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Araştırma / Research

INVESTIGATION OF THE EFFECT OF N_2 GAS FLOW RATE ON THE OPTICAL, STRUCTURAL AND MORPHOLOGICAL PROPERTIES OF INDIUM-GALLIUM-NITRIDE TRIPLE THIN FILMS OBTAINED BY SPUTTER TECHNIQUE

Erman ERDOĞAN[1] (ORCID: 0000-0003-2566-3284)*
Mutlu KUNDAKÇI[2] (ORCID: 0000-0002-1764-2566)

[1]*Department of Physics, Faculty of Art and Science, Muş Alparslan University, Muş, 49250, Turkey*
[2]*Department of Physics, Faculty of Science, Atatürk University, Erzurum, 25240, Turkey*

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ABSTRACT

In this research work, InGaN (Indium Gallium Nitride) triple compound was grown under different N_2 gas flow rates by using sputtering technique. The structural, optical and morphological characteristics of the InGaN compound have been studied in detail. In the XRD analysis, films exhibited hexagonal crystal structure. The films appear at lower wavelengths in visible region and absorption values begin to increase sharply from about 550-560 nm and reach the highest absorption value in the Near-UV region. When gas flow rates increased, the optical band gaps of the film increased. In SEM, the film exhibits dense coverage of the material on the surface of the substrate without the presence of voids, pinholes or cracks. In the results of the AFM, there are locally peaks and valleys, and partially homogeneous and circular-like clusters are arranged. Films are suitable structures for use in device applications.

Keywords: III-V compounds, sputtering, nitrogen gas flow, physical characteristics, thin films

N_2 GAZ AKIŞ HIZININ, SPUTTER TEKNİĞİNDEN ELDE EDİLEN İNDİYUM-GALYUM-NİTRÜR ÜÇLÜ İNCE FİLMİNİN OPTİK, YAPISAL VE MORFOLOJİK ÖZELLİKLERİNE ETKİSİNİN İNCELENMESİ

ÖZET

Bu araştırma çalışmasında, InGaN (İndiyum Galyum Nitrür) üçlü bileşiği, saçtırma tekniği kullanılarak farklı N_2 gaz akış oranları altında büyütülmüştür. InGaN bileşiğinin yapısal, optik ve morfolojik özellikleri detaylı olarak incelenmiştir. XRD analizinde, filmler hegzagonal kristal yapı sergilemiştir. Filmler, görünür bölgede daha düşük dalga boylarında ortaya çıkmıştır ve soğurma değerleri, yaklaşık 550-560 nm'den itibaren keskin bir şekilde artmaya başlar ve Near-UV bölgesinde en yüksek soğurma değerine ulaşmıştır. Gaz akış hızı arttığında, filmin optik bant aralıkları artmıştır. SEM'de film, altlığın yüzeyi üzerindeki malzemenin boşluklar, iğne delikleri veya çatlaklar olmadan yoğun bir şekilde kaplanmasını göstermiştir. AFM sonuçlarında, yerel tepeler ve vadiler vardır ve kısmen homojen ve dairesel benzeri kümeler düzenlenmiştir. Filmler, cihaz uygulamalarında kullanım için uygun yapılardır.

Keywords: III-V bileşikleri, saçtırma, azot gazı akışı, fiziksel özellikler, ince filmler

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*Corresponding author / Sorumlu yazar. Tel.: +90 0436 249 4949; e-mail / e-posta: e.erdogan@alparslan.edu.tr

