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Effect of DC power on the AlN plasma of magnetron sputtered thin film

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Abstract: AlN thin films deposited at room temperature by reactive DC magnetron sputtering of Al target in pure argon and nitrogen atmosphere. Thin films were deposited on glass and silicon (Si) substrate at different DC power. The variation of DC power on the structural, electrical and optical properties of the films were investigated. Enhancement of crystallinity and conductivity was observed with increase in DC power. The optical characteristics of films, such as refractive index, extinction coefficient, and average thickness, were calculated by Swanepoel's method using transmittance measurements. The refractive index and average roughness value of the films increased with film thickness. Film deposited on Si substrates at an DC power of 100W showed a minimum resistivity of $1.27 \times 10^{-3} \Omega \text{cm}$. The electrochemical behavior of the films was studied in 3.5% NaCl solutions using potentiodynamic techniques. The results indicate that AlN films grown at a higher power performed with a better electrochemical behavior compared to the lower deposition power. It has been observed that the film properties are greatly influenced by the plasma conditions during sputtering. DC plasma was studied using Langmuir Probe and Optical Emission Spectroscopy (OES). The plasma parameters such as ion density and electron temperature were determined and their dependence on properties of thin film deposited under similar plasma conditions were studied. Plasma parameters were determined for different DC powers keeping the constant distance.

Key words: Aluminum nitride, Thin film, Magnetron sputtering, Langmuir probe

I. Introduction

Aluminum nitride (AlN) is a wide band-gap (6.2 eV) [1-3] III-V compound, which has attracted attention of the scientific community due to its exotic properties. Thin film of AlN with a hexagonal wurtzite crystalline structure is used in various applications like optical hard coatings, wear resistant, high temperature microelectronics, chemical and thermal stability [4-7]. Its high thermal conductivity ($180 \text{ W m}^{-1} \text{ K}^{-1}$) [8, 9], excellent piezoelectricity [10], high electrical resistivity ($\rho = 10^9 - 10^{11} \Omega \text{m}$) [11], and fast acoustic velocity [12] make AlN promising material use in optoelectronic, surface acoustic wave devices, sensor and thin film resonators [13,14]. Thermal conductive AlN films with moderate dielectric constant are considered for metal-oxide-semiconductor (MOS) applications [15]. AlN films are used for optical applications in corrosive and high temperature environments [16, 17]. A few studies also cover its electrochemical behaviour [18-21]. Quality AlN thin film requires high purity source and oxygen free environment for deposition because of the aluminium reactivity. Traditionally, AlN films are deposited by various techniques, preferably chemical vapour deposition (CVD) or molecular beam epitaxy (MBE) [22-25], pulsed laser deposition (PLD) [26-28] and electron shower method [29]. Recently, many reports on low temperature deposition of good quality AlN films by physical vapour deposition (PVD) techniques are available. In the PVD process DC reactive magnetron sputtering is the most common deposition process. Among these, sputtering has advantages over other conventionally used high temperature techniques because of its simplicity, low thermal temperature, low cost and its ability to produce good quality films with desired properties [30, 31]. Characteristic features of the prepared films depend on the processing parameters and method and are easier to control in PVD process.

In this work the AlN films are deposited on Si, steel and glass substrates by the DC reactive magnetron sputtering. To achieve the desired property on the effects of power on the phase, structure, and film formation on the substrates. All the deposition is carried out keeping constant gas ratio, deposition temperature and the base pressure. The thin films are characterized using X-ray diffraction (XRD), Atomic Force Microscopy (AFM), spectroscopic Ellipsometer. Optical properties of the films are analysed using UV-Visible spectrometer and Fourier Transform Infrared (FTIR) Spectroscopy. The optical properties of thin films have been investigated by various analytical and numerical methods [32-35]. In these methods, transmittance and reflectance data have been used to calculate thin film optical constants, such as refractive index $n(\lambda)$, extinction coefficient $k(\lambda)$, and film thickness d . Plasma physicists use Langmuir probe and Optical emission spectroscopy (OES), for obtaining the information about plasma. The properties of AlN thin films can be optimized by carefully selecting the process parameters during deposition. In the case of sputtered films, DC power is one of the process parameters that can be controlled during the film growth. This chapter presents the dependence of DC power on the AlN film properties.

II. Experimental details

A. Deposition

AlN thin films were deposited by DC magnetron Sputtering deposition system which consists of a vacuum chamber, cathode (connected to 1kV DC power supply), mass flow controller, sample holder and view port. Pure Al target (50 mm diameter, 5 mm thick, 99.99% pure) was mechanically clamped to the magnetron cathode of the sputtering system.

Silicon, stainless steel (SS) and glass substrates of size 10 X 10 mm² and thickness ~ 3mm were ultrasonically cleaned and dried at room temperature prior to deposition. Substrates were mounted on the sample holder and a base pressure of ~10⁻⁶ mbar was obtained. The target surface was sputter etched by Ar at 100 W for 20 min to avoid contamination before deposition. After sputter cleaning, Ar: N₂ gas flow rate was adjusted to 2: 3 ratios and working pressure of ~ 2 x 10⁻² mbar was maintained during deposition. In this way, films were obtained at four different DC power (100, 200, 300 and 400 W). The substrate temperature was monitored using a thermocouple. Film growth conditions are summarized in Table 1. It was found that the physical appearance of the deposited films is good.

