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INVESTIGATION OF MECHANICALLY HARD, CHEMICALLY INERT ANTIREFLECTION COATINGS FOR PHOTOVOLTAIC SOLAR MODULES

Final Technical Report for April 1, 1980—March 31, 1981

By
T. J. Moravec

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Solar Energy

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Honeywell Corporate Technology Center
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ACKNOWLEDGEMENTS

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SECTION I
SUMMARY

The overall objective of this program is to determine the optical properties of i-Carbon (diamond-like) films and determine if these films can be developed into antireflecting (AR) coatings for silicon solar cells. The i-C films have been produced on glass, silicon, and KCl by radio frequency (RF) plasma decomposition of the alkane gases. Films were also produced on silicon solar cells by low-energy ion beam techniques. These coatings did not perform as well as those made from hydrocarbon gases.

Significant progress has been made in understanding the deposition parameters that affect the optical properties of the films. The optical constants n and k have been determined over a large range of process parameters and source gas. The degree of hydrogen incorporation in these films has been studied by SIMS analysis. It was found that the lower optically absorbing films contain more hydrogen. This hydrogen does not, however, manifest itself in fundamental C-H absorption bands in the infrared.

Very efficient single-layer quarter-wave i-C AR coatings have been produced on single-crystal and SOC Si solar cells. An increase in cell efficiency of 40% over uncoated cells has been achieved.

SECTION II
INTRODUCTION

This program is an outgrowth of a program started at the Honeywell Corporate Technology Center (CTC) to study the preparation and properties of diamond-like carbon thin films. These films can be made by several techniques. Two techniques that we have employed are radio-frequency (rf) plasma decomposition of a hydrocarbon gas (i.e., butane), and low-energy carbon ion beam deposition. We have deposited both types of films on various metals, semiconductor, and insulator substrates. The goal of the present program is to develop these diamond-like carbon films as an AR and, possibly, encapsulating coating for low-cost silicon photovoltaic modules.

Previous studies have shown that acid rain common in an industrial environment results in the most rapid degradation to photovoltaic modules. This degradation could be reduced by coating with a diamond-like coating due to the insolubility of diamond (carbon) in all acids and bases. The diamond-like coatings are mechanically hard (knoop hardness 1850) and adhere well to silicon, thus resisting mechanical abrasion. Abrasion resistance is important for protection against erosion due to the washing processes required for periodic maintenance of solar cells. Encapsulation must protect all exposed metal as well as the silicon.

Under a previous program, it was noted that the carbon films absorb visible light. Sections IV and V describe the experiments preformed to study this absorption and also to quantify the optical properties of the films. There exists a considerable variation in these properties. Although much data have been obtained on the optical properties of the films, the nature of the optical absorption has not been clearly discerned. Some possible mechanisms are discussed in Section VII. The two different methods used to prepare the carbon films are first described. Then, the results of the optical properties study and secondary ion mass-spectrometry (SIMS) analysis are discussed. Data are also presented on the response of AR-coated solar cells. Weissmantel[1] recommends that the diamond-like carbon films be referred to as i-C carbon (i-C) films.

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1 Weissmantel, C.H.R., Bewilogua, K. and Schurer, C., "Characterization of Hard Carbon Films by Electron Energy Loss Spectrometry," Thin Solid Films 61, 21 (1979).

SECTION III
DEPOSITION OF I-C FILMS

PLASMA CVD FROM HYDROCARBON FILMS

This technique is similar to that described by Holland and Ojha.[2-4] A hydrocarbon gas is decomposed by the action of an RF plasma with the formation of a film. Substrates to be coated are placed on a negatively biased RF electrode. Under the proper power and pressure conditions,[2-4] the RF plasma decomposed the hydrocarbon (e.g., butane) gas sufficiently to result in a mostly carbon film. This process is similar to plasma-induced chemical vapor deposition (CVD) except that the substrate is typically on a water-cooled electrode.

Our experimental arrangement is as follows. The films were deposited in a conventional RF sputtering system pumped by an oil diffusion pump. The substrates to be coated were placed on a 5-inch-diameter water-cooled electrode that comprised the cathode or "hot" side of an RF power supply, usually referred to as "RF etch mode." A negative d-c bias can also be applied concurrently to this electrode (shown schematically in Fig. 1). The gas to be decomposed by the RF plasma is inlet above the samples. Typically, flows are 1 to 2 standard cubic centimeters per minute for the gases examined. Much of the work in this program was spent in studying the films produced from butane, C_4H_{10}. Later on, three other alkane gases, ethane (C_2H_6), propane (C_3H_8), and methane (CH_4), were briefly studied. As discussed below, these gases produced films that had lower visible light absorption and thus produced better results on solar cells than those from butane. We have found no dependence upon the electrode composition, but built-up deposits must be removed periodically to reduce plasma instabilities. The RF power (in watts) and chamber deposition pressure (in torr) were the main variables. We designate these two as power/pressure or p/p ratio. It should be remembered that this is for a 5-inch-diameter electrode. The pressure was measured with a Schulz-Phelps ionization gauge tube corrected for hydrocarbon gases. Very uniform coatings were obtained over the entire 5-inch electrode area.

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2 Holland, L. and Ojha, S.M., Thin Solid Films 38 L17 (1976).
3 Ojha, S.M. and Holland, L., Thin Solid Films 40 L31 (1977).
4 Holland, L. and Ojha, S.M., Thin Solid Films 48 L21 (1978).