1. INTRODUCTION

Rapid developments in today's technology are in line with the understanding of the properties of large-scale semiconductors and the growth of new compound semiconductors. Therefore, this situation increases the importance of semiconductors and causes more and more researchers to focus on this topic day by day. For this reason, semiconductor technology emerges as one of the elements that pave the way for the advancement of science and technology, contributes to the development of living conditions in many ways [1]. When we enter the 21st century, semiconductor nanostructures are widely used in the fields of electronics, optoelectronics and photovoltaics. Especially in optoelectronic applications; in applications that emit and detect light in the visible region of the semiconductor electromagnetic spectrum that has a direct band gap structure, the magnitudes of the optical band gaps allow for use in the entire visible region. Thin film can be produced either directly by a physical process by controlling the condensation of atomic, molecular or ionic species onto a solid material as a substrate, chemical or electrochemical reactions [2]. III-nitride semiconductors have many uses in optoelectronic and electronic technology due to their physical properties [3]. The optical band gaps of these compounds range from a near infrared region to the ultraviolet region (0.7 eV (InN), 3.4 eV (GaN) and 6.1 eV (AlN)) of the electromagnetic spectrum [4]. This feature is crucial in the production and development of many optoelectronic devices such as light-emitting diodes (LEDs), laser diodes, photodetectors, optical modulators and switching devices operating in visible and ultraviolet regions [5].

Growth of InGaN thin films can be obtained by using several methods such as; molecular beam epitaxy (MBE) [6-7], metal organic chemical vapor deposition (MOCVD) [8-9] and sputtering technique [10-16]. Sputtering thin film growth technique is basically a process in which after an inert gas (noble gas, usually argon), which is sent between two differently polarized electrodes, is converted into a positive ion, the material is accelerated towards the target material in the cathode (negative electrode) these scraped particles will grow one by one on the base placed exactly opposite the target [17-18].

Although the r.f. sputtering technique seems to complicate the production process, which has many production parameters, the ability to produce high quality homogeneous films carries the sputtering technique to an important position for almost all known target materials. Applied r.f. power, voltage difference and distance between anode and cathode, substrate temperature, distance between target material and substrate, oxygen and argon partial pressures, substrate heating and cooling times, working pressure and gas flow rate parameters are used for sputtering technique. Among these parameters, different parameters determine the different properties of produced films. Only small parts of the improved properties were obtained under gas flow rates, working pressures and substrate temperature. The process parameters used for the sputtering technique mentioned above will have different values for the different kinds of materials desired to be produced. These changes have a crucial factor on physical properties of the thin films. However, research of nitrogen gas flow rate effect on structural, optical and morphological properties of InGaN thin films growth on quartz substrate by sputtering method have not been investigated, yet.

Because of the fact that fundamental difficulties in obtaining materials with high crystal quality, the deposition of III-nitride epitaxial layers are intensively studied. The crystal perfection of epitaxial structures is one of the main factors affecting the physical properties of the thin films. Sapphire is the most commonly used substrate for hexagonal InGaN growth. However, due to the large discrepancy between sapphire and III-Nitride (16% for GaN on sapphire and 29% for InN on sapphire) and thermal incompatibility (-134% for GaN on sapphire and -100% on InN on sapphire) epitaxial films result in high dislocation densities [19]. Compared with other III-nitride alloys, epitaxial grown $In_xGa_{1-x}N$ films have a very high dislocation density up to 10^{10} dislocations/cm^2 [20]. Quartz is an alternative substrate for InGaN applications. In addition to this, quartz are easily available at large area production with low cost. Quartz has been extensively used for different type of materials in the form of thin films, especially growing by sputtering, as a substrate [21-30].

In this research, we try to study the effect of nitrogen gas flow rate (0 sccm, 1 sccm, 2 sccm) on morphological, structural and optical properties of InGaN thin films deposited on quartz substrate. The characterizations of the films were carried out in a broad range with XRD, AFM, SEM and UV-Vis Spectrometer and the atomic size measurements were compared with the results in the literature.

2. EXPERIMENTAL

InGaN thin films were deposited by sputtering technique using %99.95 InGaN target at East Anatolian High Technology Application and Research Center (DAYTAM). Schematic representation of the r.f. sputtering technique is shown in Fig.1. The substrate cleaning is very important in the deposition of thin films. The quartz substrates were first cleaned by a mild soap solution then degreased with acetone, etched with 5% of HCl for 30 min. ultrasonically cleaned by de-ionized water and finally dried in the air.

