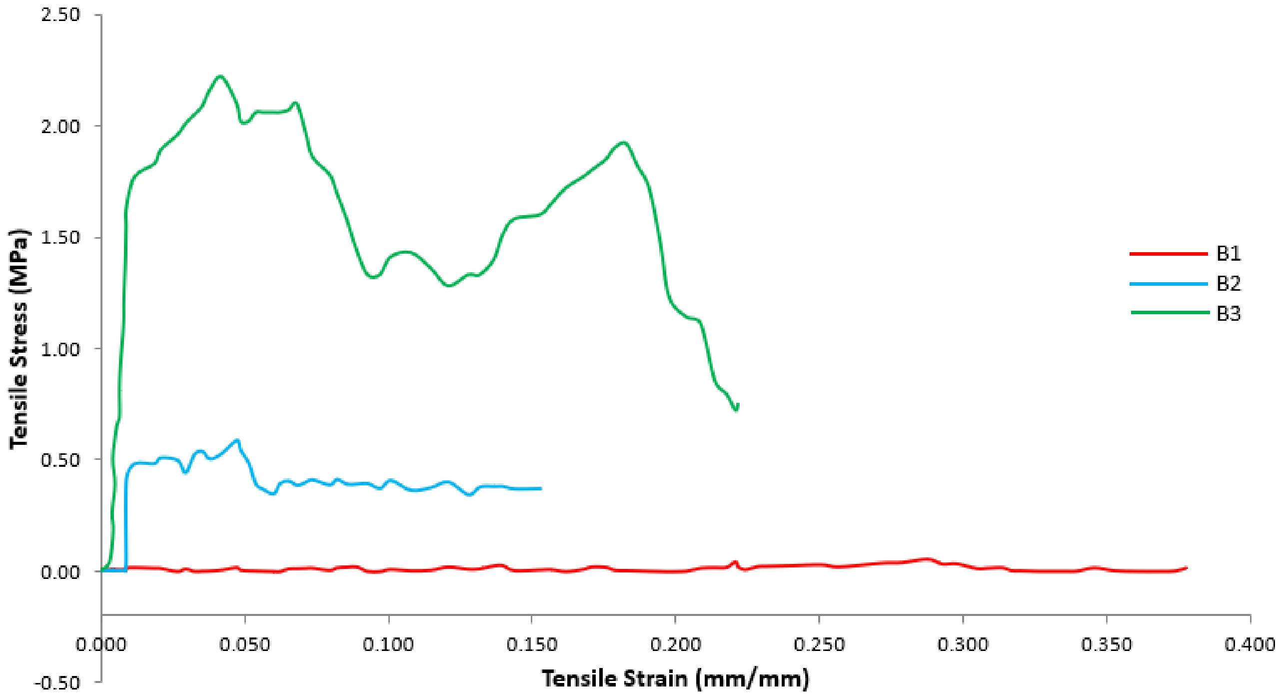
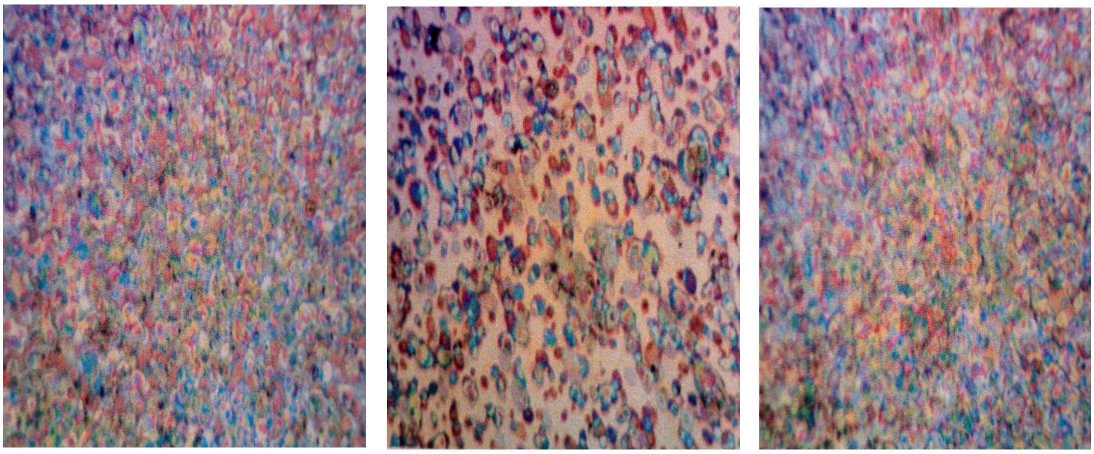


Figure 4: Graph of tensile stress against tensile strain of B1, B2, B3 at 2% dopant composition

3.3. 3% boron-doped ZnO film

3.3.1. Metallurgical Analysis

ZnO films were placed on a substrate made of soda lime glass, and metallurgical microscopy observations were made. Figure 5 illustrates how the grain size of the ZnO films rises with temperature when deposited in a 3% dopant solution at 300, 350, and 375°C. This is consistent with literature (Saadeldin et al., 2019, Temiz et al., 2020).