B. Plasma diagnostics

Langmuir probe is one of the simplest techniques for obtaining information about the ions in plasma. The Langmuir probe assembly consists of a tungsten wire, 0.5 mm in diameter and 5 mm in length, supported by a glass sleeve (Fig. 1). The probe current -voltage (I- V) characteristics are plotted for different DC powers (50W to 200W). Probe current is measured for bias voltages in the range of -45 to +45 V. Great care was taken to prevent the probe feed wires being exposed to the plasma since this will contribute to the measured probe current. To ensure a clean probe surface, the probe wire was replaced frequently.

To investigate the ionic species present in the DC plasma in detail optical emission spectra (OES) of plasma plume generated during the DC sputtering of AlN target was recorded using a 0.32m monochromator and charge coupled device (CCD) detector. OES examines the light given off by species in the plasma that have been raised to excited states by electron impact. Excited species radiate at wavelengths corresponding to the quantum mechanically allowed transitions between electron energy levels. It is an attractive option for process control because it is fast, non intrusive, can monitor multiple elements simultaneously and provides information about deposition conditions in the plasma [36]. In the present investigation, the OES studies were made through the side window of the chamber (Fig. 1).

III. Results & Discussions

Thin film depends on the kinetics of the arrival species at the substrate and is directly dependent on the deposition parameters. The deposition parameters are summarized in Table 1. Furthermore, it is grain formation onto initial layers that controls structural and optical properties of the film. The AlN films are deposited by varying the DC power and keeping other parameters constant. Fig. 2 shows the dependence of film thickness of AlN on DC power. The film thickness increases with increasing the DC power. The current on the cathode increased as we increase the DC power supply, supplying more electrons into plasma for ionization; so that more Ar ions generated in the plasma strike the target surface with a higher energy, resulting in effectively increased amount of target materials ejected from the target surface and deposited on the substrate [37]. For this reason the increase in thickness with DC power supply is observed. The XRD pattern of AlN thin films deposited at different DC power 100, 200, 300 and 400 W shown in Fig. 3. The Crystallinity of the deposited films was confirmed using the JCPDS data tables. When the power was 100 to 300 W, diffraction peaks observed at $2\theta = 33.31^\circ$ correspond to (100) AlN (JCPDS card No. 79-2497). In Fig. 3(d), peak observed at $2\theta = 82.32^\circ$ correspond to Al (222) phase (JCPDS card No. 85-1327). When the power was increased to 400 W, the Al phase appeared in the XRD spectra, because part of the Al atoms sputtered from the target surface were not nitride and were deposited directly on the substrate.

The surface topography of AlN thin films is characterized by AFM. Fig. 4 shows two and three dimensional AFM image of 150 nm×150 nm deposited at 100W. Smooth and uniform AlN film surface is found at this growth condition. Fig. 5 shows the root mean square (RMS) surface roughness derived from the corresponding AFM images. The surface roughness of the AlN films increases with the increase of the DC power. Kajikawa et al. studied the correlation between process conditions and film structure, and suggested that the morphology of reactive-sputtered nitride films can be affected at both initial and growth stages.

In the initial stage, four processes occur, which include (I) nucleation due to epitaxial relationship or due to surface and interfacial energy anisotropy, (II) coarsening due to surface and interfacial energy anisotropy, (III) grain growth, and (IV) resputtering due to ion bombardment. In the growth stage, three processes occur at the surface, including (I) precursor sticking, (II) surface diffusion of adatoms, and (III) resputtering due to ion bombardment [38]. Since nitride film formation during reactive sputtering occur at the substrate and deposited surface, Increase in the plasma power may have resulted in increasing the number of ejected Al species from the target, which results in the increase of film thickness with the increasing DC power as shown in Fig. 2.

On the other hand, the incident energy of the high energetic ions will contribute to the surface migration and surface reaction to form a flat surface, which leads to the increase of the RMS surface roughness with increasing the DC power as observed in Fig. 5.

Fig. 6 shows the wavelength dependence of optical transmittance of the AlN films grown at different DC powers on the glass substrate. The transmittances of different samples in the wavelength range 200-800 nm. We can observe that by increasing the power value, the film transparency decreases. Most probably, the thicker film is denser and more defective, resulting in a decrease in film transparency. The films exhibited around 80 - 85% transmittance in the visible and ultraviolet range and steep absorption edges at approximately 250 nm. Hence the film deposited at 100 W is the most transparent compare to other films. A sharp drop is approximate 200 nm is observed. The refractive index and thickness of the deposited films have been determined from their normal incidence transmission spectra by Swanepoel's method. [33] Fig.7 shows both, the calculated and simulated refractive indices n for samples with different thickness.

It is obvious that the experimental results are in good agreement with the results obtained using the Cauchy dispersion relation. The Cauchy dispersion relations for 100 W and 200 W are

$$n(100) = 1.56 + \frac{1.3 \times 10^{-4}}{\lambda^2},$$

$$n(200) = 1.72 + \frac{1.6 \times 10^{-4}}{\lambda^2}.$$

In Fig. 8 the variation in refractive index as a function of wavelength for samples 100 W and 200W.