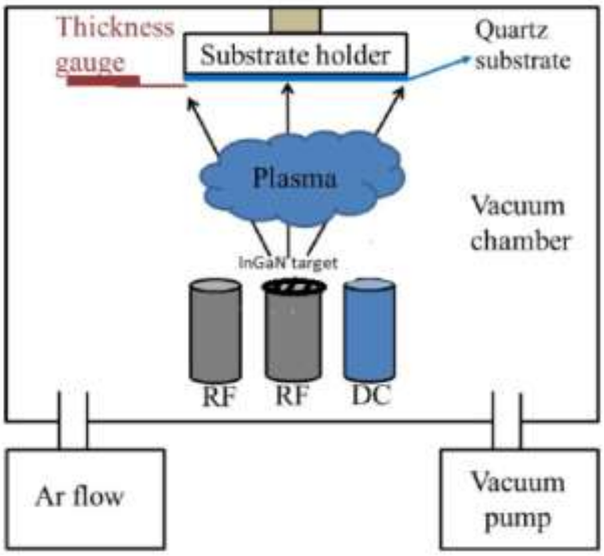

Fig. 1. Schematic representation of the r.f. sputtering technique

Structural, optical and superficial properties of InGaN thin films produced at different N_2 gas concentrations have been investigated. Before the deposition, the target was pre-sputtered for a few min each time to remove any impurities from the target surface and to reach stability conditions. The experimental parameters used to obtain InGaN thin films by sputtering are shown in Table 1.

Table 1. Growth parameters of InGaN thin films for different N_2 gas flow rates

Nitrogen Gas Flow	Argon Gas Flow	RF Power	Substrate Temp.	Base Pressure	Growth Pressure	Growth Rate	Growth Time
0 sccm	50 sccm	75W	300°C	$8x10^{-7}$ Torr	8.1 mTorr	0.2 Å/s	65 min
1 sccm	50 sccm	75 W	300°C	$1.2x10^{-6}$ Torr	8.1 mTorr	0.2 Å/s	75 min
2 sccm	50 sccm	75 W	300°C	$6.8x10^{-7}$ Torr	8.1 mTorr	0.2 Å/s	91 min

During the deposition the substrate stage maintained a constant rotation speed. The sputtering was manually terminated and the approximately 150 nm thickness of the thin film was obtained by using an Au coated quartz crystal thickness monitor controlling. In addition, the film thicknesses were confirmed after deposition with the help of a profilometer, and the thickness of all the obtained films was measured as 150 nm. Microstructures, crystal orientations and crystallographic information of InGaN thin films produced by reactive r.f. sputtering technique were obtained by using PANalytical Empyrean X-ray diffraction device. AFM and average surface roughness measurements were performed with Hitachi AFM 5000 II device to obtain information about surface structures. SEM images were taken with Sigma 300 Model Zeiss Gemini FEG-SEM device. Reflection measurements were performed using double-beam UV-vis Spectrometer with UV-3600 Plus model.

3. RESULTS AND DISCUSSION

3.1. XRD Investigations

For the determination of the structural properties of thin films XRD analysis was used. XRD analyzes of InGaN thin films at different nitrogen gas flow rates produced on quartz substrate by sputtering technique were carried out by taking diffraction patterns at 10<2θ<80 using CuK_α rays with $\lambda = 0.1540$ nm wavelength. Structural investigations were conducted with the N_2 gas flow rates of 0 sccm, 1 sccm and 2 sccm.