(a) C1 (b) C2 (c) C3

Figure 5: Metallurgical microscope image of ZnO films for 3% dopant

3.3.2. X-ray Diffraction Analysis

3.3.2.1. Mechanical Properties

Using an X-ray diffractometer, the mechanical characteristics of 3% boron-doped ZnO thin films produced by spraying technique under varied temperature conditions were ascertained. Table 3 displays the 3% doped ZnO's impact and hardness values. At 300°C, the impact value is 19.72 J, the hardness value is 15.13, and at 350°C, the hardness and impact values are 14.82 and 20.30 J. Finally, at 375°C, the values are 15.50 and 19.86 J for both hardness and impact. Both the impact and hardness levels are not constant when the dopant concentration is raised to 3%. But in contrast to previous study, the hardness values for the 3% dopant solution are lower than those of the undoped solution (Senol et al., 2019) and lower than values obtained for 2% dopant solution.

Table 3. Mechanical properties of 3% solution

Sample	Temperature (°C)	Hardness Value	Impact Value (J)
C1	300	15.13	19.72
C2	350	14.82	20.30
C3	375	15.50	19.86

3.3.2.2. Tensile Properties

The x-ray diffraction pattern of a 3% boron-doped ZnO thin film is used to further determine the tensile properties of doped ZnO films. The mean maximum tensile stress rises with an increase in dopant concentration, according to the tensile characteristics of doped ZnO measured using an X-ray diffractometer. Figure 6 shows a graph of tensile stress (MPa) against tensile strain (mm/mm) at 3% concentration. The mean maximum stress is 1.23543 MPa, the mean strain is 0.02179 (mm/mm), the Young's modulus is 67.014 MPa, the percentage elongation is 2.1796%, and the percentage decrease is 0.027833%. When the dopant additive is raised to 3%, the mean maximum tensile stress rises.

