Structural ordering, morphology and optical properties of amorphous Al\textsubscript{x}In\textsubscript{1-x}N thin films grown by plasma-assisted dual source reactive evaporation

M. Alizadeh\textsuperscript{a,⇑}, V. Ganesh\textsuperscript{a}, H. Mehdipour\textsuperscript{b,c}, N.F.F. Nazarudin\textsuperscript{a}, B.T. Goh\textsuperscript{a}, A. Shuhaimi\textsuperscript{a}, S.A. Rahman\textsuperscript{a,*}

\textsuperscript{a}Low Dimensional Materials Research Centre (LDMRC), Department of Physics, Faculty of Science, University of Malaya, 50603 Kuala Lumpur, Malaysia
\textsuperscript{b}Plasma Nanoscience \& Complex Systems, The University of Sydney, New South Wales 2006, Australia
\textsuperscript{c}Department of Physics, Sharif University of Technology, Tehran 11155-9161, Iran

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Amorphous aluminum indium nitride (Al\textsubscript{x}In\textsubscript{1-x}N) thin films were deposited on quartz substrates by plasma-assisted dual source reactive evaporation system. In-rich (x = 0.10 and 0.18) and Al-rich (x = 0.60 and 0.64) films were prepared by simply varying an AC voltage applied to indium wire. The X-ray-diffraction patterns revealed a small broad peak assigned to Al\textsubscript{x}In\textsubscript{1-x}N (002) plane, but no perceivable peaks assigned to crystalline Al\textsubscript{x}In\textsubscript{1-x}N were observed for the films with x = 0.18, 0.60 and 0.64. The morphology of the film was changed from clusters of small grains to uniformly shaped particles with decrease of x. The band gap energy of the films increased from 1.08 eV to 2.50 eV as the Al composition varied from 0.1 to 0.64. Also, Raman results indicated that E\textsubscript{2}(high) and A\textsubscript{1}(LO) peaks of the Al\textsubscript{x}In\textsubscript{1-x}N films are remarkably blue-shifted by increasing x and the A\textsubscript{1}(LO) phonon mode of the Al-rich films exhibits two-mode behavior. A bowing parameter of 4.3 eV was obtained for AlInN films. The extrapolated value from bowing equation was 0.85 eV for band gap energy of InN.

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1. Introduction

The III-nitride ternary semiconductors have become increasingly important in applications such as light-emitting diodes, laser diodes, solar cells, high electron mobility transistors and highly reflective Bragg reflectors in the ultraviolet region [1–4]. Among the III-nitride ternary alloys, Aluminum indium nitride (Al\textsubscript{x}In\textsubscript{1-x}N) has a wide variable band gap of 0.7–6.2 eV [5] depending on the indium (or aluminum) composition in the film structure. This provides great degree of freedom in customizing the electronic structures of the alloy films for specific applications. For example, Al\textsubscript{x}In\textsubscript{1-x}N thin films with tunable band gap of 0.7–2.4 eV can be used for multi-junction solar cell devices since this band gap energy range covers most part of solar spectrum [6].

Crystalline Al\textsubscript{x}In\textsubscript{1-x}N (c-Al\textsubscript{x}In\textsubscript{1-x}N) films have been fabricated over the years by various techniques such as molecular beam epitaxy (MBE) [7–10], metal organic chemical vapor deposition (MOCVD) [11,12], and sputtering-based deposition [13–15]. In general, except for sputtered films, c-Al\textsubscript{x}In\textsubscript{1-x}N films are obtained at high substrate temperatures on specified substrates such as sapphire and GaN. Therefore, these methods are restricted by the type and temperature of substrate. On the other hand, it has been shown that amorphous III-nitride semiconductor films have several advantages such as cost-effectiveness, compositional flexibility, low temperature deposition, no dependency on substrates and greater control of electrical property [16–22]. Shim et al. [20] fabricated blue light emitting a-GaN films at room temperature. Itoh et al. [21] reported low temperature deposition of photo-conductive a-In\textsubscript{1-x}Ga\textsubscript{x}N films and suggested that the films could be utilized as photo-absorptive layer of top cell in multi-junction thin films solar cell. Amorphous In\textsubscript{1-x}Ga\textsubscript{x}N films with considerable photosensitivity were obtained by Suzuki et al. [22] using simultaneous rf magnetron sputtering. Therefore, much investigation should be considered on the properties of amorphous III-nitrides alloys for the aim of effective use of these materials in semiconductor device applications considering the above-mentioned advantages.