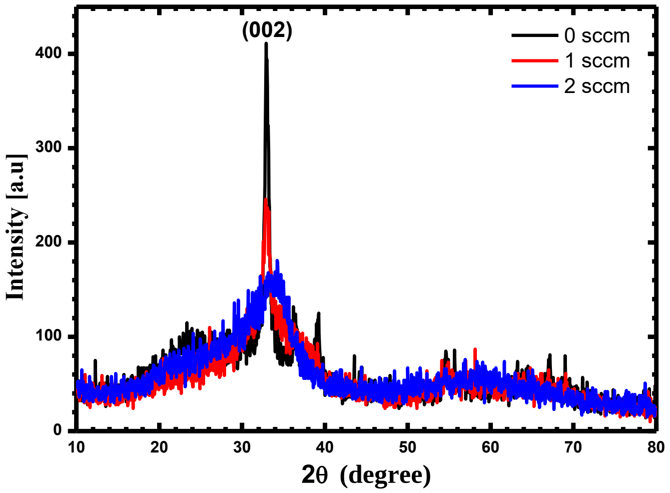

Fig. 2. XRD patterns deposited InGaN thin films at different N_2 gas flow rates

The effect of N_2 gas flow change is shown in the Fig.2. XRD patterns grown by the r.f. sputtering method on the InGaN thin film quartz substrate indicate that InGaN thin film seems to have greater intensity at 0 sccm. The N_2 gas flow rates are increased, the intensity of diffraction pattern of the InGaN thin films are decreased. The x-ray diffraction patterns of the InGaN thin films deposited on the quartz substrate for all N_2 gas flow rates exhibited single diffraction peak corresponding to the (002) peak of the InGaN.

Certain characteristics of the crystals grown by utilizing the XRD patterns are determined and given in Table 2. The particle size of the enlarged crystal size (CS) was calculated using the Debye-Scherrer formula [31-32]:

$$CS = \frac{0.94\,\lambda}{\beta\cos\theta} \tag{1}$$

where λ = 1.54050 Å. The wavelength of the x-rays is the θ Bragg diffraction angle. β is calculated from the plot along with the width of half the maximum height of the diffraction peak.

Table 2. Parameters that indicate the structural properties obtained from XRD patterns of InGaN thin film grown on quartz substrate (FWHM: full width half maximum, CS: crystal size, S: microstrain, DD: dislocation density, CN: crystal number per unit area)

Quartz/InGaN	**2θ**	**FWHM**	**CS(nm)**	**S**	**DD(1/nm^2)**	**CN(m^{-2})**
0 sccm	32.90	0.652	15.15	0.136	0.004	4.36×10^{16}
1 sccm	32.85	1.618	6.10	0.340	0.026	6.60×10^{17}
2 sccm	34.25	8.769	1.14	1.812	0.769	1.01×10^{20}

Strain (S) is defined as the deformation of an object divided by its ideal length. Micro strain broadening is the result of small changes in local lattice parameters resulting from defects, imperfections, and variations in the crystal structure. The degree of strain of the crystal is found by the following formula:

$$S = \frac{\beta cos\theta}{4} \tag{2}$$

Dislocations are linear defects in the crystal structure and they play a significant role in the growth mechanism of producing films. Dislocation density (DD) is a measure of the number of dislocations in a unit volume of a crystalline material. The dislocation density of the films is calculated by following equation:

$$DD = \frac{1}{CS^2} \tag{3}$$

Additionally, crystal number (CN) of produced InGaN thin films calculated using the following equation:

$$CN = \frac{t}{CS^3} \tag{4}$$

where t is the thickness of the producing film.

The larger crystallite size and smaller FWHM and micro strain values indicate better crystallization. The micro strain value of 0 sccm N_2 gas flow rate exhibit good crystalline film respect to low dislocation density compared to that of 1 sccm N_2 gas flow rate of InGaN film. InGaN thin films were grown on quartz substrate at different nitrogen gas flow rates with sputtering technique by Wang and colleagues. In the study, it was reported that the nitrogen gas flow rate was a major effect of changing the crystal orientation and the intensity of the peaks [33].

The high power derived from the applied high voltage difference causes the higher energy ions to impact the target material and more of the particles to break up and accumulate on the substrate, while the high amount of high power welded has a negative effect on plasma dynamics. A large number of different particles of different species with a given field of view cause undesired collisions to occur and therefore diverge from the direction in which the particles are oriented and move at different points. The reduction in the number of particles present in the plasma environment with higher energetic particles in this count will reduce the number of undesired collisions and provide the expected increase in the thin film growth rate.

3.2. Optical Investigations

In general, absorption is defined as the loss of energy within a material resulting from interaction with electromagnetic waves on electrical charges. In the basic absorption case, when the energy of a photon on a semiconductor material is equal to or greater than the forbidden energy range of the semiconductor, this photon is absorbed by an electron in the valence band of the semiconductor to form an electron pair of electrons. Thus, an electron in the valence band passes into a conductivity band.

