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ELLIPSOMETRIC AND OPTICAL STUDY OF SOME UNCOMMON INSULATOR FILMS ON III-V SEMICONDUCTORS

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ABSTRACT

Optical properties of three types of insulating films that show promise in potential applications in the III-V semiconductor technology were evaluated, namely a-C:H, BN and CaF_2. The plasma deposited a-C:H shows an amorphous behavior with optical energy gaps of approximately 2 to 2.4 eV. These a-C:H films have higher density and/or hardness, higher refractive index and lower optical energy gaps with increasing energy of the particles in the plasma, while the density of states remains unchanged. These results are in agreement, and give a fine-tuned positive confirmation to an existing conjecture on the nature of a-C:H films (1). Ion beam deposited BN films show amorphous behavior with energy gap of 5 eV. These films are nonstoichiometric (B/N approximately 2) and have refractive index, density and/or hardness which are dependent on the deposition conditions. The epitaxially grown CaF_2 on GaAs films have optical parameters equal to bulk, but we found evidence of damage in the GaAs at the interface.

INTRODUCTION

Insulator films on semiconductors have a wide variety of applications, including passivation, insulation, capping, etc. In the case of silicon, the silicon based films SiO_2 and Si_3N_4 have been successfully used for all the required applications. However, the need for high speed devices and integrated circuits requires the use of III-V semiconductor devices and substrates. SiO_2 and Si_3N_4 proved to be inadequate for some of these applications for III-V semiconductors. Thus, new types of insulator films for one or more of the required applications are needed

In this study we have chosen insulator films belonging to three groups of materials: amorphous hydrogenated carbon (a-C:H), boron nitride (BN) and a fluoride (CaF_2). All three groups are new in the search for adequate gate dielectrics for III-V semiconductor devices, the most difficult and crucial application of an insulator film. They can be used for a variety of applications, and they have rather useful properties. Some of these properties are: (a) a-C:H.

Easily prepared as a homogeneous film (2), very hard mechanically and chemically, high breakdown voltage (3) and resistivity, relatively low interface density of states on Si (4) and InP (3), variable energy gap (1 and 5), can be used in metal-insulator-metal MIM structures (6). (b) Boron nitride, BN, has been suggested for use with III-V semiconductors (7). It is a high temperature dielectric (8), suggested for passivation (9), protective coating (10) or x-ray lithography masks (11). The films can be prepared by ion beam (12 and 13), low temperature CVD (14 and 15), plasma deposition (16 and 17), RF glow discharge (18), sputtering (19), borazine pyrolisis (20) and others (7 and 21). The properties of the BN films, as in the a-C:H case, depend on the preparation conditions. (c) Fluorides, (CaF_2 in this report) are of interest as epitaxial growth (nonlattice matched) has been reported on GaAs (22), InP (23) and Si (24). It has a high bandgap (12 eV), relatively high dc dielectric constant (25), can be used in very fine line (approximately 3 nm) lithography (26). Double heterostructures promising three dimensional integrated circuits have been reported in GaAs (2), InP (28) and Si (29). Thus the possibility of growing Si, fluoride and III-V combinations for optical and microwave circuits intermixing is very real. Working FET devices were reported in Si (30 and 31).

In this work, we report a study of a-C:H, BN and CaF_2 films using ellipsometry, UV-visible absorption, IR reflection and transmission, and Auger electron spectroscopy (AES). In the case of a-C:H we correlate our results with existing conjectures (1) regarding optical gap, hardness and graphite content. In the case of BN and CaF_2 we analyze our results to obtain the quality of the films and interfaces and the possible chemical impurities in them.

EXPERIMENTAL

The sample preparation will be discussed only very briefly. a-C:H films were prepared by 30 KHz plasma deposition (1 and 32) using several power settings with a constant pressure, or several pressures with a constant power. In one instance, a constant power and pressure were kept, while the temperature of the substrate was varied. Substrates used in this study were InP (for ellipsometry), quartz (for UV-visible absorption) and Si (for IR Multiple Internal Reflection-MIR). BN films were prepared by Spire Corporation using an ion beam technique and borazine (10 and 13). The ion beam energy was kept very low (150 eV) to reduce the damage to the III-V semiconductor, while the other conditions were set for good quality material (10 and 13), i.e. temperature of 200 and 350 °C, ion beam current density of 100 $\mu A/cm^2$ and using a heated tungsten filament through the ion beam for ion beam neutralization. Four runs were done, using InP, GaAs, Si, quartz and Ge-MIR substrates. Only two runs gave homogeneous samples, and will be reported here. The BN films did not adhere to the GaAs, InP and Ge-MIR substrates. Thus, only good quality films were obtained on Si and quartz substrates. The CaF_2 films on

line in amorphous semiconductors, according to Mott and Davis (38). The absorption coefficient α is given by:

$$\alpha = \frac{4\pi}{nc} \frac{\sigma_{min}}{\Delta E} \frac{(E - E_0)^2}{E} \qquad (1)$$