Band gap energy (E\textsubscript{g}) of Al\textsubscript{x}In\textsubscript{1-x}N is closely related to deposition technique and the properties of the film. There have been remarkable disagreements among the band gap energies measured for AlInN films using various growth methods. One possible reason is that some of the measurements were carried out before the establishment of 0.7 eV [5] for band gap energy of indium nitride.
(InN). The bowing parameter of ternary AlInN films as well as band gap energies of InN and AlN have been estimated in many reported works using different techniques. The reported experimental values for the AlInN bowing parameter are quite different varying in the wide range of 2.4–6.2 eV [23,24]. This is possibly due to the fact that different values were obtained for $E_g$ of InN films by various growth techniques [7,23]. Terashima et al. [8] reported a bowing parameter of 4.96 eV for AlInN films by RF-MBE using values of 0.70 and 6.14 eV as the optical band gap of InN and AlN, respectively. Wang et al. [12] employed MOCVD method for growth of AlInN films and obtained a bowing parameter of 6 eV considering the band gap energy of InN at below 1.0 eV. Moreover, it has been shown that highly crystalline films have continuous band gap energy with respect to the films composition resulting in smaller bowing parameters [23]. He et al. [15] reported a bowing parameter of 3.68 eV for highly crystalline rf-magnetron-sputtered AlInN thin films. However, the obtained $E_g$ value for InN was 1.34 eV, much larger than its intrinsic value 0.7 eV.

In this work Al$_{1-x}$In$_x$N thin films with high and low Al compositions were deposited by plasma-assisted dual source reactive evaporation technique and the effect of the applied voltage to In source on the films properties was evaluated. The structural, compositional, morphological and optical characteristics of the films will be presented and discussed in the following section.

2. Material and methods

The Al$_{1-x}$In$_x$N films were deposited on quartz substrate using a home-built plasma-assisted dual source reactive evaporation system. A schematic of the system is shown in Fig. 1. Prior to the deposition, the quartz substrate was ultrasonically degreased in de-ionized water, ethanol and acetone and subsequently dried by nitrogen gas. Two separate tungsten wires were coiled and used as the hot filament for evaporation of aluminium and indium wires (with 99.999% purity) and have been labeled $F_{Al}$ and $F_{In}$ in Fig. 1, respectively. The films were clamped perpendicularly to each other between the grounded substrate holder and a radio-frequency (RF) powered stainless steel electrode. In order to activate the surface bonds (prior to the deposition), the substrates were treated in H$_2$-plasma for 10 min at a fixed hydrogen flow rate of 100 sccm. The deposition process was conducted in three steps: (1) $N_2$ plasma was generated in the chamber and stabilized for 5 min, (2) $F_{Al}$ was heated to the temperature required for evaporation of Al atoms (~1650°C) and, then cooled down to 1450 °C to avoid high evaporation rates, (3) Immediately, an AC voltage was applied to $F_{In}$ filament and the Al$_{1-x}$In$_x$N film formation was initiated. Conditions for the film growth were maintained under $N_2$ plasma for 5 min. The applied voltage to $F_{In}$ ($V_{In}$) was set at 75 V, while $F_{Al}$ AC voltage ($V_{Al}$) was varied in 5 V increment from 65 to 80 V in order to obtain Al$_{1-x}$In$_x$N films with different Al(In) compositions. Since the $F_{Al}$ was clamped close to the $F_{In}$ (see Fig. 1), deposition of the evaporated Al species on the $F_{In}$ was unavoidable. Therefore, a voltage higher than the required for In evaporation was applied to the filament in order to generate sufficient amount of In atoms. The quartz substrates were initially heated to 200°C and during the deposition process the final substrate holder temperature was varied from 320 to 360 °C (depending on $V_{In}$) which was monitored by a thermocouple inserted under the substrates. After the deposition process, the $F_{In}$ was switched off, $F_{Al}$ temperature was fixed at 1300 °C and the obtained samples were annealed in $N_2$ plasma (RF power: 50 W) for 30 min. The deposition parameters for films studied in this work were summarized in Table 1.