The absorption graph of InGaN thin films at different N_2 gas flow rates is indicated in Fig.3. The absorption graph for the film obtained at 2 sccm starts at lower wavelengths in Visible region and absorption values begin to increase sharply from about 550-560 nm and reach the highest absorption value in the Near-UV region. We can say that for a film grown at 0 sccm, it starts absorbing in larger wavelengths, and then reached the lowest absorption value in the Near-UV region.

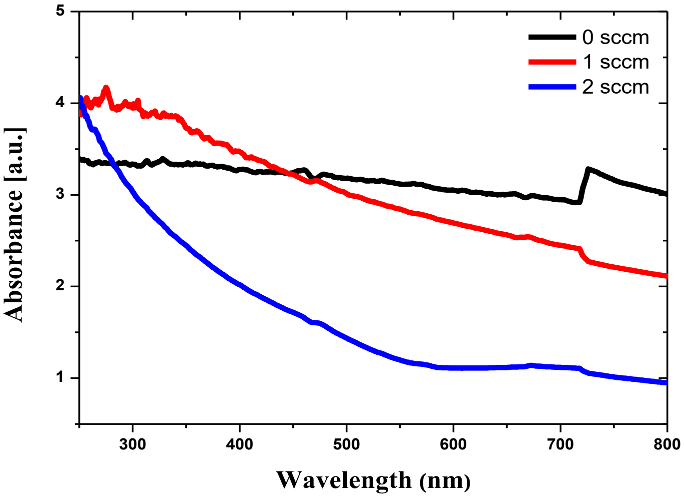

Fig.3. Absorbance graph against wavelength of the InGaN thin films grown at different N_2 gas flow rates

Using the Tauc equation, the optical band gap can be calculated [34]:

$$(\alpha h\nu) = B\left(h\nu - E_g\right)^p \tag{5}$$

where hυ is the energy of the incoming ray, B is a unitless constant that expresses the likelihood of passage of Eg films between the optical band gap value and p is a unitless constant of 0.5 for direct transitions and 2 for indirect transitions.

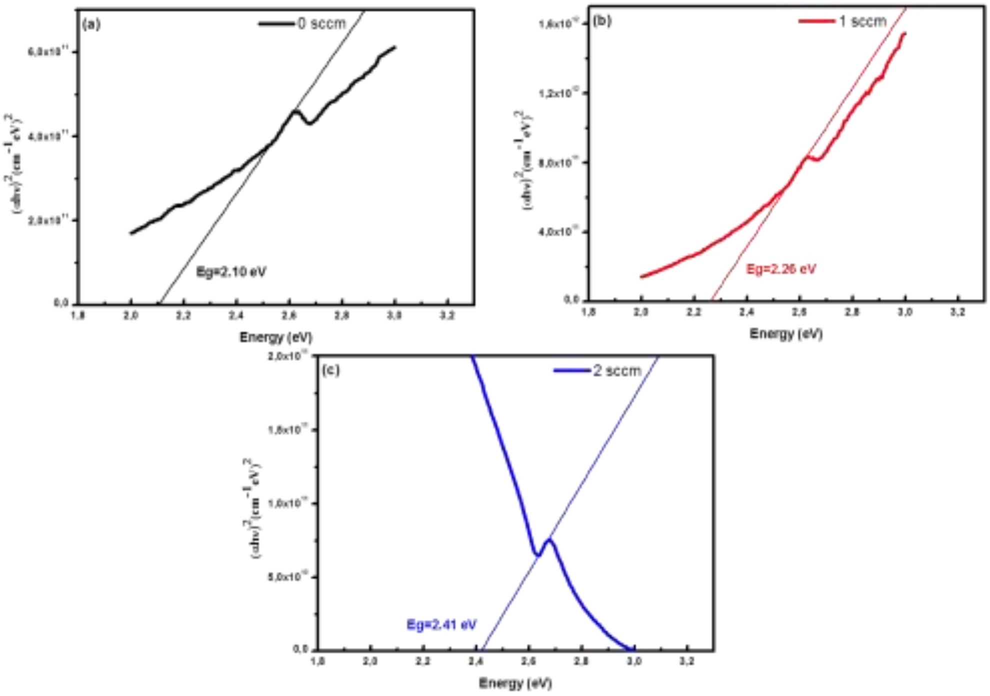

Fig.4. $(\alpha h\upsilon)^2$ (cm-1 eV)^2 against energy graphs of InGaN thin film grown at different N_2 gas flow rates