We observed that the refractive index initially decreases with an increase in wavelength and finally becomes constant at a higher wavelength. We found that the refractive index of 100 W samples in the range of 1.67 to 1.74 and for 200 W the range is 1.79 to 2.12 with an accuracy of 2-3% estimated using eqs. (6) and (7). We can observe that the refractive index increases with the increase of the film thickness. Although an increase in surface roughness with thickness can scatter the incident light more and can contribute to the decrease in transparency and increase in refractive index. For 100 W samples at a wavelength of 550 nm the film refractive index n was found to be 1.64. The extinction coefficient for these films in the visible range is $k = 5 \times 10^{-3}$ at $\lambda = 300$ nm. The film thickness obtained by Swanepoel's method is 271 ± 9 nm. The Al-N bond information obtained using the FTIR spectroscopy at room temperature. The spectra are recorded in the transmission mode for all films as shown in Fig.8. It is observed that there are two peaks 754 and 883 cm^{-1} . These two peaks are related to the Al-N bond, displays the strong absorption band which confirms the existence of nanoparticle [39, 40]. The range of high IR transmission was found to be between $500\text{--}4000 \text{ cm}^{-1}$.

The film surface resistivity was measured using standard four-point probe method. The resistivity was calculated using Van der Pauw method. According to which the resistivity is given by, $\rho = (k \times V \times t) / I$, where $k = CF1 \times CF2$; CF1 and CF2 are the two geometric correction factors, V/I is the slope of plot obtained by four point probe measurements and t is the film thickness. In our study, the applied correction factor (k) is taken as 0.95 [41]. The resistance values of the films were taken at different places on the surface of the film. Measurements of electrical resistivity and conductivity are shown in Table 2. For the films deposited on Si substrates, the highest resistivity value corresponds to 400 W and the lowest value corresponds to 100 W. On the other hand, resistivity values of 300 and 400 W deposited film gave near about same values of $\sim 10^8 \Omega \text{ cm}$.

From literature, it has been reported that electrical resistivity of AlN varies from $> 10^7 \Omega \text{ cm}$ up to $\geq 10^{14} \Omega \text{ cm}$, [41-43] both for thin films or undoped single crystals, this range of resistivities being typical of insulating materials. For our samples, the obtained results of electrical resistivity values fall within the reported values for AlN. As increase the electrical conductivity with decrease the DC power.

The electrolyte was 3.0% NaCl solution open to air at room temperature. Prior to the beginning of polarization, the samples were kept in the solution for 15 min in order to establish free stationary potential. The specimens were previously cleaned and fixed in a Teflon holder. The surface area contacting the electrode was 1 cm^2 .

Potentiodynamic polarization curves of the substrate and each film are shown in Fig. 9 and the corrosion parameters in Table 3. The corrosion current density and the corrosion potential were obtained by the intersection of the extrapolation of anodic and cathodic Tafel curves. The corrosion current densities of the films were much lower than that of the stainless steel substrate and the 400 W deposited AlN films showed the lowest corrosion current density, indicating the lowest corrosion rate. The 200W deposited AlN film showed better corrosion resistance than the 100W deposited AlN film on the basis of corrosion current density and polarization resistance. From polarization test results, the protective efficiency, P_i (%) of the films can be calculated by Eq. (1):

$$P_i(\%) = [1 - (i_{\text{corr}}/i_{\text{corr}}^0)] \times 100 \quad (1)$$

Where i_{corr} and i_{corr}^0 indicate the corrosion current density of the film and substrate, respectively [44]. The protective ability of the film increased with the increase the DC power. The 400W deposited AlN film showed the highest protective efficiency of 88.3% caused the film thickness is more than other films and the lowest corrosion current density of $12.43 \mu\text{A}/\text{cm}^2$.

A. Langmuir probe measurement

A typical Langmuir probe I-V characteristic of the DC plasma is shown in Fig. 10. The floating potential (V_f) of the plasma is the probe bias for which the ion current equals electron current. This was estimated from the intersection of the characteristic with the voltage axis. When the potential applied to the probe is greater than the plasma potential (V_p), the electrons from the plasma are attracted to the probe and the ions repelled. The V_p is marked by the change in the slope of the I-V characteristic. The electron temperature (kT_e), ion/electron density (N_e) plasma potential V_p and floating potential V_f , are calculated. The portion of the I-V characteristic below -10 V was used in the calculation of ion density in order to eliminate the error in the probe current due to the contribution of highly energetic electrons coming on to the probe. Probe current is measured for bias voltages in the range of -40 to +45 V (Fig. 10). Great care was taken to prevent the probe feed wires from being exposed to the plasma since this will contribute to the measured probe current. A typical I-V curve consists of 3 regions: namely the positive ion current region, transition region and the electron saturation region (in Fig. 10). These regions are exploited separately to obtain the plasma parameters. From the characteristics we can determine the plasma potential (V_p) and floating potential (V_f). The difference between plasma potential and floating potential ($V_p - V_f$) gives a measure of energy of the sputtered particles bombarding the substrate. $V_p - V_f$ is plotted in the Fig. 11 as a function of DC power. In the present investigation $V_p - V_f$ shows slight decreases with increasing DC power.