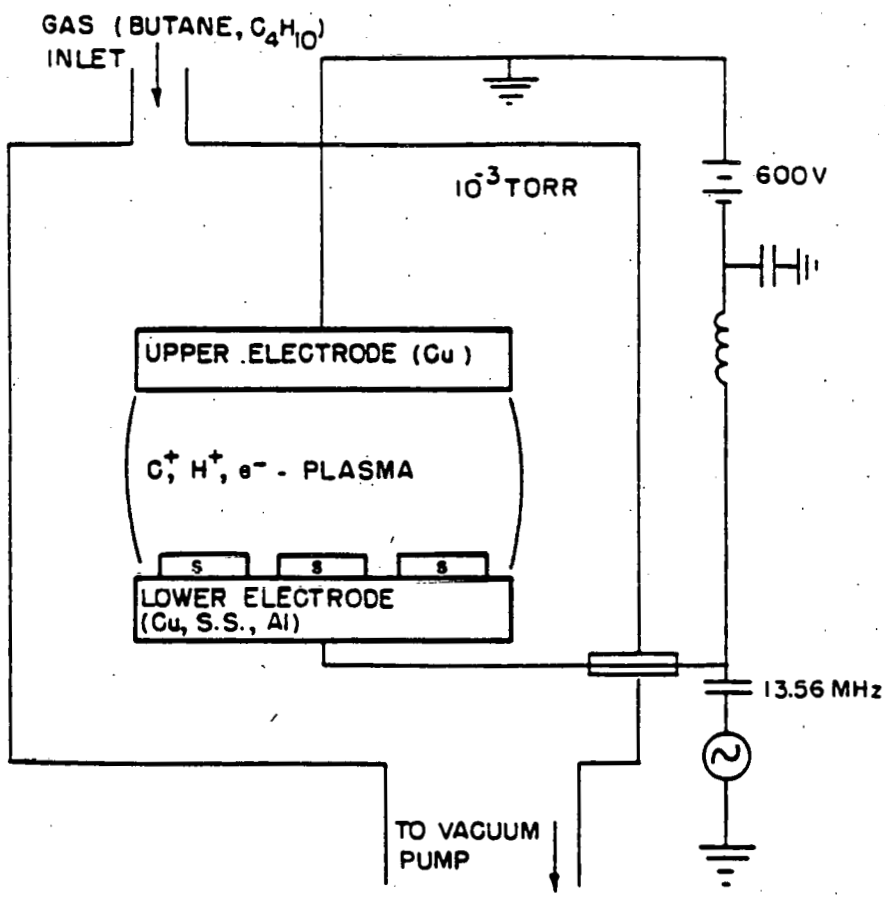

Figure 1. Schematic of RF plasma reactor for producing i-C films from a hydrocarbon gas.

A light argon plasma etch was performed for 5 to 10 minutes on each sample before deposition. This improved the adhesion of the i-C film.

A few experiments were run with a graphite electrode for the upper electrode, splitting the RF power between upper and lower electrode, and mixing argon with the hydrocarbon gas. This should result in the argon sputtering off carbon from the graphite electrode and incorporating more carbon in the resulting film. (This should be compared with ordinary sputtering of graphite which results in a black, amorphous carbon deposit.) i-C films were obtained this way, which showed similar characteristics to those obtained with only the hydrocarbon gas.

ION BEAM DEPOSITION

Low-energy ion-beam-deposited films up to 0.5μm thick have been deposited on metals, semiconductors and insulators by the arrangement shown in Fig. 2, which is similar to that described by Joy et al.[5] Sputtering gas (G), e.g., argon, was inlet through a carbon disc electrode into a discharge chamber maintained by a high-voltage arc and magnetic field (H). Carbon ions sputtered off the electrode were extracted by grids 2, 3, and 4, and impinged on substrate 5. Typical voltages (in volts) with respect to ground were (1) -2000, (2) -IE, (3) -1500, (4) 0, (5) -10, where IE is the desired ion energy at the substrate, typically 50 to 100V.

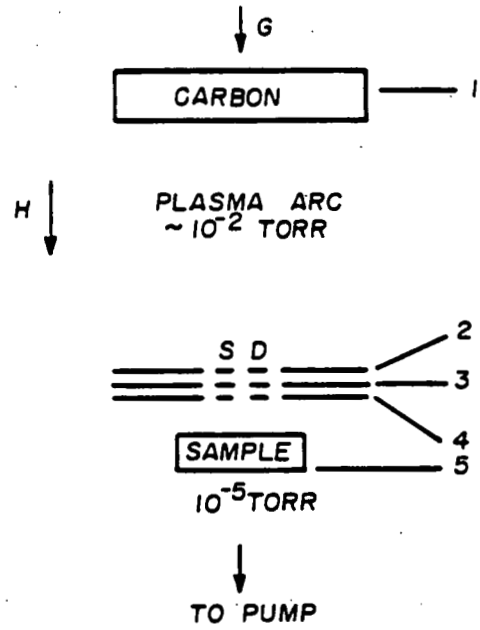

Figure 2. Schematic of carbon ion beam source. G: sputter gas inlet, H: magnetic field, SD: secondary discharge region; electrical connections 1: cathode, 2: anode grid, 3: acceleration grid, 4: screen grid, 5: sample connection.

The biggest problem in performing ion-beam deposition was to make a deposit uniform over a 5-cm^2 solar cell. The pattern of the deposition from a single aperture in the grid resembles a bull's eye with the center being the thickest. In order to increase deposition uniformity, multi-holed grids were fabricated. This resulted in films composed of many individual bull's eye

5 Joy, D.C., Spencer, E.G., Schmidt, F.H and Sanssalone, F.J., Proc. of Sixth European Congress on Electron Microscopy, Jerusalem, 1976 unpublished.

patterns. Increasing the grid-to-substrate distance resulted in the deposition becoming more uniform, but the deposition rate decreased enormously ($\sim$ several days for 500Å film).

It was decided to rotate the substrate under a set of grids that contained three large holes. This seemed to be the best compromise between rate and uniformity. However, we were still not able to obtain a ±5% uniform coating over an entire 5-cm^2 silicon solar cell. In order to maintain a hard coating, a bias of 10 to 30 volts is required on the substrate. This bias partially refocusses the low-energy ion beam (energy ≈ 100eV) and contributes to nonuniformity. Thus, the cells coated to date were not very uniform and exhibited poor enhancement from AR coating (see below). This problem may be solvable but there was not time in the present program to attempt more solutions.

SECTION IV
OPTICAL PROPERTIES

The optical properties were studied to learn something about the optical response of the i-C films. The i-C films were deposited onto one side of microscope (1-inch by 3-inch) glass slides and also 1-inch-diameter polished KCl discs. The reflectivity and transmission from 0.4 to 0.8μm was measured on the coated glass slides by a CARY-14 Spectrophotometer. The transmission of the coated KCl was measured from 2.5 to 25μm by a Digilab Fourier Transmission Spectrometer (FTS-14). The thickness of the films was measured by a diamond stylus step-height measuring instrument (Talystep). The thickness may also be estimated by interference color on silicon.[6] The optical constants n and k of the films were calculated from the measured reflectivity, transmission, and thickness by the method of Nilsson.[7] Due to the uncertainty in the measured values, the accuracy of the calculated n and k by this method is no better than 5%.

Figure 3 shows results obtained for films generated from butane, the gas studied the most in this program, for two different power/pressure (p/p) values. In both cases, the absorption increases (k value) in a smooth manner from long to short wavelengths while the refractive index remains roughly constant, within experimental error, between n = 2.16 and n = 2.28 (average n = 2.22). The k curves show no sharp features indicative of an absorption edge or impurities. Thus, it is difficult to assign a mechanism responsible for the absorption in this region. However, the k values are considerably less for the lower p/p conditions. Since the absorption coefficient, α, is given by

$$\alpha = 4\pi k/\lambda$$

where λ is the wavelength of light, the films made at p/p = 1 x 10^3 W/torr have lower overall absorption. We have determined that at even lower p/p

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6 Moravec, T.J., "Color Chart for Diamond-like Carbon Films on Silicon," Thin Solid Films 70, L9 (1980).