In Fig.4, the value of the band gap energy is found to be 2.10, 2.26 eV and 2.41 eV for 0 sccm, 1 sccm and 2 sccm N_2 gas flow rates, respectively. When gas flow rates increased, the optical band gaps of the film increased. Wang and his friends investigated optical properties of InGaN thin films deposited on quartz substrate with the same substrate temperature (300˚C). Their study was focused on the alteration of the working pressure. The optical band gaps of InGaN thin films for different working gas pressures have been found from 2.62 eV to 2.78 eV [35].

3.3. SEM Investigations

One of the imaging methods used to determine the morphological characteristics of the samples is a SEM. SEM images at different nitrogen gas flow rates of deposited InGaN are shown in Fig.5. Top images of the films are SEM images at 30,000 magnifications. From the view, the film was found to be intensively coated on the surface of the substrate without the voids or cracks. The film consists of normal round grains. The formation of such grains depends on the growth conditions.

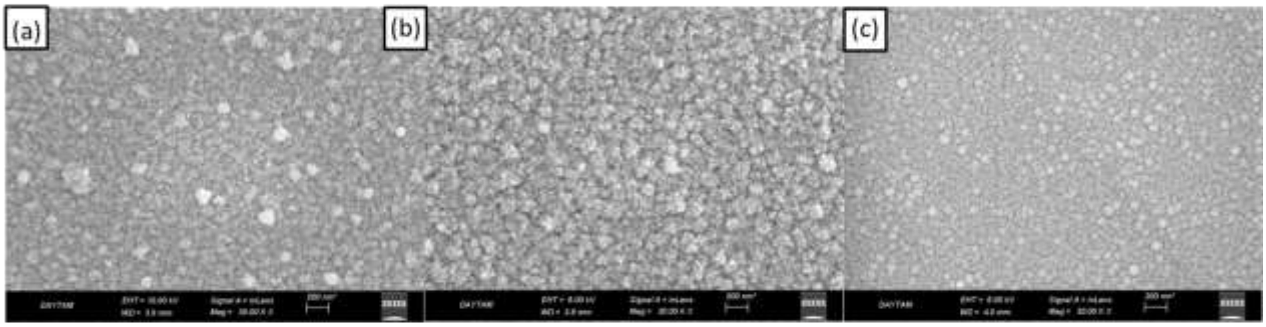

Fig. 5. SEM images of deposited films (a) 0 sccm (b) 1 sccm (c) 2 sccm N_2 gas flow rates

3.4. AFM Investigations

In Fig.6, the InGaN thin films grown on quartz by sputtering method are seen in two-dimensional and three-dimensional AFM images of 5-micron, where hills and pits are seen. The roughness value is about 8 nm with a maximum height of 27 nm and a maximum depth of 23 nm. The surface roughness value Rq\RMS value is 10 nm, which is almost consistent with the linear roughness value for 0 sccm.