1. Ion Density

The saturation ion portion of the characteristics is used to determine the ion density [26 45]. The ion current drawn by the probe is given by the equation

$$I_i = \frac{Ae^3 N}{2\sqrt{\pi}} \left(\frac{3T_e}{m_i} \right)^{\frac{1}{2}} \left(1 - \frac{V - V_f}{T_e} \right)^{\frac{1}{2}}$$

Where A is the surface area of the probe, N is the plasma density, m_i is the ion mass and T_e is the electron temperature in electron volts (eV). Taking the derivative of with respect to V and rearranging we get

$$N_i^2 = \frac{4\pi m_i}{3A^2 e^3} \left(\frac{\partial I_i^2}{\partial V} \right)$$

Where n_i is the ion density, m_i is the ion mass and A is the collection area of probe which is given by

$$A = 2\pi r l + \pi r^2$$

Where r is the radius of the probe (100 μm) and l is the probe length (5 mm). Ion mass was taken as $m_i = 40m_p$ (where the m_p is the mass of the proton ($1.667 \times 10^{-27} \text{Kg}$)), on the assumption that the major contribution of ions are given by Argon. This equation is based on the fact that n_i is equal to electron density n_e because plasma is neutral.

Fig. 11 shows the dependence of ion density on DC power. Ion density is found to increase linearly with increase in DC power. The increase in ion density is due to the greater ionization resulting from collisions that may occur at higher DC power.

2. Electron temperature

The state of plasma is essentially specified if the composition of the plasma is known and if the energy of each constituent can be determined. Electron temperature can be determined from the I-V characteristics [45]. In I-V characteristics graph, plot of natural logarithm of current ($\ln I$) versus probe voltage in the region between floating potential and plasma potential. The graph will be a straight line. The reciprocal of the slope of the straight line plot will give the value of kT_e . In the present investigation, electron temperature decreased with increase in DC power (Fig. 12). the drop in electron temperature and $V_p - V_f$ is due to the increase in ion density [46].

The electron velocity and ion velocity [47] were calculated using the equations,

$$V_e = \left(\frac{8kT_e}{\pi m_e} \right)^{\frac{1}{2}}$$

$$V_i = \left(\frac{8kT_e}{\pi m_i} \right)^{\frac{1}{2}}$$

Where k is the Boltzmann constant, T_e is the electron temperature, m_e is the mass of electron and m_i is the ion mass.

Ion velocity is of the order of 10^3 m/s and electron velocity is of the order of 10^5 m/s. Both ion velocity and electron velocity decreased with increase in DC power (Fig. 13). The variation of velocity with DC power showed a similar behavior as that of electron temperature.

B. Optical Emission Spectral Studies

Optical emission spectroscopic (OES) studies were carried out on the plasma generated at various RF powers in the argon ambient gas pressure. The spatial variations of emission intensities of different species in the plume were investigated as a function of DC power. Figure 15 shows typical OES spectra. Several strong emission lines of argon neutral (ArI) were found in the wavelength range 600 nm to 750 nm. A few emission lines of aluminum neutral were found but its intensity is very low. However, the NH band (~336 nm) with Ar(I) lines (> 700 nm) were observed with lateral-viewing of the plasma using the CCD, as shown in Fig. 15. The NH bands presumably resulted from the entrainment of residual gas in the chamber [48].

All the prominent lines in Fig. 15 are those of argon neutral. Nitrogen emission lines in its atomic or ionic states have not been detected in this investigation. Also the emission line corresponding to Al II (492.40 nm, 4d 205i2 - 4f 2Fm) was recorded with very weak intensity.

IV. Conclusions

AlN thin films are grown on Si and glass substrates by DC magnetron sputtering in plasma containing a mixture of argon and nitrogen with different power. AFM results show that the grown films are smooth and dense. FTIR spectroscopy reveals that the formation of Al-N bond in the films and XRD patterns confirm that a preferred orientation of (100). The film optical properties have been investigated by Swanepoel's method.

The effect of DC power is investigated with respect to film thickness, surface roughness, and transmittance of AlN films. With increase in DC power, growth rate and the RMS surface roughness increases. AlN thin film deposited at 100 W have low surface roughness and high transparency in the visible spectra. The experimental values of refractive index are in good agreement with those obtained using the two-term Cauchy dispersion relation. Also, the optical results confirm the dependence of optical properties on the film thickness. The average transmission in the visible range is greater than 80%. The film thickness increases with increase in DC power. A thicker film shows a higher refractive index. From the results, it is concluded that AlN thin films deposited at low DC power is a good candidate for optical device applications. The 400W deposited AlN film showed the lowest current density and the highest protective efficiency in the potentiodynamic test results.

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Table 1: Deposition Parameters for AlN (D.C. reactive Sputtering)

Objects	Specification
Target	Al pure (99.99%)
Substrate	Si wafer, stainless steel (SS), glass
Target to substrate distance	6.5cm
DC power	100, 200, 300 and 400 W
Substrate temperature	Room- Temperature
Sputtering time	60 minute

Table 2: Electrical resistivity- conductivity of DC sputtered AlN films

DC Power (W)	Resistivity ($\Omega\text{-cm}$)	Conductivity ($\Omega\text{-cm}^{-1}$)
100	8.1×10^4	1.2×10^{-5}
200	3.8×10^6	2.6×10^{-7}
300	2.3×10^8	1.9×10^{-9}
400	8.6×10^8	4.2×10^{-10}