Here n is the refractive constant, $(\sigma_{min}/\Delta E)$ is a factor determined by the density of states in the bottom of the conduction band and/or the top of the valence band, E is the photon energy and E_0 is the optical energy gap ($A = \alpha d$, where d is the film thickness). Usually, the factor $(4\pi\ \sigma_{min})/(nc\ \Delta E)$ is abbreviated by a single parameter, B^2. A value of $E_0 = (2.35 \pm 0.08)$ eV is obtained from Fig. 1. In addition, a rather significant "tail" is observed at low energies and low absorption values, similar to that found in a-C:H produced by glow discharge (39 and 40). Using an estimate for the thickness d, we calculate a value of $(\alpha E)^{1/2} \cong 100$ ($cm^{1/2}\ eV^{1/2}$) for the "knee, in good agreement with previous data (40). This low value of the "knee" is one of the reasons why "tail" free results are prevalent in the literature (5, 41, and 42). We tried to enforce an exponential Urbach edge (38) on the "tail". We obtain a value of 0.7 eV for the activation energy, which seems too high when compared with the results for a-Si_xC_{1-x} (43). We must point out that the experimental errors are quite high in this experimental range.

Ellipsometric measurements of a-C:H films on InP were done at six wavelengths for 6 values of the deposition power at a flow rate of 70 sccm, corresponding to a of pressure of 315 mTorr. A three phase model was used successfully in analyzing all the a-C:H ellipsometric data. Representative results of the refraction constant n and the extinction coefficient K ($K = 4\pi\alpha/\lambda$) as a function of deposition power P for several wavelengths are shown in Figs. 2 and 3 respectively. The results for K were analyzed in a Tauc plot, as shown in Fig. 4. The straight lines were drawn through the $(\alpha E)^{1/2}$ points above the "knee" at $(\alpha E)^{1/2} \approx 100$. The slopes of these lines, i.e. the parameters B as defined previously using Eq. (1), have the same value for all powers used, denoting that the density of states is not changing as a function of the deposition power in this case. The value of B is 300 $cm^{-1/2}\ eV^{-1/2}$, versus 750 $cm^{-1/2}\ eV^{-1/2}$ for a-Si_xC_{1-x} (43) and in good agreement with results deduced from plasma deposited a-C:H results (39 to 41). The optical gap E_0 found from Fig. 4 is depicted in Fig. 5, as function of the deposition power P. A decrease in E_0 is clearly observed with increasing power. We define the Ar etch rate ER as d/t_e where t_e is the Ar etch time through the whole thickness of the film and d is the ellipsometrically determined thickness. The experimental ER as a function of the deposition power is shown in Fig. 6. A marked decrease in ER versus P is apparent, denoting a hardening and/or densifying of the film with the deposition power.

GaAs were prepared at Westinghouse Research Center as described in Ref. 22, using an ion bombardment cleaning method suitable for epitaxial growth (33).

Ellipsometry was performed using a rotating analyzer ellipsometer with a laser or a Hg arc lamp as light sources. The multiple angles of incidence and wavelengths method was used in the data analysis (34 and 35). This method gives a unique determination of the refractive index and the extinction coefficient at each wavelength without any assumptions on composition of the film and a prior knowledge of the optical constants of the constituents. In most cases, a three phase model was used. However, presence of an interface or an inhomogeneous film require a four phase model (35 and 36). At least five angles of incidence were used at each wavelength. The refractive indexes and extinction coefficients of the substrates used in the ellipsometric work, i.e. GaAs, InP and Si, were taken from Aspnes and Studna (37).

Reflection and transmission at normal incidence were measured continuously in the IR (2.5 to 50 μm range) using a computer controlled Perkin Elmer 1430 spectrometer. During the measurements, the sample cell was continuously purged with dry nitrogen. In some cases, MIR plates were used to enhance sensitivity in the IR. Absorption in the UV-visible (190 nm to 3.2 μm wavelength range) was measured using a computer controlled Perkin Elmer Lambda 9 spectrometer. The data was taken discretely using a slit width of 2 nm. Up to 200 points were measured on each sample. The sample cell was continuously purged with dry nitrogen during the measurements. A blank quartz substrate was placed in the reference path, for background correction. However, no correction was made for the difference in the reflectivity of the quartz reference plate and the film.