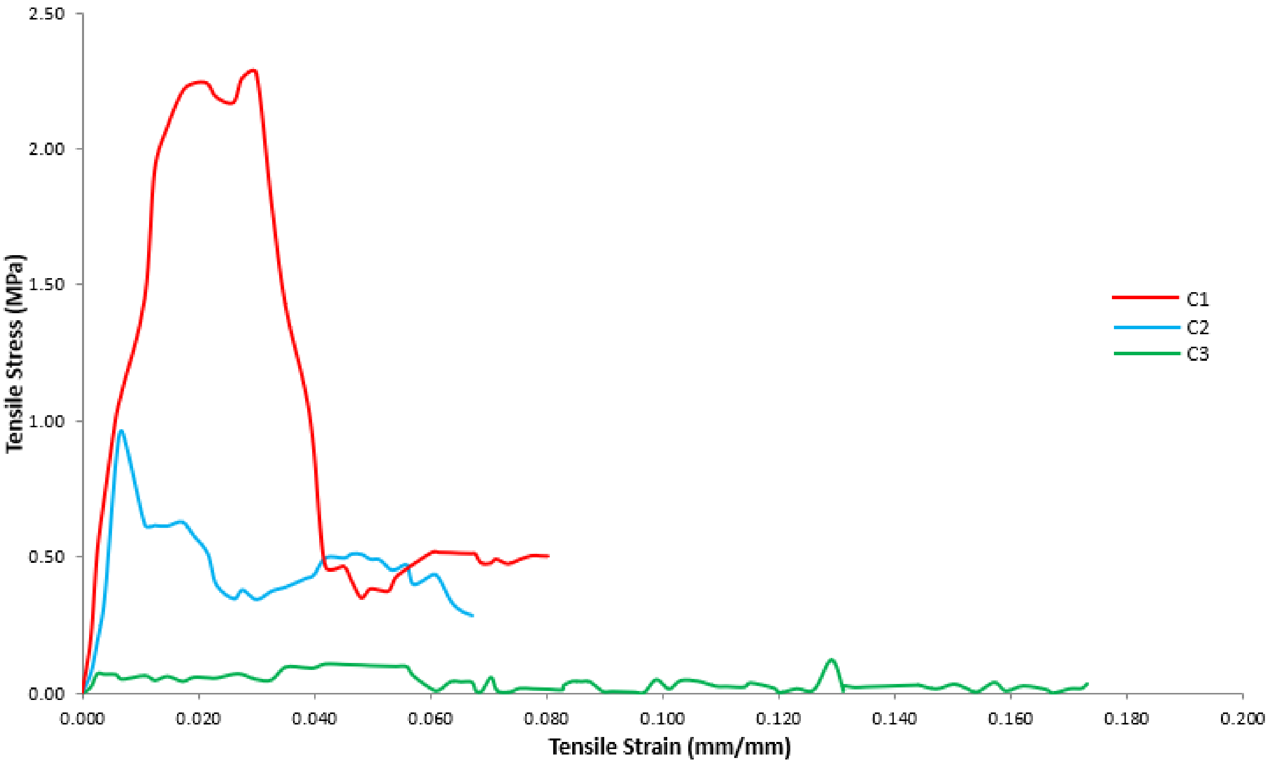
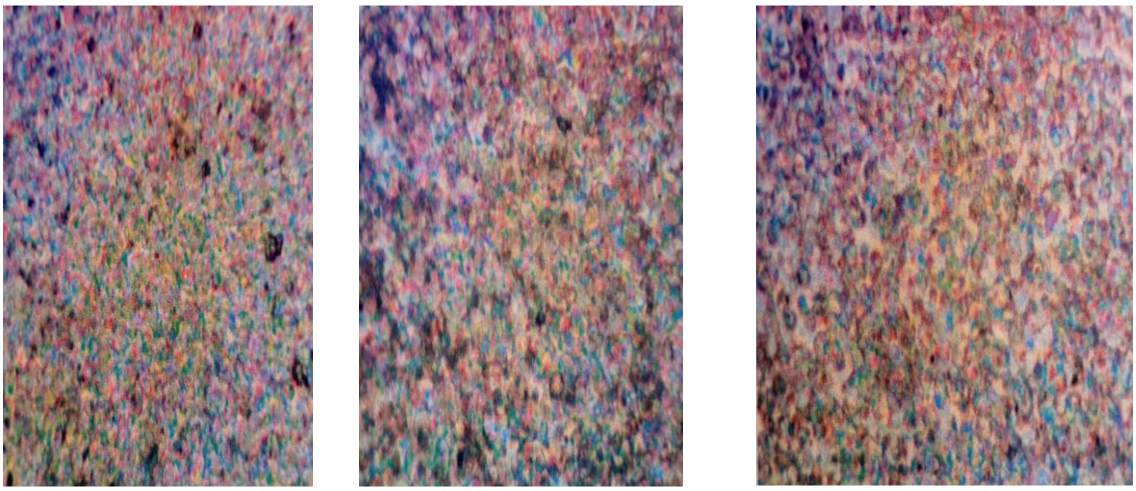


Figure 6: Graph of tensile stress against tensile strain of C1, C2, C3 at 3% dopant composition

3.4. 4% Dopant Solution

3.4.1. Metallurgical Analysis

ZnO films were placed on a substrate made of soda lime glass, and metallurgical microscopy observations were made. Figure 7 illustrates how the grain size of the ZnO films rises with temperature when deposited in a 4% dopant solution at 300, 350, and 375°C. This is consistent with previous research (Saadeldin et al., 2019, Temiz et al., 2020).



(a) D1 (b) D2 (c) D3

Figure 7: Metallurgical microscope image of ZnO films for 4% dopant solution

3.4.2. X-ray Diffraction Analysis

3.4.2.1. Mechanical Properties

Using an X-ray diffractometer, the mechanical characteristics of 4% boron-doped ZnO thin films produced by spraying technique under varied temperature conditions were ascertained. Table 4 displays the undoped ZnO's impact and hardness values. Hardness and impact values are as follows: at 300°C, 12.80 BHN and 19.99 J; at 350°C, 12.02 BHN and 20.50 J;

and at 375°C, 12.48 BHN and 20.40 J are the values. In comparison to data obtained from a 3% dopant solution, the hardness values are lower and the impact values are higher. In contrast to previous research, the hardness values for the 4% dopant solution are lower than those of the undoped solution (Senol et al., 2019).