7 Nilsson, P.O., "Determination of Optical Constants from Intensity Measurements at Normal Incidence," Appl. Opt. 7, 435 (1968).

values the absorption at λ = 0.65μm does not go below k = 0.05 for either ethane or butane until the transition region (from hard to soft film) is reached (see Fig. 6).

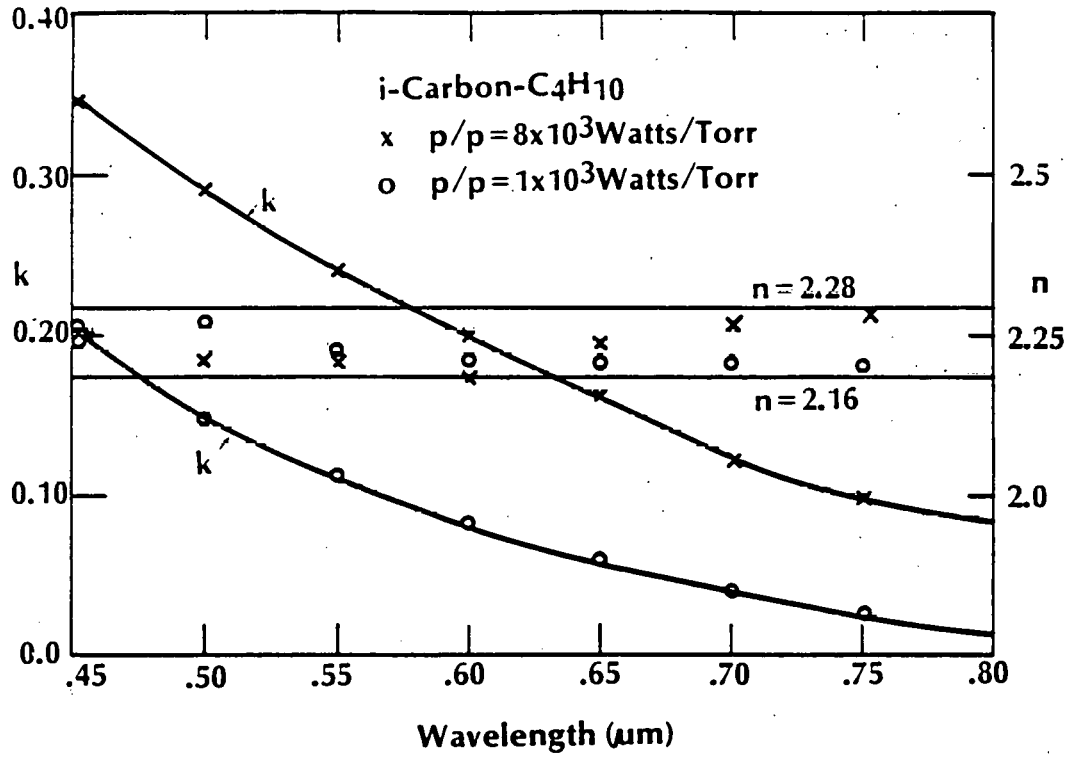

Figure 3. n and k versus wavelength for butane-generated i-C films. n for both films falls within the values n = 2.15 and n = 2.28.

Figure 4 shows the variation of n at λ = 0.65μm over three orders of magnitude of p/p. From p/p $\sim 1 \times 10^4$ to 8×10^1 W/torr, n decreases very slowly. But from p/p = 4×10^2 to 2×10^2 W/torr, n decreases precipitously. It is in this region that the lowest visible light absorption i-C films have been made. No correlation has been noted between refractive index or absorption and d-c bias on the substrate electrode; however, more C-H bonding appears to be present in films made without this bias.[8] The other alkane gases, propane (C_3H_8), ethane (C_2H_6), and methane (CH_4), were examined to a limited extent. It was found that the films prepared from these gases exhibited optical behavior similar to butane. They possessed refractive index curves similar to Fig. 4. The absorption was high at high ratios of power pressure

8. Moravec, T.J. and Orent, T.W., "Electron Spectroscopy of Ion Beam and Hydrocarbon Plasma-generated Diamond-like Carbon Films," J. Vac. Sci. Tech. 18, 226 (1981).

and correspondingly low for low ratios. However, we were able to make more consistently lower absorbing films and thus better AR coatings on solar cells with propane and methane than with butane and ethane (see Table 2).

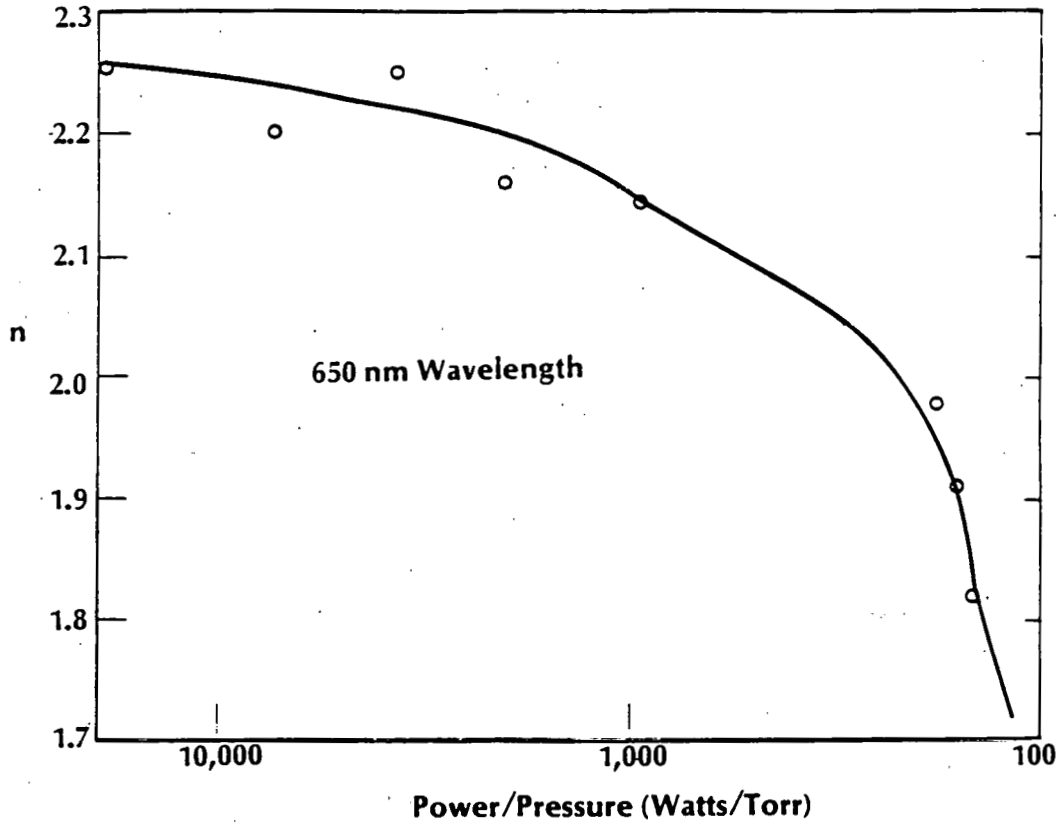

Figure 4. Variation of n versus power/pressure at 0.650μm wavelength. Power/pressure scale is logarithmic.