AES was used in conjunction with Ar ion sputtering for depth profiling. Absolute etch rates were determined by comparison with the ellipsometrically determined film thickness. Absolute values of the atomic concentrations were obtained by correcting the peak to peak signal amplitude by the corresponding elemental sensitivity factor.

RESULTS AND DISCUSSION

a-C:H Films

Absorption in the UV-visible range was taken on a-C:H films made on quartz substrates from methane gas (CH_4), at a power of 150 W and a flow rate of 50 sccm, corresponding to a pressure of 245 mtorr. The directly measured parameter is the absorbance A. In Fig. 1 we show a Tauc plot, i.e. $(AE)^{1/2}$ versus E. This plot is a straight

At this stage, we will compare our results with the conjecture forwarded by S. Kaplan et al. (1). They claim that since double bond hydrogenation is an exothermic process, "graphitic" behavior is favored over tetrahedral bonding in higher energy growth environments. They show evidence of this assumption by comparing a-C:H films made by five different experimental configurations. As a-C:H properties are dependent quite strongly on the many variables encountered in different preparation conditions, it seems that a better test of this assumption is in order. In addition, their results show a rather striking feature: a-C:H films exhibiting more "diamondlike" behavior, i.e. larger bandgap and more tetrahedral bonding show a steep decrease in their hardness as compared with the more "graphitic" films.

Our results confirm this model, including the hardness test. The higher the plasma deposition power, the more sp^2 versus sp^3 bonds are made, giving a more "graphitic" film, with smaller bandgap (Fig. 5) and higher hardness or density (Fig. 4). We further tested this model using the correlation of the refractive index n in the visible with deposition power (Fig. 2), i.e. higher n for the more "graphitic"-like films, grown at higher deposition powers. Fig. 7 shows the ellipsometrically measured refractive index in the visible as function of the deposition temperature. An increase in n, i.e. more "graphitic" behavior, is evident as the energy growth environment is increased. Similar behavior was found in the literature (40 and 44). The last point in Fig. 7, at 250 °C, is anomalous. We believe that the deposition process is somewhat changed above 200 °C. For example, we could not deposit any a-C:H films at all (2) on III-V-semiconductors above 200 °C. Changes in the deposition above 200 °C were also reported elsewhere (44). In Fig. 8 we show a general decrease in ellipsometrically measured refractive index n with increasing deposition pressure. The higher the pressure, the lower is the average energy per particle, i.e. we expect less "graphitic" film. Finally, we show in Fig. 9 a Tauc plot for a-C:H film made using n-butane, C_4H_{10}. The power (150 W) and pressure (245 mtorr) are equal to that used for the film shown in Fig. 1. However, the optical energy gap is now (3.05±0.20) eV versus 2.35 eV. The average energy per mass (or the particles' momentum) is smaller for the heavier butane molecule as compared to methane. Thus we obtained a higher bandgap in agreement with the model.

BN Films

An AES profile of one of the BN films, made at 200 °C substrate temperature is shown in Fig. 10. The film is nonstoichiometric with a B/N ratio of about two. The same ratio was found in the film made at 350 °C. Small oxygen and carbon contaminations are also evident, with excess oxygen at the interface and a large excess carbon on the surface. It is believed that the optical properties of boron oxide are similar to that of BN, and therefore the excess oxide will not be detected. Nonstoichiometric BN films, with B/N ratios of two have