Table 4. D1 – D4 4% dopant solution

Sample	Hardness Value	Impact Value (J)
D1	12.80	19.99
D2	12.02	20.50
D3	12.48	20.40

3.4.2.2. Tensile Properties

X-ray analysis was used to assess the mechanical characteristics of 4% boron-doped ZnO thin films that were created by spraying technique at varied temperatures. The x-ray diffraction pattern of a 4% boron-doped ZnO thin film is used to further determine the tensile properties of doped ZnO films. The X-ray diffractometer's analysis of the tensile characteristics of 4% doped ZnO shows that an increase in dopant concentration results in a rise in the mean maximum tensile stress. Tensile stress (MPa) vs tensile strain (mm/mm) is plotted in Figure 4 at 4% D concentration. The mean maximum stress is 144405 MPa, the mean strain is 0.01487 (mm/mm), the Young's modulus is 137.009 MPa, the percentage elongation is 1.2197%, and the percentage reduction is 0.001174%.

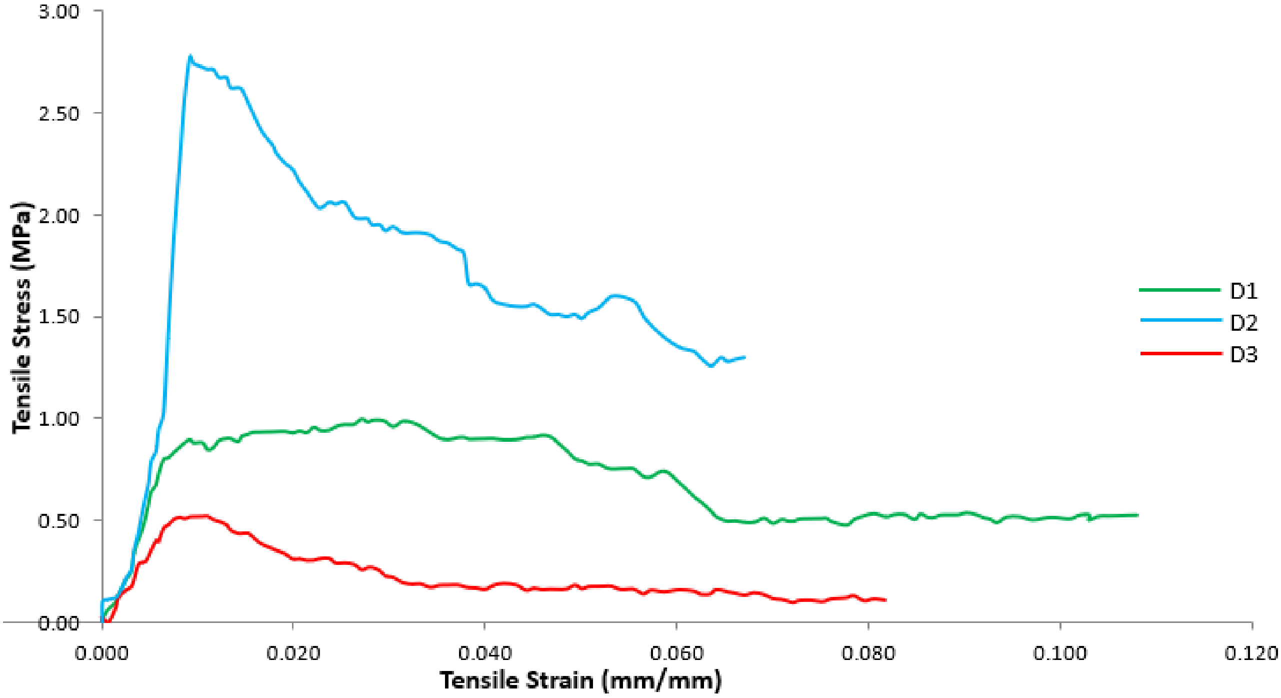


Figure 8: Graph of tensile stress against tensile strain of D1, D2, D3 at 4% dopant composition

4. Conclusion

According to the study's findings, the addition of dopant solution causes the grain size of thin films to grow. As glass experiences plastic deformation, its impact, hardness, and tensile strength all increase. Temperature influences the maximum stress of undoped ZnO and boosts its hardness and impact. With increasing dopant concentration, doped ZnO's maximum stress and impact value continued to rise while its hardness decreases. The investigation's conclusions suggested that doping ZnO thin films with boron in the right ratio will improve their mechanical properties, and that spray pyrolysis is an affordable method for producing them. Other optoelectronic devices and heterojunction solar cell technology may find applications for these films.

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Conflict of Interest

The authors hereby declare that there is no conflict of interest

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