As mentioned in Section III, several experiments were tried co-sputtering a graphite target with argon onto the electrode where the i-C film was forming. It was thought that this might lower the optical absorption if the carbon was incorporated in a structure other than graphite. In general, the optical constants obtained this way with C_2H_6 were similar to those obtained with this way with C_2H_6 were similar to those obtained with just C_2H_6 flowing. Thus, this method did not increase or decrease the optical absorption.

SECTION V
STUDY OF HYDROGEN AND ELEMENTAL ANALYSIS

It is natural to look for hydrocarbon absorption in the i-C films, since they are made from hydrocarbon gases. It is well known that soft, hydrocarbon films can be made by plasma polymerization techniques in the region of $p/p < 100$ W/torr.[9] All hydrocarbon films have a fundamental absorption in the infrared in a band at 3000 to 2840 cm^{-1} (photon frequency or wavenumber equivalent to 3.33 to 3.52μm wavelength) due to C-H bond stretching and weaker absorptions at 1450 cm^{-1} and 1357 cm^{-1}. These are always prominent and easily identified as shown in Fig. 5. This shows the transmission spectra from 4000 cm^{-1} (2.5μm) to 400 cm^{-1} (25μm) of a 1μm thick plasma polymerized ethane coating made by the method of ref. 9 on both sides of a KCl forged polycrystalline disc.

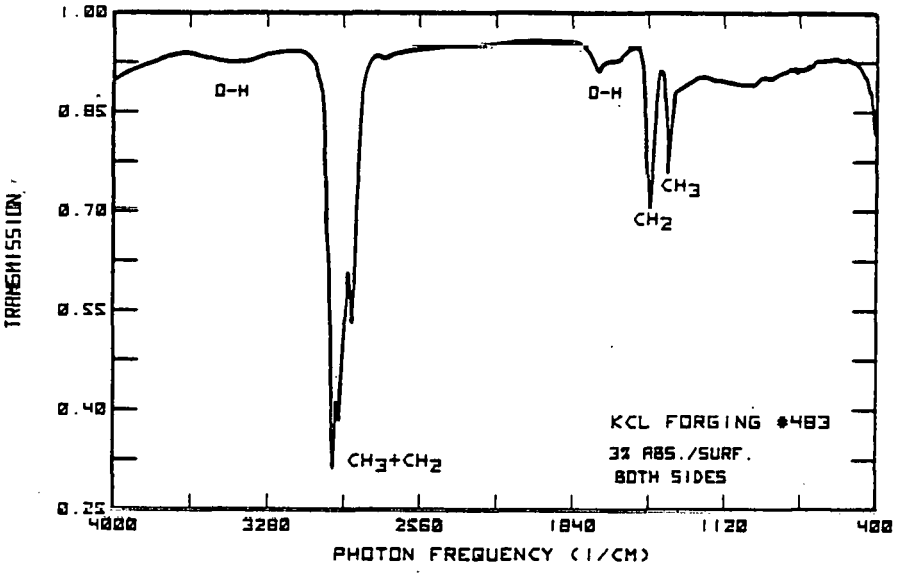

Figure 5. Transmission of a KCℓ polycrystalline disc coated on both sides with a 1μm-thick plasma polymerized ethane hydrocarbon film. The 3% absorption/surface is at 10.6μm.

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9. Hiratsuha, H., Ahovali, G., Shen, M. and Bell, A.J., "Plasma Polymerization of some Simple Saturated Hydrocarbons," J. Appl. Sci. 22, 917 (1978).

The strong fundamental absorption is labeled CH_3 + CH_2 and the weaker absorptions are labeled CH_2 and CH_3. Also evident are weak O-H absorptions due to absorbed water on the KCl. Fig. 6 shows the transmission of uncoated KCl (curve 1) and that of three i-C films made from ethane by the process described in Section II. Curve 3 is of a film 0.45μm thick and displays an interference minimum near 2300 cm^{-1}. Curves 2 and 4 are for a film 0.2um thick and exhibit a smooth decrease in transmission to shorter wavelengths due to scattering. This scattering results from film separation from the KCl due to stresses. Curve 4 has been offset by 15% for clarity. Note that there is almost no C-H absorption at 3000 to 2840 cm^{-1}. Only curve 4 exhibits some absorption here. This film was produced differently by pulsing the RF plasma at 1 kHz and this may have caused some C-H species to deposit in the film. All i-C films made by ethane or butane exhibited spectra similar to curve 2. The film shown in curve 2 was made with p/p = 200 W/torr. Thus, most hydrogen in the i-C films is not infrared active.