been shown to exhibit properties not much different than the stoichiometric composition (15). Thus, we believe that our results are at least qualitatively, representative of ion-beam sputtered BN with almost stoichiometric composition. Additional experimental results on the sample analyzed in Fig. 10 will now be shown. In Fig. 11, we show IR reflection (R in arbitrary units) and transmission (T) spectra of BN on Si. The usual BN peaks (9, 18 to 21, and 45), shown to be present in the amorphous phase (18 and 21), at 1370 and 800 cm^{-1}, are observed. The peak at 1370 is wide and asymmetric, as previously reported (18 and 19), possibly due to contribution from boron oxide. The transmission results show two additional "valleys," that are due to the Si substrate. N-H and the other bands could not be detected due to the low sensitivity. In Fig. 12 the absorption coefficient α, in the visible range, is shown versus the photon energy E. The thickness was obtained from ellipsometry and the absorbance measured directly. We observe a peak in α at 3.2 eV, a "shoulder" just below 5 eV and a steep increase below 5 eV. A Tauc plot using the same results for α as shown in Fig. 12 is shown in Fig. 13. We found an optical gap of (5.0±0.4)eV with a density of state parameter of $B = (850\pm50)cm^{-1/2}\ eV^{-1/2}$. Very similar results were found for the BN film deposited at 350 °C, with an optical gap of (5.1±0.4)eV, $B = (1000\pm80)cm^{-1/2}\ eV^{-1/2}$, a α peak at approximately 3.2 eV and a "shoulder." These results are in contrast with a direct optical gap observation (21), in agreement with the values of the optical gap of approximately 5 eV reported earlier (18 and 19) for amorphous BN, but a little higher than 4 eV reported much earlier (9). The result obtained in Fig. 13 points to the amorphous nature of this film. It is believed that only 20 percent or less of the ion beam deposited BN is in the cubic phase, with the remainder being amorphous, but definitely not hexagonal (10). The exact origins of the peak at 3.2 eV and the "shoulder" at 5 eV are not known. We tried to fit the "shoulder" to an Urbach edge, but it does not shown exponential behavior. The refractive index n versus wavelength as obtained by ellipsometry and using a three phase model, is shown in Fig. 4 for two BN films. The extinction coefficient is either zero at $\lambda \geq 546.1$ nm, or very small, in agreement with the absorbance data. We see a small decrease in n with increasing wavelength, typical of high band gap material. There is a definite decrease of n with increasing substrate temperature. The value of n reported here is in agreement with some of the reported data, especially when higher temperature or higher energy deposition was involved. The higher the energy or the substrate temperature, the lower is the reported refractive index (15, 18, 20, and 45), again in good agreement with our data. We note that this trend is opposite to that found in a-C:H. We will now suggest an explanation of this trend in n versus deposition temperature, at least in this case. We calculated the absolute Ar etch note ER of BN, similar to the case of a-C:H (see Fig. 6). We obtained ER = 7.7 nm/min and 3.8 nm/min for the 350 and 200 °C substrate temperatures respectively. This shows that a harder and/or denser film was obtained at the lower substrate temperature, a fact that by itself is pointing to a higher refractive index n. A similar dependence of n versus the hardness

was found in a-C:H, but it occurred when the a-C:H substrate temperature was raised. We have also tried to fit the ellipsometric data to a four phase model. We found that there is no improvement in our fit while the fourth phase was an interface layer. However, when the top 100 Å were assumed to be different from the rest of the sample (36), we obtained a decrease by a factor of 5 in the variance. Thus, the top layer was shown both by AES and by ellipsometry to be different from the rest of the film. Numerically, the top layer has a slightly lower refractive index, while the absolute value of n for the bulk of the film was pushed up by roughly 5 percent versus the data shown in Fig. 14.

CaF_2 Films

The epitaxial CaF_2 films were grown on GaAs. Thus, only IR and ellipsometric optical characteristics could be performed. A reflection spectrum (R in arbitrary units) is shown in Fig. 15. The main feature observed is the large peak at approximately 280 cm^{-1}, typical of bulk CaF_2 fluorite material (46). Ellipsometric data was obtained for two CaF_2 films. Detailed results for one sample (NCG-1) are shown in Table I. The parameter δ is the variance, defined by (36)

$$\delta = \sum_{i,j} [(\psi - \psi')^2 + (\Delta - \Delta')^2]/(N - M) \qquad (2)$$

Here ψ and Δ are the experimental values, ψ' and Δ' are the calculated values, N is the number of experimental ψ and Δ values (56 in this case) and M is the number of free parameters. The summation is on all angles of incidence i and wavelengths j. The results shown in column A were obtained using a three phase model with n and K of GaAs from Ref. 37. The film has exactly the same refractive index (within experimental error) of bulk CaF_2 (26). The result $K = 0$ was also positively checked. However, the value of δ is very high. In column B we used a three phase model, a fixed value of n for the CaF_2 film, (1.449 for all wavelengths shown) and varying n and K for the substrate. The result shown in column B, Table I for GaAs shows n and K values which are approximately 5 percent different from those reported by Aspnes and Studna (37). This discrepancy is higher than the experimental error, pointing to a change in the top layer of GaAs. Another approach, that seems more plausible, was to try a four phase model. We fixed the values of n and K of the GaAs and the CaF_2 according to literature (26 and 47) and got the result shown in column C. The variance is as low as in column B, and the result shows an interface with n and K corresponding to roughly 85 vol % GaAs and the rest voids. Analysis was done using the effective medium approximation, as shown elsewhere (36). We believe that the damage to the top GaAs layer, (which became later the interface) was done during the cleaning step (33).