The structural properties of the grown Al$_{1-x}$In$_x$N films were investigated by X-ray diffraction (SIEMENS D5000 X-ray diffractometer, Cu Kα X-ray radiation $\lambda = 1.54060 \AA$) and Raman spectroscopy (Renishaw inVia Raman Microscope, with a laser excitation wavelength of 514 nm). The compositional uniformity and bonding configuration of the samples were analyzed by X-ray photoelectron spectroscopy (XPS, Shimadzu, Kratos Axis Ultra DLD). Field emission scanning electron microscopy (FESEM, FEI, Quanta 200) was employed to observe surface morphologies of the grown films. The thickness of the films was measured using surface profiler (KLATenstor). Also, the optical transmittance and reflectance for the fabricated Al$_{1-x}$In$_x$N thin films were measured using an UV-vis-infrared spectrophotometer (Lambda 750, PerkinElmer).

3. Results and discussion

The elemental composition was analyzed from XPS spectra of the Al$_{1-x}$In$_x$N films deposited at different applied voltage, $V_{In}$. The In3d$_{3/2}$ core-level photoelectron spectra of the Al$_{1-x}$In$_x$N films prepared at $V_{In} = 65$–80 V are shown in Fig. 2(a–d). A significant increase in the intensity of In3d$_{3/2}$ core-level is observed when $V_{In}$ is increased, implying that higher amounts of In adatoms were incorporated in Al$_{1-x}$In$_x$N film structure at higher voltages. The core-level photoelectron spectra for all samples were deconvoluted into three main sub-peaks centered at 445.3 eV, 444.3 eV and 443.3 eV which are attributed to In–O, In–N and In–In bonds, respectively [25,26]. The corresponding Al2p core-level photoelectron spectra of Al$_{1-x}$In$_x$N films deposited at $V_{In} = 65$–80 V are presented in inset of Fig. 2(a–d). The all spectra were also deconvoluted into three main components at 75 ± 0.2 eV, 74 ± 0.1 eV and 72.6 ± 0.2 eV which are assigned to Al–O, Al–N and Al–Al bonds, respectively [27,28]. The appearance of In–N, In–O, Al–Al and Al–O bonds is possibly due to higher concentration of reactive In and Al atoms reaching the growth sites compared to the reactive N atoms and high affinity of the metallic adatoms to oxygen. The analysis on the chemical composition of the films revealed that the estimated Al mole fractions (Al composition) in the Al$_{1-x}$In$_x$N films, x, are 0.64, 0.60, 0.18 and 0.10 which were determined from the ratio of the integrated intensity of the Al–N and In–N peaks (Al – N/Al – N + In – N) [22] for the films deposited at $V_{In}$ of 65, 70, 75 and 80 V, respectively.

Fig. 3 displays dependence of $V_{In}$ temperature and aluminum incorporation into the films composition on the applied voltage to $F_{In}$ ($V_{In}$). An increase from 1010 ± 10 °C to 1105 ± 10 °C was recorded for $V_{In}$ temperature by increasing the $V_{In}$ from 65 to 80 V which clearly shows that $V_{In}$ controlled the $V_{In}$ temperature. The increase in $V_{In}$ temperature results in enhanced generation and deposition of reactive indium atoms at the substrate surface and, hence, decrease of aluminum mole fraction (x) in the films composition. Guo et al. [29] reported similar tuning capability of the Al composition in Al$_{1-x}$In$_x$N films by varying the applied RF power to indium target using sputtering technique.

Fig. 4 shows the X-ray diffraction (XRD) spectra of Al$_{1-x}$In$_x$N films deposited at different $V_{In}$. The spectra of the films grown at $V_{In} = 65$, 70 and 75 V show no diffraction peaks assigned to crystalline Al$_{1-x}$In$_x$N. The XRD pattern of the films deposited at the highest $V_{In}$ of 80 V shows a small broad peak at 2θ = 31.30° which is very close to the position of (002) plane of wurtzite Al$_{10}$In$_{90}$N based on Vegard’s law:

$$c_{AlIn}_{1-x}N = x c_{AlN} + (1 - x)c_{InN},$$

(1)

where $c_{AlN} = 5.012$ Å and $c_{InN} = 5.793$ Å are the c lattice constant of crystalline AlN and InN, respectively [30,31]. Interestingly, there is a good agreement between the Al composition (x) calculated from XPS analysis in this work and that obtained from Vegard’s law. Phases of In and Al crystalline grains are observed from XRD spectra of the films grown at $V_{In}$ of 65, 70 and 80 V. The presence of these metallic phases was also deduced from the XPS spectra of the films. Basically, the films studied in this work are amorphous in structure with presence of In and Al crystalline grains scattered within the amorphous matrix of the AlInN structure.