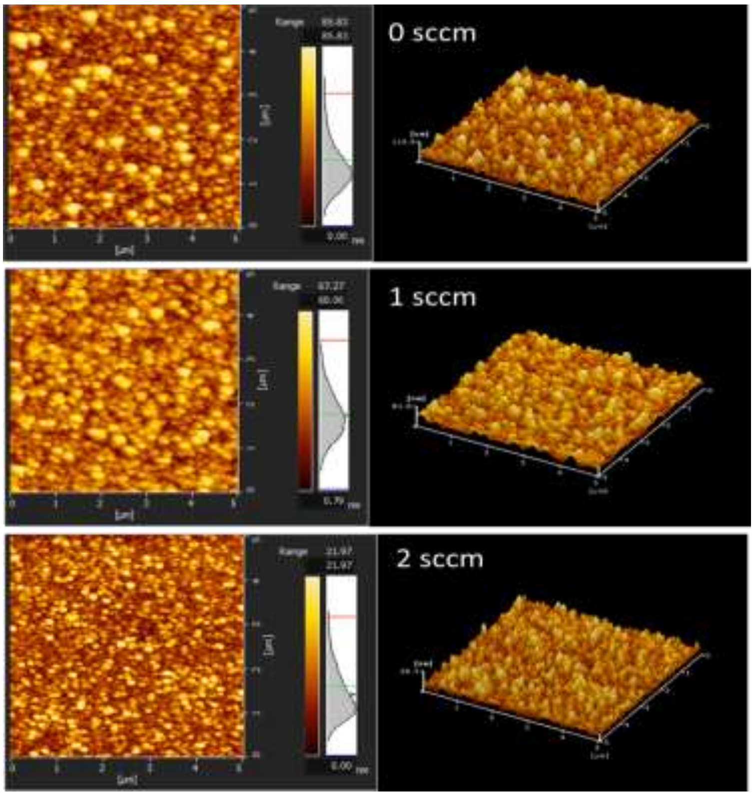

Fig. 6. Two and three dimensional AFM images of InGaN thin film grown on quartz substrate

Abud et al. synthesized $In_{0.08}Ga_{0.92}N/AlN/Si$ thin films with photo electrochemical etching technique. They have investigated the effects of etching durations on films morphology. Root mean square (rms) roughness of that film was found to be 2.2 nm [36]. The optimum nitrogen gas flow parameter for InGaN films grown at low nitrogen gas flow rates is 0 sccm, showing that the XRD results of the grown films have a crystalline structure. The increase in the flow of nitrogen gas will reduce the partial pressure of Argon and reduce the bombarded Argon ions towards the target. This causes more irregular lattice formation.

Table 3. Parameters obtained from AFM images of InGaN films deposited on different N_2 gas flow rates

	Quartz/InGaN		
	0 sccm	**1 sccm**	**2 sccm**
Roughness average(Ra)	8.26	9.20	2.56
Surface roughness (Rq)	10.53	11.19	3.58
Peak (Rp)	27.63	29.85	15.29
Valley (Rv)	22.92	28.82	5.74
Peak- Valley (Rt)	50.55	58.67	21.03
Skewness (Ssk)	1.69	0.92	0.60
Kurtosis (Skr)	41.13	11.51	20.56

The skewness value of the InGaN thin films are positive and decreases with increasing N_2 gas flow rates (see Table 3). Thus, the positive value shows that the peaks are superior on the surface. Skewness defines the asymmetry of the height being scattered and is equal to 0 for a Gaussian surface [37]. It is noticed that positive skewness values of the InGaN film state higher adhesion and contact forces and number of contacting roughness than the Gaussian case and also results in lower friction coefficient values than the Gaussian case [38].

4. CONCLUSION

In this research work, InGaN triple compound was investigated by using radio frequency magnetron sputtering technique by changing the nitrogen gas flow rates. The structural, optical and morphological characteristics of the InGaN compound have been studied in detail. InGaN thin film deposited at different gas flow changes on quartz substrate appears at lower wavelengths in visible region and absorption values begin to increase sharply from about 550-560 nm and reach the highest absorption value in the Near-UV region. When gas flow rates increased, the optical band gaps of the film increased. In general, hexagonal InGaN structure is formed from the XRD patterns grown by r.f. sputtering on the quartz substrate. The x-ray diffraction patterns of the InGaN thin films deposited on the quartz substrate for all N_2 gas flow rates exhibited single diffraction peak corresponding to the (002) peak of the InGaN. The larger crystallite size and smaller FWHM and micro strain values indicate better crystallization. From the AFM results, the roughness value is about 8 nm with a maximum height of 27 nm and a maximum depth of 23 nm. The surface roughness value Rq\RMS value is 10 nm, which is almost consistent with the linear roughness value for 0 sccm. Due to their morphological properties, we can say that they are suitable structures for optoelectronic applications and friction applications in engineering. We can also say that films with hexagonal crystal structure can be used in device applications such as LED, laser diode and power electronics. In conclusion, in the present study, the effects of N_2 gas flow rates play an important role on the properties of sputtered InGaN thin films.

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