Table 3 Results of potentiodynamic polarization tests

Sample	E_{corr} (mV)	I_{corr} (mA/cm ²)	Protective efficiency % P_i
Uncoated SS	-180.7	103.5	-
100W	-203.5	56.64	45.7
200W	-214.6	37.18	64.1
400W	-230.3	12.43	88.3

Figure captions

- Figure 1. Schematic Diagram of DC Magnetron Sputtering System.
 Figure 2. Film thickness of AlN films on DC power.
 Figure 3. GIXRD diffractograms of deposited films prepared at different DC power; (a) 100W, (b) 200W, (c) 300W, and (d) 400W.
 Figure 4. (a) 2-D and (b) 3-D AFM image of topography of AlN thin films deposited at 100 W.
 Figure 5. Variation of the AlN surface roughness as a function of the DC power.
 Figure 6. FTIR transmission spectra of AlN thin films deposited at 100 W.
 Figure 7. Variation of refractive index (n) versus wavelength (λ) for AlN films at 100 and 200 W.
 Figure 8. Optical transmittance spectra of AlN grown under various DC power.

Figure 9. Potentiodynamic polarization curves.

Figure 10. Langmuir probe I-V characteristic for 200W DC power.

Figure 11: Variation of Plasma potential (V_p) and ($V_p - V_f$) with DC power.

Figure 12. Variation of ion density as a function of RF power.

Figure 13. Variation of electron temperature with DC power.

Figure 14. Variation of electron and ion velocity with DC power.

Figure 15. Optical emission spectrum of DC plasma generated with Al target at DC power of 100 W.