Fig. 5(a) shows Raman spectra for Al$_{1-x}$In$_x$N films with different aluminum mole fraction, x. For reference and comparison, the Raman spectrum of the AlN films (grown at $V_{In} = 0$) is presented in Fig. 5(b). Two major peaks centered at 502 and 580 cm$^{-1}$ are observed in Raman spectrum of Al$_{10}$In$_{90}$N films. These peaks are assigned to $E_g$(high) and $A_{1g}$(LO) phonon modes of the In-rich AlN films [32]. The peaks are slightly blue-shifted when the aluminum incorporation is increased to 0.18. With further increase of the aluminum composition to x = 0.60 and 0.64, the shift becomes more pronounced and the $E_g$(high) peaks appear at 609 and 617 cm$^{-1}$, respectively. Considering the $E_g$(high) phonon mode of the grown AlN films which is centered at 685 [33] (see Fig. 5(b)), it can be seen that the $E_g$(high) phonon mode of the Al$_{10}$In$_{90}$N and Al$_{64}$In$_{36}$N thin films are red-shifted by 56 and 48 cm$^{-1}$, respectively.
Uniquely, the Raman spectra of Al$_{0.60}$In$_{0.40}$N and Al$_{0.64}$In$_{0.36}$N films reveal one hitherto unknown Raman feature located at around 500 cm$^{-1}$ (labeled as $\nu$) which was detected by repeated micro-Raman spectroscopies at various test points. Because it was also observed in Raman spectrum of AlN samples (Fig. 5(b)), it cannot be assigned to any E$_2$ (high) mode. This peak does not seem to correspond to the allowed Raman modes of hexagonal AlN based on group theory and may be related to acoustic phonon modes of AlN [34]. A broad peak at around 800 cm$^{-1}$ was detected from Raman spectra of the AlInN films with Al compositions of $x=0.60$ and 0.64 which may be formed from overlapping of some Raman structures. In order to identify each of the contributions, we have analyzed the Raman spectrum of Al$_{0.64}$In$_{0.36}$N films in 670–900 cm$^{-1}$ range using curve fitting method (Fig. 5(c)). The observed broad peak at around 800 cm$^{-1}$ was deconvoluted into two main components at around 740 and 820 cm$^{-1}$ which are assigned to InN-like A$_1$(LO) and AlN-like A$_1$(LO) phonon modes of AlInN, respectively [35,36] as shown in Fig. 5(c). This is in good agreement with Raman results reported by Kang et al. [35] and Jiang et al. [36] who showed that A$_1$(LO) phonon mode of Al$_x$In$_{1-x}$N films ($x \geq 0.30$) exhibits two-mode behavior.

Fig. 6 displays the FESEM images of the surface of AlInN films with different composition. It is obviously seen that the surface morphology of Al$_x$In$_{1-x}$N films strongly depends on Al composition of the films, $x$. The surface morphology of Al$_{0.64}$In$_{0.36}$N thin film (Fig. 6(a)) shows a smooth and compact microstructure of nanoparticles with an average size of 50 nm. Agglomeration of these nanoparticles on the surface results in formation of grains of larger dimension $\sim$100–300 nm. A decrease in the Al composition $x$ to 0.60, enhances the agglomeration of the nanoparticles and a few excessively large grains with the size of $\sim$500–800 nm are formed as a result (see Fig. 6(b)). The growth of these grains perpendicular to the surface forms a rough surface. Distribution of pebble-like particles is observed in the FESEM image of the more Al-diluted Al$_{0.18}$In$_{0.82}$N films (Fig. 6(c)). However, some relatively large grains

![Schematic of the experimental setup of plasma-assisted dual source reactive evaporation system.](image)

Table 1: Plasma-assisted dual source reactive evaporation conditions used for preparing Al$_x$In$_{1-x}$N thin films.