Results of measurements at 6328 Å for two CaF_2 films are compared in Table II. It happened that this wavelength was a very strong "resonant" line (35) for the NCG-1 sample, thus showing very high values of δ. These values are due to experimental error in setting and measuring the absolute value of the angle of incidence. Two sets of results are shown, which are equivalent to columns A and B in Table I. The main conclusion from Table II is that both films behave like bulk CaF_2 and in both cases there is a definite change in the GaAs values of n and K as compared to the results of Aspnes and Studna. Ellipsometric measurements done on the same NCG-1 film elsewhere (48) and analyzed for the GaAs optical constants n and K show better agreement with Ref. 37 for values of n, but the same discrepancy in K.

CONCLUSION

Our studies on a-C:H are in good agreement with the conjecture forwarded by Kaplan et al. (1) regarding the direct correlation between the energy environment during deposition and the "graphitic" behavior. We have tested and confirmed this assumption with measurements of the optical energy gap, the sputtering etch rate and the refractive index. Work is now in progress to correlate these parameters with the hydrogen content (47) as measured by IR and by nuclear reaction methods (2). The main conclusion regarding the ion beam deposited BN is that the material is amorphous and has an optical bandgap of 5 eV. Refractive index and hardness are dependent on the deposition temperature. These films are not optimized yet, but the high bandgap is promising for applications as a dielectric. The work in CaF_2 shows a good quality film, with optical properties similar to bulk. There are indications that the cleaning procedure has damaged the top layer of the substrate. The quality of the film shows good promise for future applications in semiconductor devices.

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TABLE I. - ELLIPSOMETRIC RESULTS FOR AN EPITAXIAL CaF_2 FILM ON GaAs

[Three assumptions are used for analysis, as explained in the text, giving results A, B and C.]

λ (Å)	A CaF_2		B GaAs		C Interface	
	n	K	n	K	n	K
3650	1.442	-	3.948	2.03	3.32	2.32
4047	1.445	-	4.634	2.02	3.58	1.85
4358	1.447	-	5.25	1.43	3.92	1.46
5461	1.439	-	4.347	0.03	2.70	----
5770	1.440	-	4.175	0.06	1.96	----
d(A)	1494.2		1496.4		d_1 = 22.4 Å; d_2 = 1478.5 Å	
δ	0.687		0.114		0.144	

TABLE II. - COMPARISON OF ELLIPSOMETRIC RESULTS OF 6328 Å FOR TWO EPITAXIAL CaF_2 FILMS ON GaAs.

[In both cases the extinction coefficient of CaF_2 was fixed at 0.]

Sample	CaF_2		GaAs		δ
	n	d(A)	n	K	
NCG-1	1.447	1488.5	3.856[a]	0.196[a]	0.624
	1.450	1497.1	3.914	~0	0.565
NCG-2	1.432	1578.[illegible]	3.856[a]	0.196[a]	0.194
	1.455	1547.[illegible]	4.110	~0	0.117

[a]Fixed.

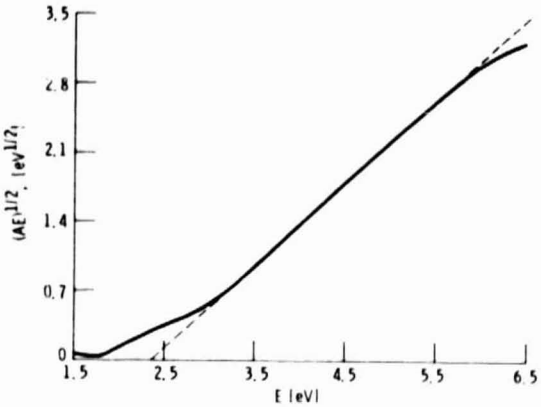

Fig. 1. - Tauc plot of $(\alpha E)^{1/2}$ vs. E for a-C:H CH_4 plasma deposited film on quartz at 150 watts, 245 mTorr for 20 minutes.

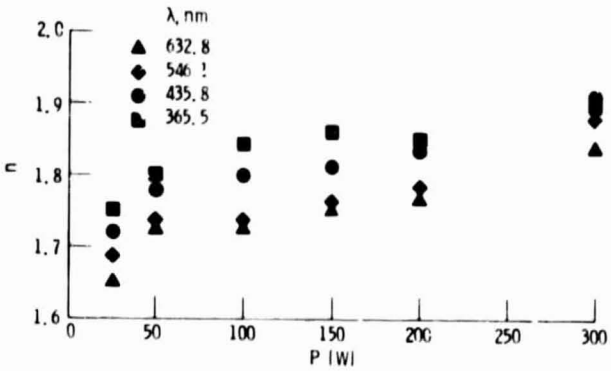

Fig. 2. - Refractive index n vs. deposition power P for a-C:H CH_4 plasma deposited film on InP at 315 mTorr for several wavelengths λ.