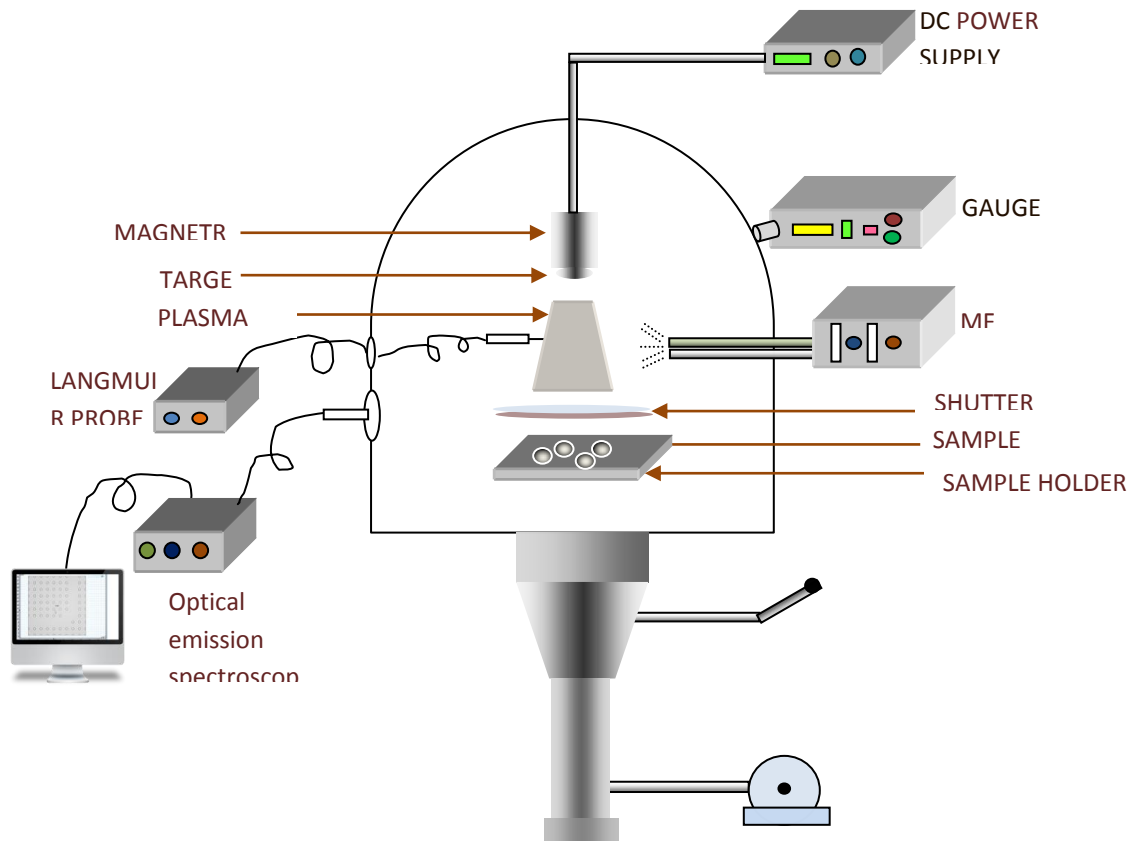


Figure 1

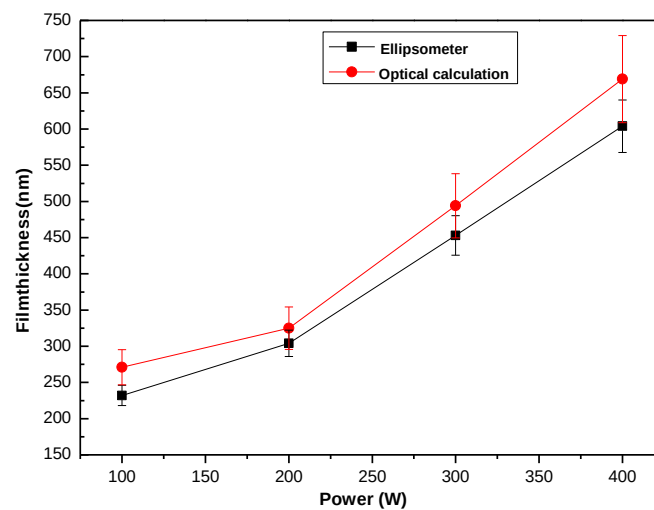


Figure 2

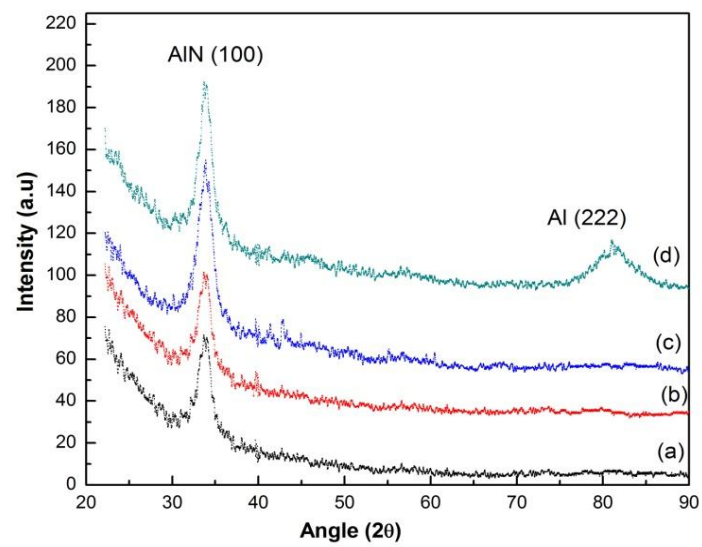
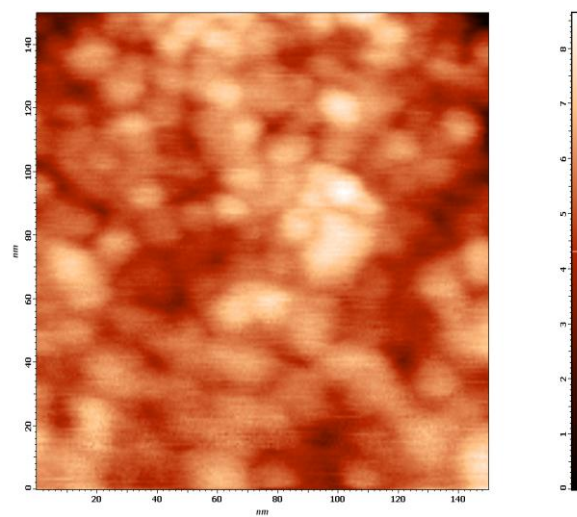
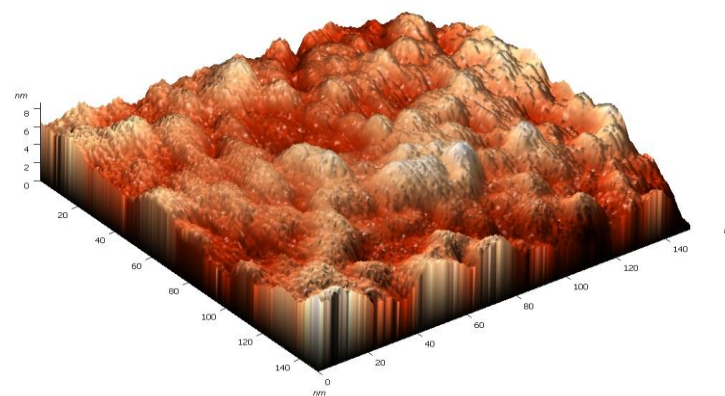