<table>
<thead>
<tr>
<th>Deposition parameters</th>
<th>Values</th>
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<tbody>
<tr>
<td>N$_2$ flow rate (sccm)</td>
<td>40</td>
</tr>
<tr>
<td>RF power (W)</td>
<td>250</td>
</tr>
<tr>
<td>F$_{Al}$-substrate distance (mm)</td>
<td>10</td>
</tr>
<tr>
<td>F$_{In}$-substrate distance (mm)</td>
<td>15</td>
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<tr>
<td>Substrate holder temperature (°C)</td>
<td>320–360</td>
</tr>
<tr>
<td>Al temperature (°C)</td>
<td>1450</td>
</tr>
<tr>
<td>In temperature (°C)</td>
<td>1000–1100</td>
</tr>
<tr>
<td>Process time (min)</td>
<td>5</td>
</tr>
</tbody>
</table>
are also seen from the morphology. The surface morphology of Al$_x$In$_{1-x}$/C$_0$N with Al composition of $x = 0.10$ (Fig. 6(d)) exhibits features of near-spherical-shaped nanoparticles distributed uniformly over the entire surface. This kind of morphology is favorable in solar cell application as the spherical particles are good channels for charge carrier transport\cite{15}.

The optical properties of Al$_x$In$_{1-x}$/C$_0$N films with different compositions were studied by obtaining transmittance and reflectance spectra of the films. Fig. 7(a) displays the transmittance curves of Al$_x$In$_{1-x}$/C$_0$N films with Al composition of $x = 0.10$, $0.18$, $0.60$ and $0.64$. In-rich films ($x = 0.10$ and $0.18$) show an absorption in near-infrared (NIR) region and an insignificant transmittance (e.g., $T \sim 0\%$) in the range of 300–1000 nm. As $x$ is increased toward Al-rich region, the absorption edge of the films shifts toward shorter wavelength and Al$_x$In$_{1-x}$/C$_0$N films with higher Al content become more transparent in visible and near infrared (NIR) regions. To further clarify the optical results, we have measured transmittance of the AlN film (see inset in Fig. 7(a)). Absorption edge of the sample occurred at around 200 nm which is in good agreement with reported absorption edge wavelength of AlN films\cite{37–40}. Fig. 7(b) depicts the wavelength dependence of optical reflectance spectra of the AlInN films with different aluminum compositions $x$. It can be clearly observed that the minimum of the reflectance is shifted toward longer wavelength when the aluminum incorporation ($x$) is reduced from 0.64 to 0.10. Since

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Fig. 2. In3d$_{5/2}$ core-level photoelectron spectra of Al$_x$In$_{1-x}$/C$_0$N films deposited at $V_{In} = 65$ (a), $70$ (b), $75$ (c) and $80$ V (d). The inset of (a–d) are the corresponding Al2p core-level photoelectron spectra.

Fig. 3. Dependence of Al composition in the Al$_x$In$_{1-x}$/C$_0$N films and $F_{In}$ temperature on the applied AC voltage to indium wire.

Fig. 4. XRD pattern of the Al$_x$In$_{1-x}$/C$_0$N thin films grown at different $V_{In}$. 
and $\theta$ are proportional constant and band gap energy, respectively [24]. Direct band gap of an AlIn$_{1-x}$N film is determined by extrapolating the liner part of $(z/h)^2$ versus $h$ curves down to the energy axis. This method has been employed to measure band gap energy of amorphous semiconductors [41]. As seen in Fig. 7, the optical band gap of the AlIn$_{1-x}$N film changes with the variation of the Al composition within the films structure. The optical band gap of grown Al$_{0.44}$In$_{0.56}$N thin film is estimated to be 2.50 eV. As $x$ decreases to 0.60, 0.18 and 0.10 the band gap energies of the films are obtained at 2.33, 1.42 and 1.08 eV, respectively. Such controlled tuning of the band gap energy is very crucial for materializing the application of AlInN alloys in multi-junction solar technology. The $E_g$ value of pure AlN shown in the inset of Fig. 8 is estimated to be 5.5 eV, which is smaller than the value 6.2 eV reported in the previous works [42]. It has been shown that the band gap of an AlN film can be reduced by several factors such as high carrier concentration, nonstoichiometric composition, and impose of residual stresses as well as presence of structural defects and oxygen impurity in the film structure [43,44]. The AlN band gap energy obtained in this work is very close to the values reported in other similar works [37,39,45]. It is also much larger than the $E_g$ (of AlN films) reported by Cho et al. [46] and Wu et al. [47] who prepared the AlN films by reactive magnetron sputtering and laser molecular beam epitaxy methods, respectively.