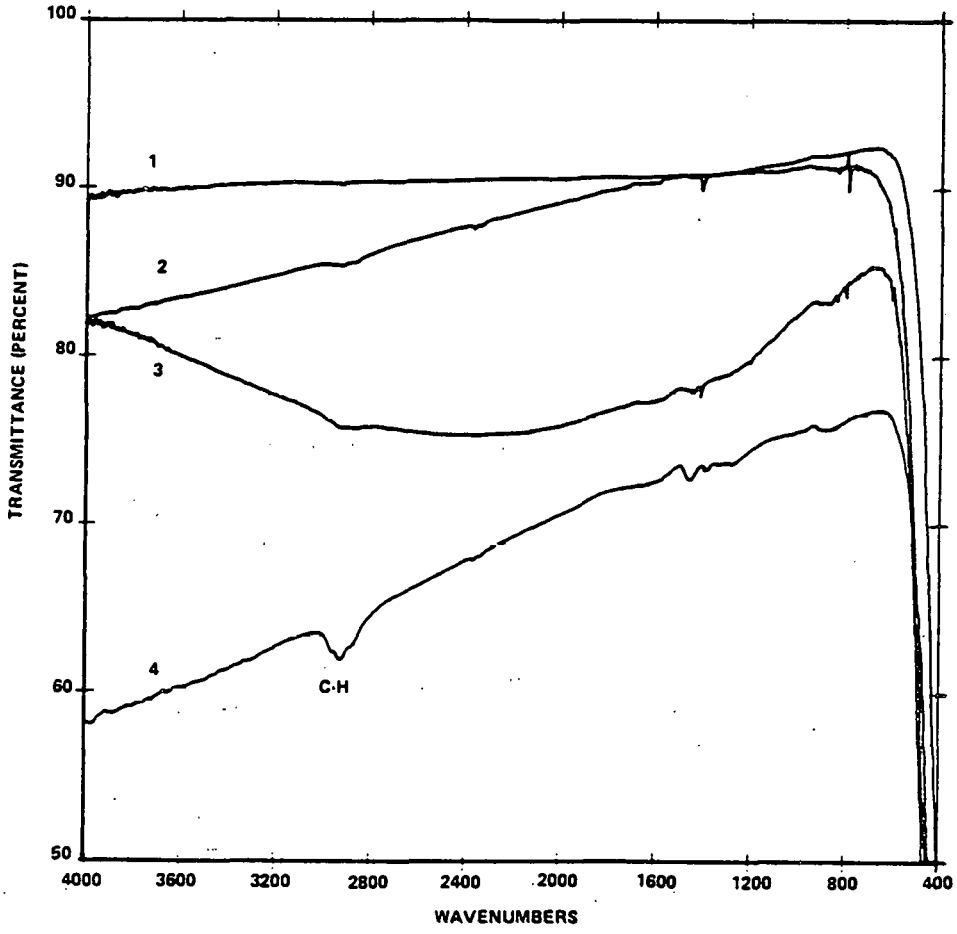


Figure 6. Infrared transmission of (1) uncoated KCl, (2) 0.2μm-thick i-C, (3) 0.45μm-thick i-C, (4) 0.2μm-thick i-C made by pulsing RF at 1-kHz (scale for curve is offset by 15% for clarity).

SIMS was performed on both butane and ethane i-C films deposited on Si wafers. The data were taken at Perkin-Elmer Physical Electronics Industries (Eden Prairie, MN) on a SIMS-II instrument.

Figure 7 illustrates a typical SIMS survey for a film made by RF plasma decomposition of C_2H_6. (SIMS surveys of films made with C_4H_{10} look similar.) The presence of only three elements - carbon, hydrogen and oxygen - is seen in this spectrum. Oxygen and OH are present mainly in the surface and are also generated from the oxygen directed at the surface to increase secondary ion yield. Some of the hydrogen is due to the background of the vacuum system. No impurities greater than several parts per million were detected in the SIMS survey.

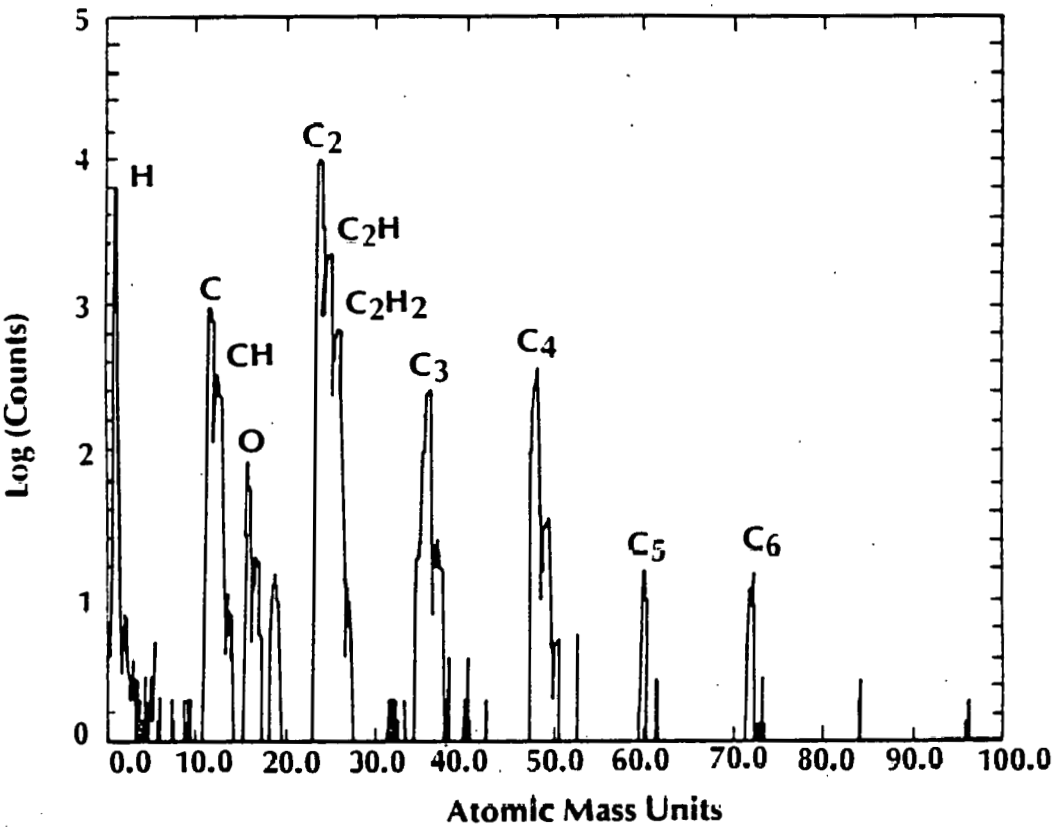


Figure 7. SIMS data for an i-C film prepared by RF plasma decomposition of C_2H_6.

In all the films examined, large fragments of C and H were also detected, i.e., C_2, C_2H_2, C_3, C_3H_3, and very small amounts of C_4, C_4H_4. In Fig. 8, the ratio of C_3 to C_3H_3 molecules detected from the films as they are sputtered away are plotted versus p/p. There appears to be almost a linear