Figure 3



(a) 2d image



(a) 3d image

Figure 4

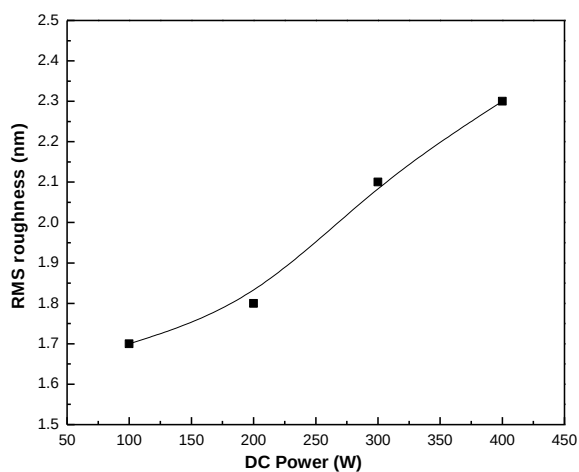


Figure 5

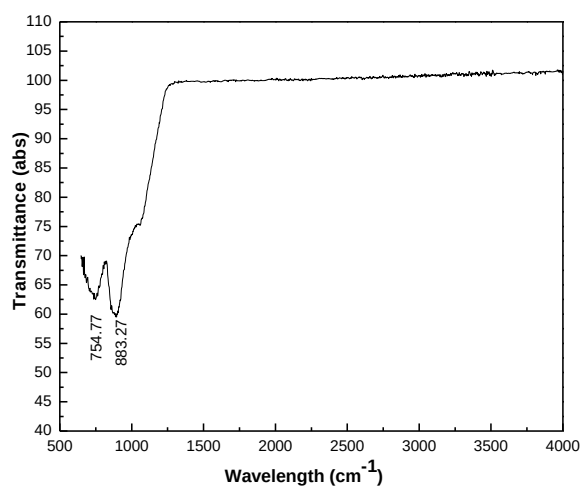


Figure 6

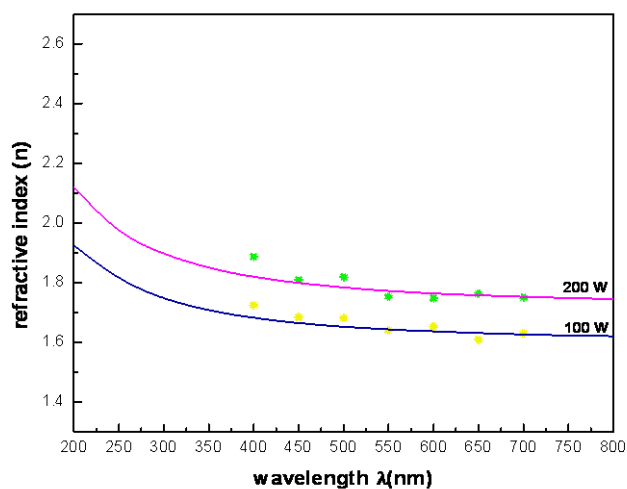


Figure 7

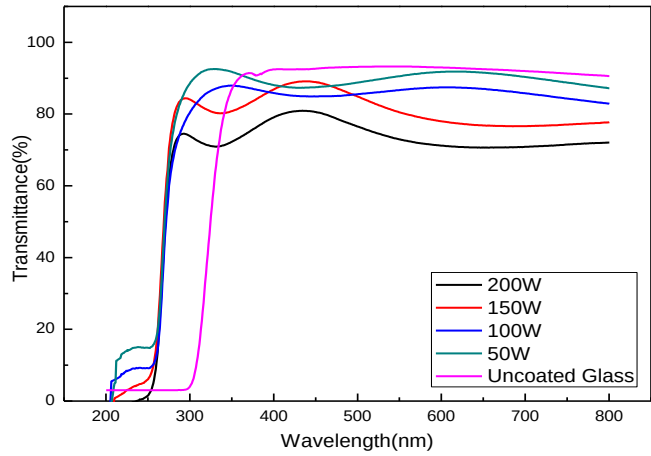


Figure 8

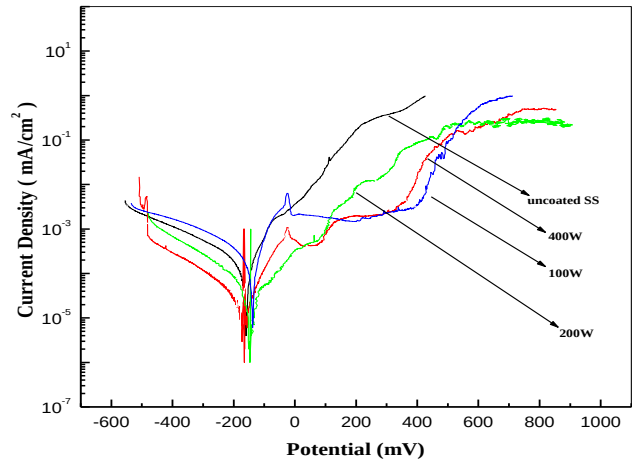


Figure 9