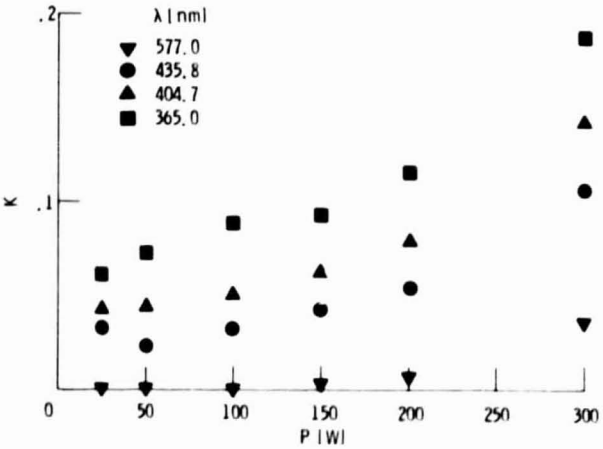

Fig. 3. - Extinction coefficient K vs. deposition power for a-C:H CH_4 plasma deposited film on InP at 315 mTorr for several wavelengths λ.

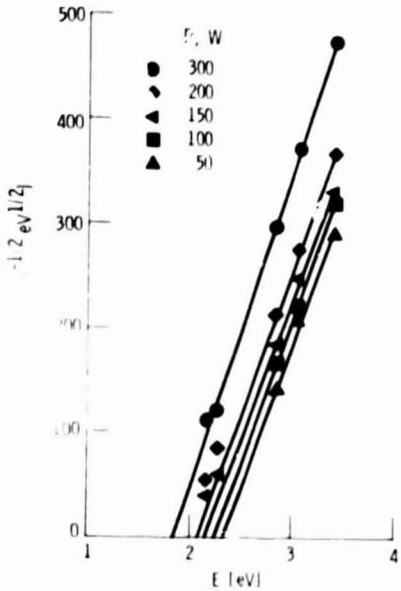

Fig. 4. - Tauc plot of $(\alpha E)^{1/2}$ vs. E for a-C:H CH_4 plasma deposited film on InP at 315 mTorr for several deposition powers P.

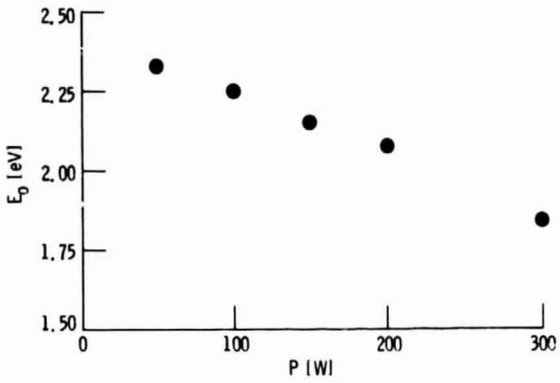

Fig. 5. - Optical energy gap E_0 vs. deposition power P for a-C:H CH_4 plasma deposited film on InP at 315 mTorr.

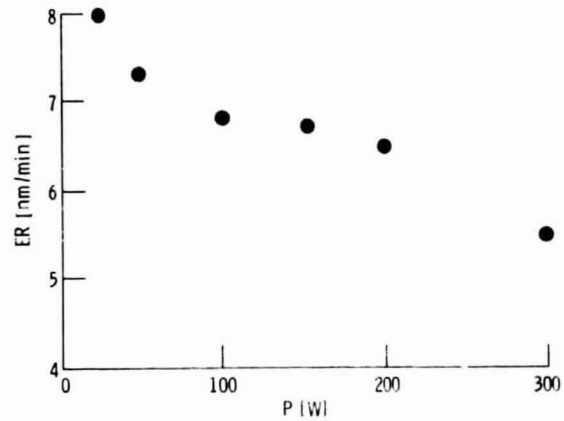

Fig. 6. - Absolute argon etch rate ER vs. deposition power for a-C:H CH_4 plasma deposited film on InP at 315 mTorr.

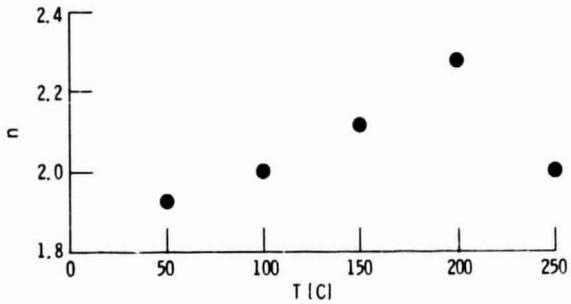

Fig. 7. - Refractive index n vs. substrate temperature for a-C:H CH_4 plasma deposited film on Si at 100 watt, 245 mTorr.