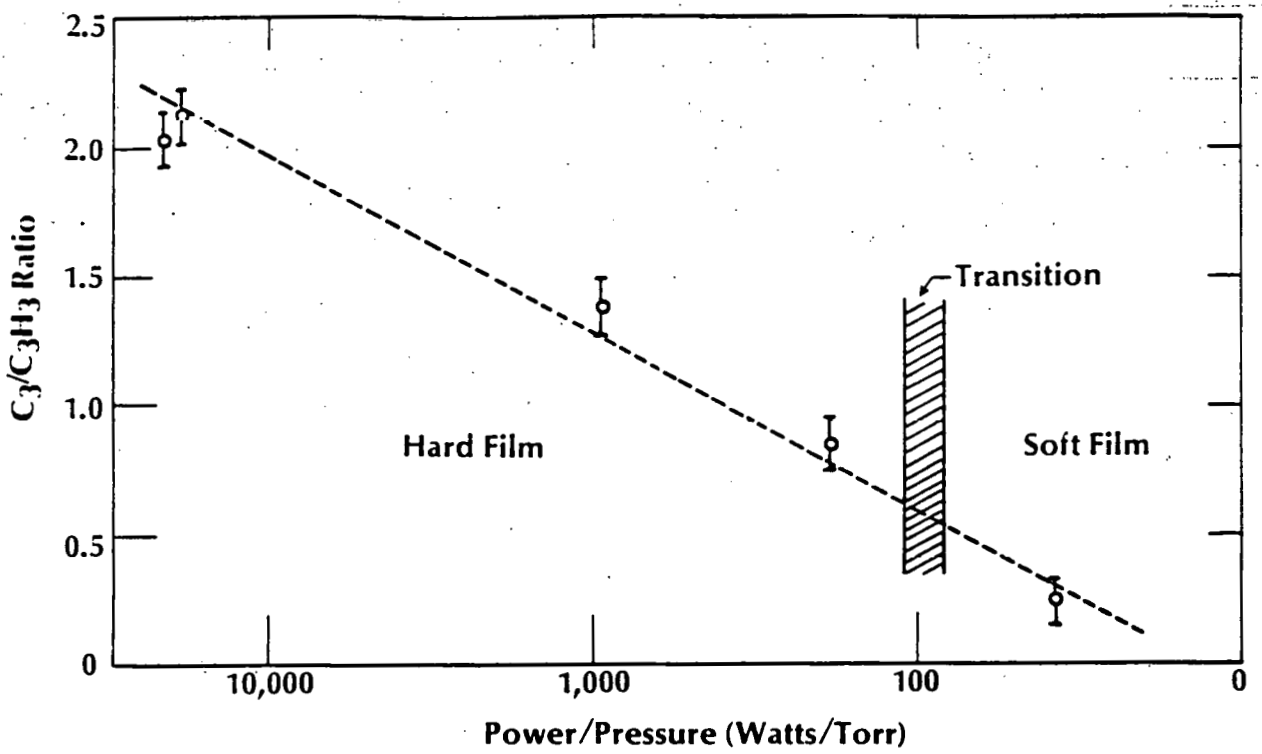


Figure 8. SIMS data of the ratio C_3/C_3H_3 versus power/pressure. Power/pressure scale is logarithmic.

relationship between this ratio and p/p. The error bars arise from data collected at different locations on the substrate. We found a strong transition between a film on glass easily scratched by a razor blade and one that strongly resisted such abrasion near 100 W/torr. This transition was found for all of the alkanes. The soft film is very similar in appearance and low refractive index (n = 1.5 at 0.6μm) to typical hydrocarbon films. The larger the C_3/C_3H_3 ratio, the less hydrogen in the film. Referring to Fig. 8, note that the lower absorbing films have more hydrogen containing species and vice-versa, yet the films remain mechanically hard in this region. The fact that this hydrogen does not form C-H bonds that exhibit infrared absorption is puzzling. Thus, this incorporation of hydrogen in i-C films may be similar to its role in a-Si (amorphous silicon). Since these films were seen to contain a significant amount of hydrogen, it did not appear necessary to confirm this by heating these films in ultra-high vacuum and monitoring the hydrogen by a residual gas analyzer.

SECTION VI
SOLAR CELL AR COATINGS

Low-absorption films produced to date have been with p/p <1000 W/torr for the gases studied. From Fig. 4, this condition results in n ∿ 1.9 to 2.0. This is an ideal value for n for a single-layer quarter-wave AR coating on silicon, since it equals the $\sqrt{n}$ for Si. In Fig. 9, the reflectivity of a 700Å-thick i-C film (p/p = 200 W/torr $-C_2H_6$) on a polished Si wafer is compared with that reported by Kern and Troy[10] (RCA) for sprayed-on TiO_2 single-layer. We believe its low reflectivity below 0.5μm is due more to absorption in the i-C film than AR characteristics.

Figure 10 shows this spectra again with a scale expansion at the AR minimum. The extremely low reflectivity attained at 0.55μm (close to 0.1%) is noteworthy and implies that i-C films made under these conditions are very efficient quarter-wave AR coatings for Si.

Figure 11 compares the spectral quantum efficiency of three AR-coated single-crystal cells, two with i-C films, and one with SiO. The improvement in efficiency for low p/p is apparent. However, neither film is as good as SiO

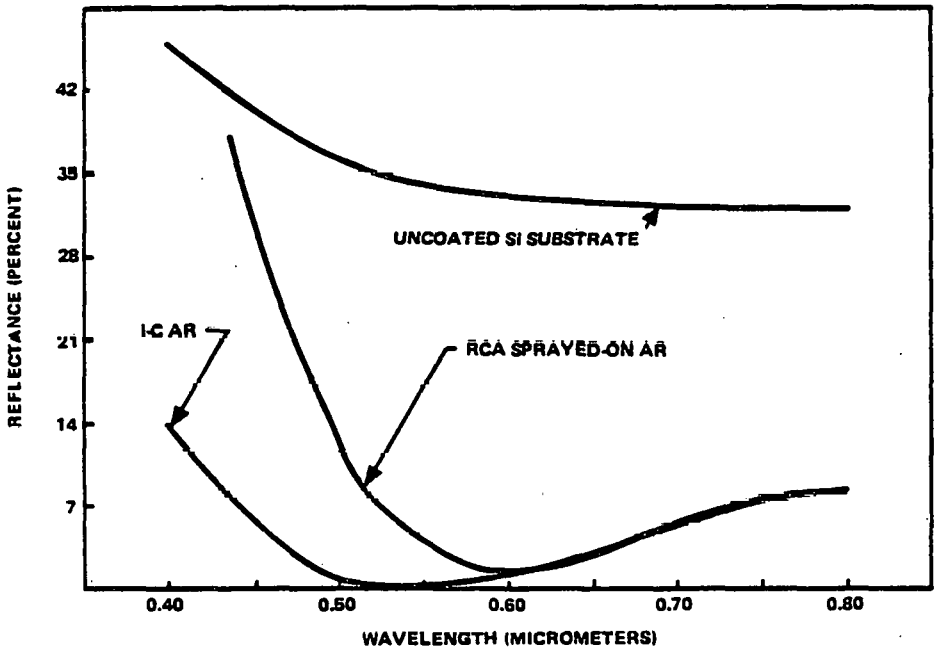


Figure 9. Reflectivity versus wavelength for i-C on Si, RCA sprayed-on AR on Si, and uncoated Si for a i-C film with p/p = 200 W/torr from C_2H_6.

10 Kern, W. and Tracy, E., "Titanium Dioxide Antireflection Coating for Silicon Solar Cells by Spray Deposition," RCA Review 41, 133 (1980).