For optoelectronic applications envisaged for Al$_{1-x}$In$_x$N film, it is essential to estimate the band gap energy based on the film composition $x$. Fig. 9 shows dependence of $E_g$ values of our Al$_{1-x}$In$_x$N films on the aluminum composition (dotted line). Obviously, the relationship between the obtained band gap values and Al composition is not linear. This phenomenon is named as “bowing effect” which is due to unstable nature of Al$_{1-x}$In$_x$N within the miscibility gap, charge exchange between forming bonds in disordered solid solution and relaxation of bond length [48]. The bandgap energy of Al$_{1-x}$In$_x$N ($E_{gAlIn}$) as a function of Al composition $(x)$ is determined using the following equation [10]:

$$E_{gAlIn} = xE_{gAlN} + (1 - x)E_{gInN} - bx(1 - x),$$  

where $b$ is bowing parameter and $E_{gAlN}$ and $E_{gInN}$ are the band gaps of AlN and InN films, respectively. Fitting Eq. (4) to the experimental data obtained in this work, gives the bowing parameter $b$ of 4.3 eV, which is close to 4.1 and 4.0 eV reported by Goldhahn et al. [49] and Yeh et al. [50] respectively, and is much smaller than 6.0 eV obtained by of Wang et al. [12]. By extrapolation the band gap energy curve to the $x = 0$, the band gap of 0.85 eV is obtained for the InN film, which is close to the reported value (0.7 eV) [5]. However, this extrapolation-led estimation of the InN band gap has to be confirmed experimentally by growing a pure InN using this technique.

4. Conclusion

Amorphous In-rich ($x = 0.10$ and 0.18) and Al-rich ($x = 0.60$ and 0.64) Al$_{1-x}$In$_x$N thin films were successfully deposited using plasma-assisted dual source reactive evaporation method. It was shown that the Al composition in the films can be adjusted by simply varying the voltage applied to the indium wire ($V_{In}$). Raman results indicated that $E_g$(high) and $A_1$(LO) peaks of the Al$_{1-x}$In$_x$N films are considerably blue-shifted by increasing $x$ and the $A_1$(LO) phonon mode of the Al-rich films exhibits two-mode behavior. The morphology of the film was changed from clusters of small grains to uniformly shaped particles with decrease of $x$. Optical characterizations showed that the band gap energy of the

minimum value of the reflection occurs in the vicinity of the wavelength related to the optical bandgap energy, the results in Fig. 7(b) show that the bandgap value of the AlN films increases by increasing $x$. A insignificant reflectance (e. g., $R \sim 3\%$) at around 200 nm is obtained from optical reflectance spectrum of AlN films (see the inset of Fig. 7(b)) which is in good agreement with the corresponding transmittance result shown in the inset of Fig. 7(a).

The absorption coefficient ($\alpha$) was also calculated for each film with the measured transmittance $T$, reflection $R$ and film thickness $d$ from equation [23]:

$$\alpha = 1/d\ln[(1 - R)^2/T].$$  

In order to obtain direct band gap of the AlIn$_{1-x}$N films, we have plotted $(z/h)^2$ as a function of photon energy $(h\nu)$ (Fig. 8). It is known that the squared absorption coefficient of a semiconductor with a direct band gap is a function of photon energy:

$$x^2 = A(h\nu - E_g)^2,$$  

where $A$ and $E_g$ are proportional constant and band gap energy, respectively [24].
Fig. 6. FESEM images of the Al$_x$In$_{1-x}$N films with Al compositions of $x = 0.64$ (a), 0.60 (b), 0.18 (c) and 0.10 (d).

Fig. 7. Transmittance (a) and reflectance spectra (b) of Al$_x$In$_{1-x}$N films with different Al compositions. The inset of (a and b) are transmittance and reflectance spectra of AlN ($x = 1$), respectively.

Fig. 8. Plots of $(\alpha h \nu)^2$ versus $h \nu$ for Al$_x$In$_{1-x}$N films with different $x$. The inset is the plot of $(\alpha h \nu)^2$ versus $h \nu$ for AlN films.

Fig. 9. Band gap energy of Al$_x$In$_{1-x}$N film as a function of Al composition $x$. 
films increased from 1.08 eV to 2.50 eV as the Al composition varied from 0.10 to 0.64. A bowing parameter of 4.3 eV was calculated for band gap energy of polycrystalline AlInN, \( N \) thin film for specific applications envisaged for such materials.

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