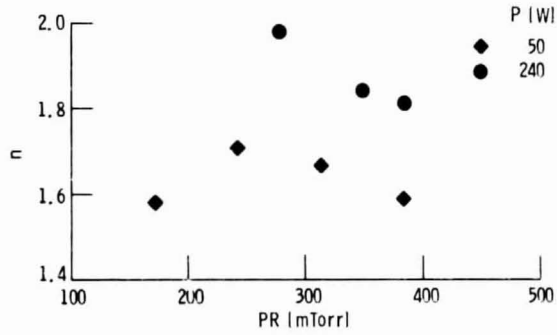

Fig. 8. - Refractive index n vs. pressure PR for a-C:H CH_4 plasma deposited film on InP at 2 deposition powers P.

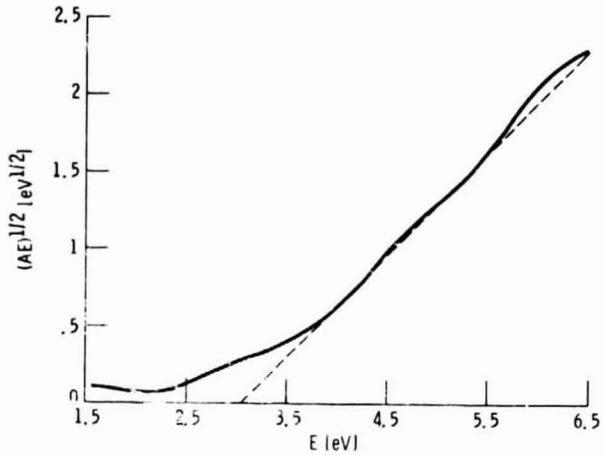

Fig. 9. - Tauc plot of $(AE)^{1/2}$ vs. E for a-C:H C_4H_{10} plasma deposited film on quartz at 150 watts, 245 mTorr for 20 minutes.

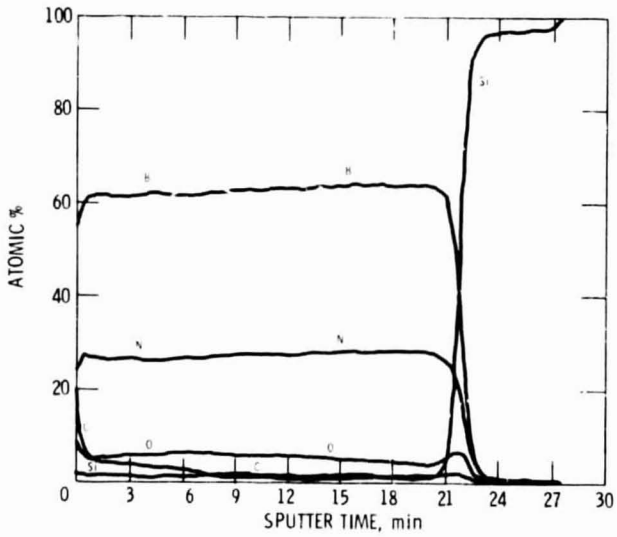

Fig. 10. - AES atomic percent vs. sputter time for ion beam deposited BN film on Si. Deposition temperature 200 °C, ion beam energy 150 eV.

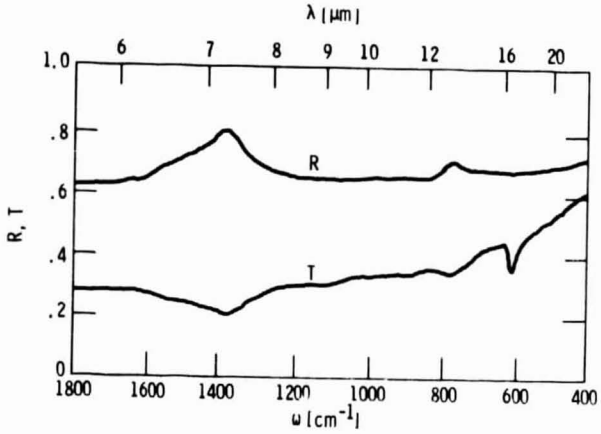

Fig. 11. - Reflection R and transmission T vs. wavenumber ω for ion deposited BN film on Si. Deposition temperature 200 °C, ion beam energy 150 eV.