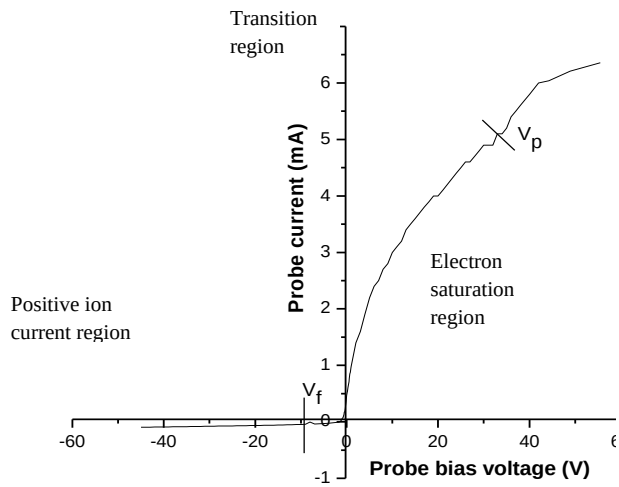


Figure 10

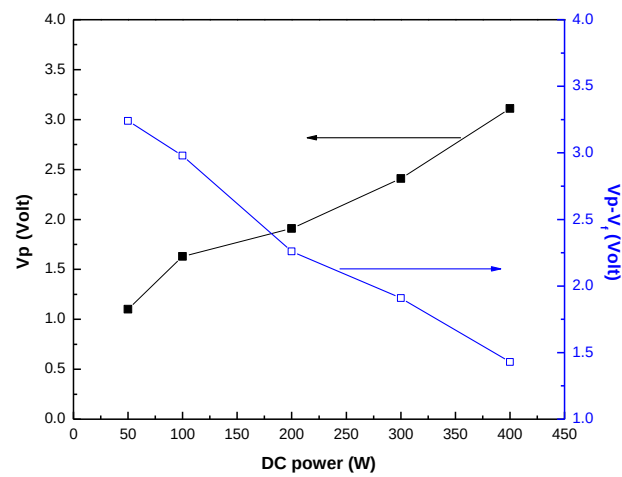


Figure 11

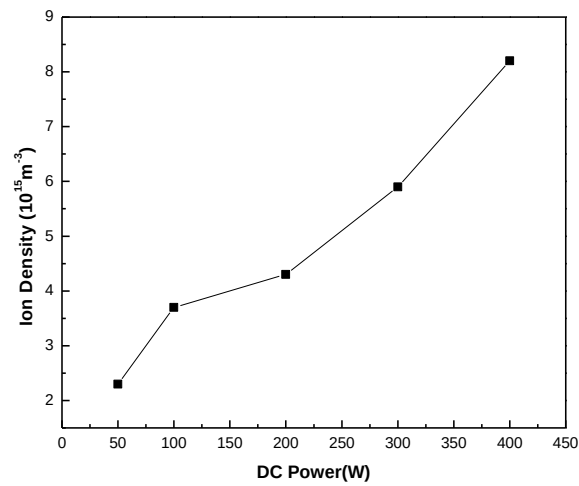


Figure 12

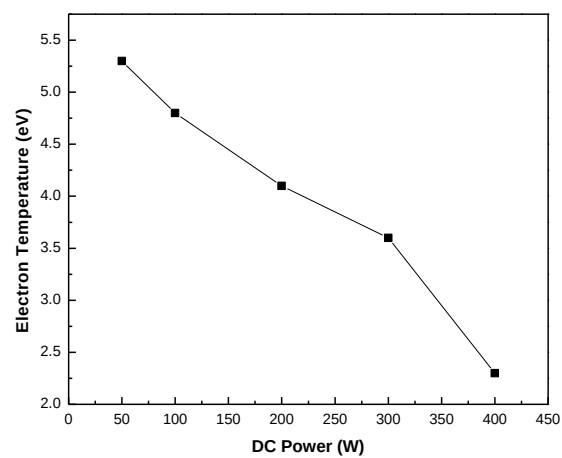


Figure 13

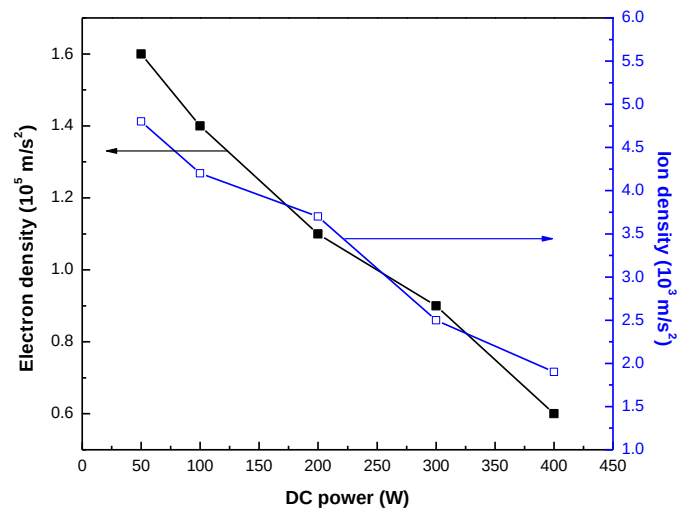


Figure 14

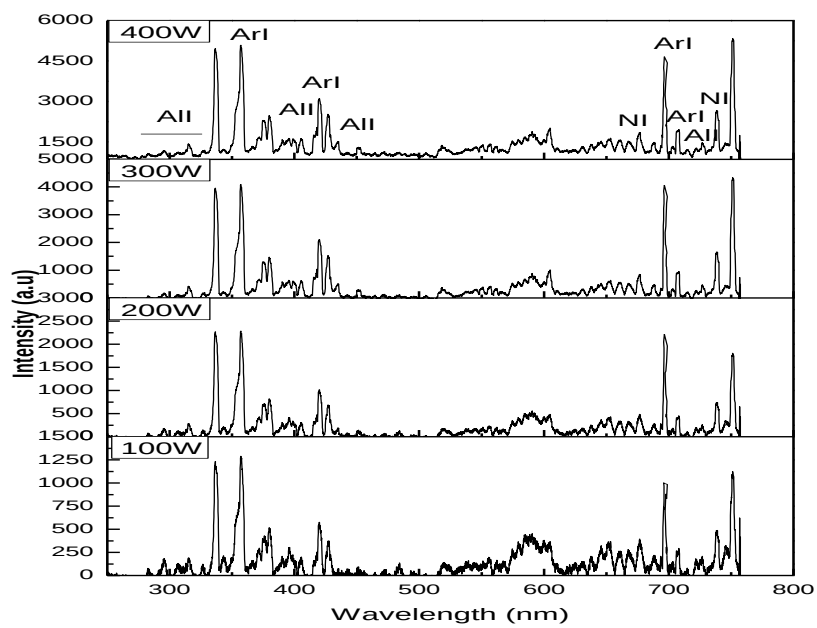


Figure 15