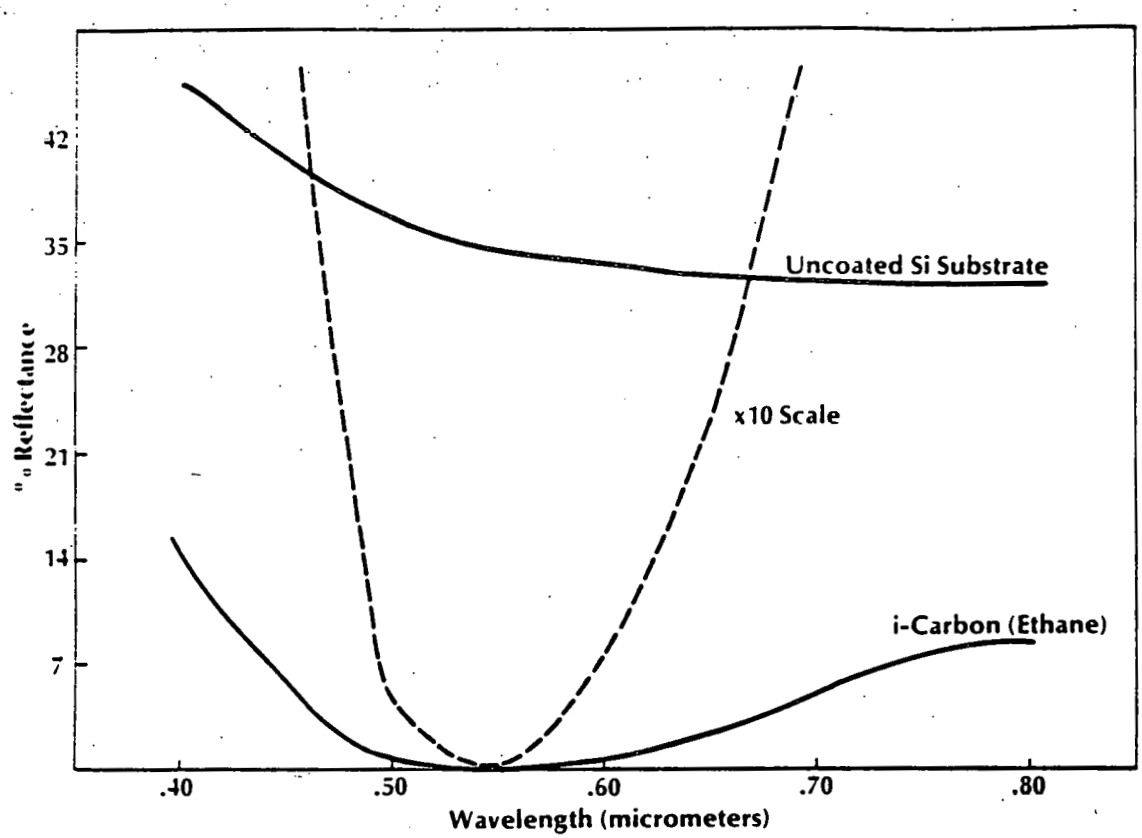


Figure 10. Reflectivity versus wavelength for the same i-C film as shown in Fig. 9.

in the region 0.4 to 0.55μm, which is consistent with the k values shown in Fig. 3.

Table 1 presents the conditions to obtain the best increase in cell efficiency. We believe that the discouraging performance of the ion-beam-deposited film is due mostly to the nonuniformity of the coating and, to a lesser extent, the higher absorption (k value) as compared with the others. The better coatings were made with propane and methane. It is not clear why these two gases should produce better AR coatings on silicon than the others. However, these increases compare very favorably with those obtained with TiO_2/SiO_2 and SiO AR coatings (typically 35 to 40% increase).

Table 2 is a compilation of the AR performance of the cells coated under this program by the gases used. More consistent improvement in efficiency was obtained with C_3H_8 than any other gas. However, CH_4 was studied very little.

Figures 12 through 14 show current-voltage responses for solar cells with better AR coatings made from C_2H_6, C_3H_8, and CH_4, respectively. In general, the response is good, but the AR coating sometimes reduces the fill factor. This may be corrected by further optimization of the deposition process.

Table 1. Conditions to obtain best increase in cell efficiency.

Gas	p/p	n	k	Increase (%)
Butane, C_4H_{10}	200	1.91	0.05	32
Propane, C_3H_8	100	1.72	0.04	38
Ethane, C_2H_6	200	1.98	0.04	31
Methane, CH_4	200	1.94	0.05	40
Graphite target and C_2H_6	a	1.90	0.04	31
Ion beam	b	1.95	0.06	8

a = power split between upper and lower electrode
b = parameters set as in Figure 2.

Table 2. Compilation of increase in efficiency (as defined as J_{sc}) of solar cells AR-coated with i-C films.

Gas	Sample Number	Increase over Uncoated (%)
C_4H_{10}	L006109	6
	L006132	18
	L009154	10
	L009153	31
	L009155A	27
	L009155B	25
C_3H_8	LU102126	32
	LU102123	34
	LV101292	29
	LV101204	35
	LV101203	30
	LV101063	35
	L0112111	39
C_2H_6	L008015	30
	L008043	31
	L009157	21
	L001032	6
	L001036	29
CH_4	L001132	6
	L01223A	32
	L01223B	40

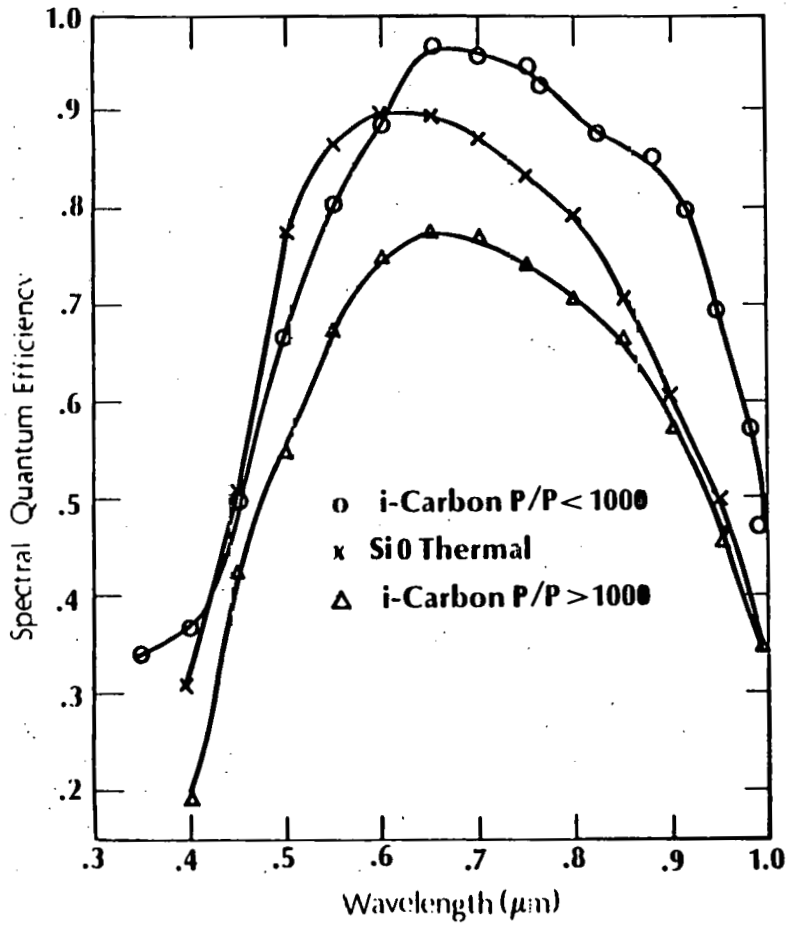


Figure 11. Spectral quantum efficiency versus wavelength for two i-C and an SiO AR-coated single-crystal cell.