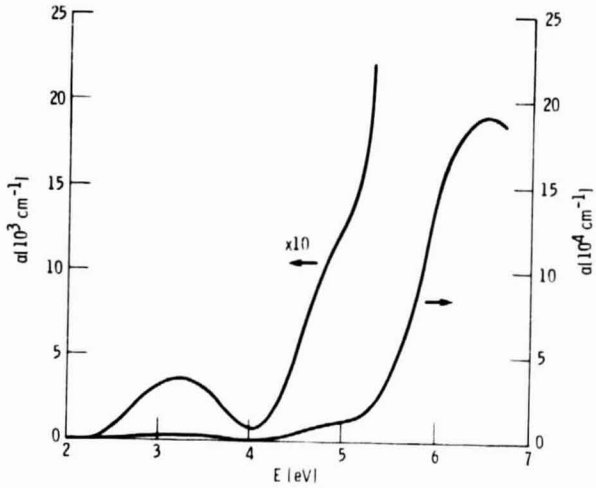

Fig. 12. - Optical absorption α vs. photon energy E for ion deposited BN film on quartz. Deposition temperature 200 °C, ion beam energy 150 eV.

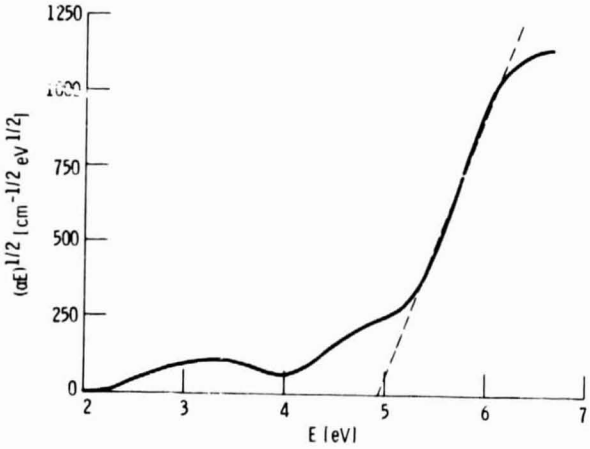

Fig. 13. - Tauc plot $(\alpha E)^{1/2}$ vs. E for ion deposited BN film on quartz. Deposition temperature 200 °C, ion beam energy 150 eV.

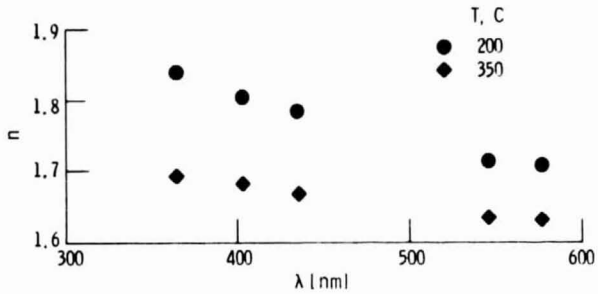

Fig. 14. - Refractive index n vs. wavelength λ for ion deposited BN films on Si at 2 substrate temperatures.

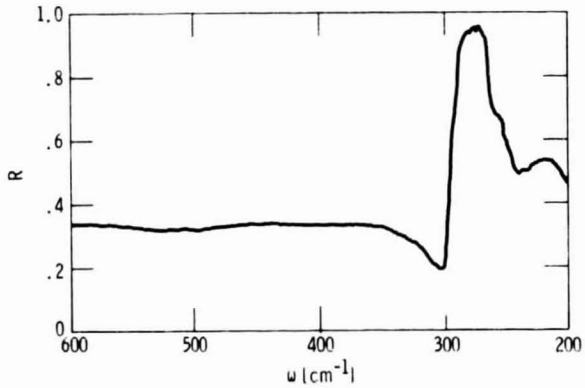

Fig. 15. - Reflectivity R vs. wavenumber ω for epitaxial CaF_2 film on GaAs.

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15. Supplementary Notes

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16. Abstract

Optical properties of three types of insulating films that show promise in potential applications in the III-V semiconductor technology were evaluated, namely a-C:H, BN and CaF_2. The plasma deposited a-C:H shows an amorphous behavior with optical energy gaps of approximately 2 to 2.4 eV. These a-C:H films have higher density and/or hardness, higher refractive index and lower optical energy gaps with increasing energy of the particles in the plasma, while the density of states remains unchanged. These results are in agreement, and give a fine-tuned positive confirmation to an existing conjecture on the nature of a-C:H films (1). Ion beam deposited BN films show amorphous behavior with energy gap of 5 eV. These films are nonstoichiometric (B/N approximately 2) and have refractive index, density and/or hardness which are dependent on the deposition conditions. The epitaxially grown CaF_2 on GaAs films have optical parameters equal to bulk, but we found evidence of damage in the GaAs at the interface.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
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