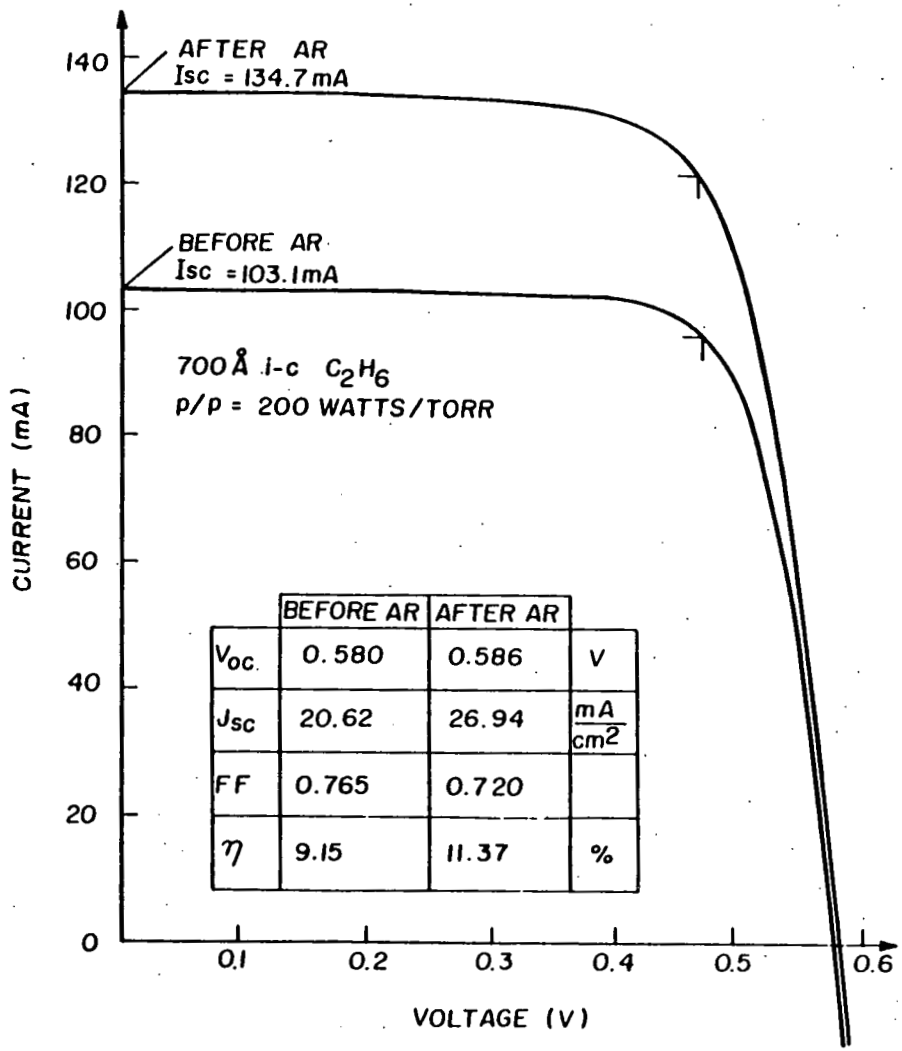
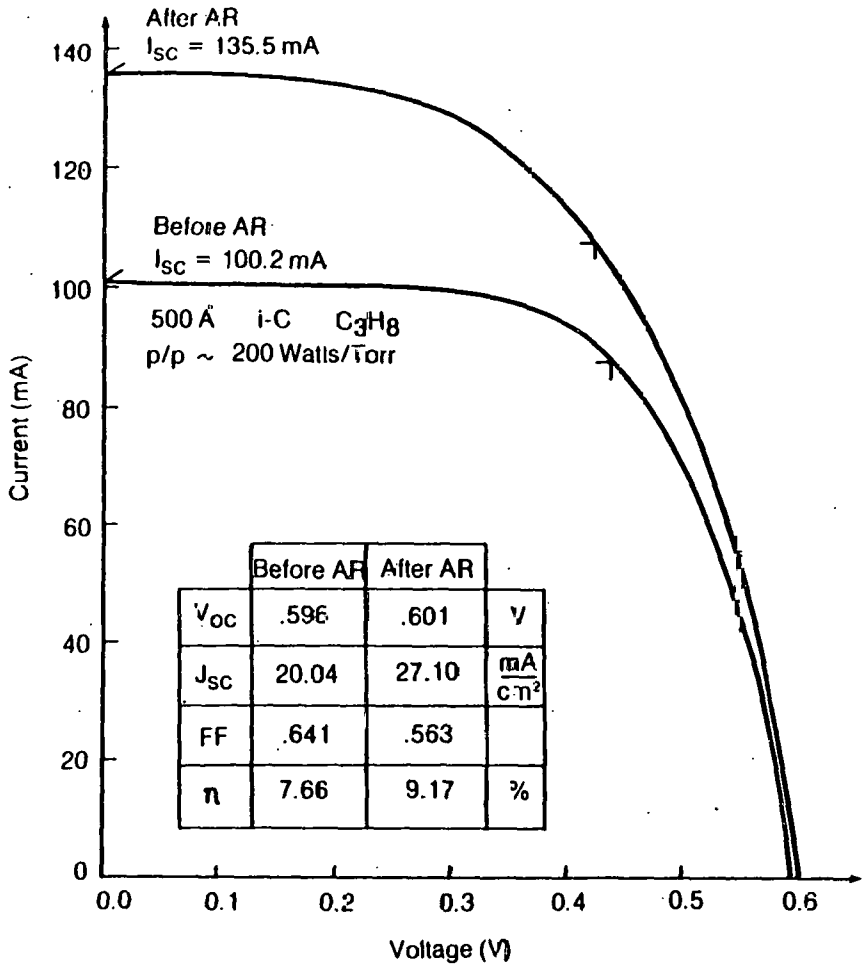
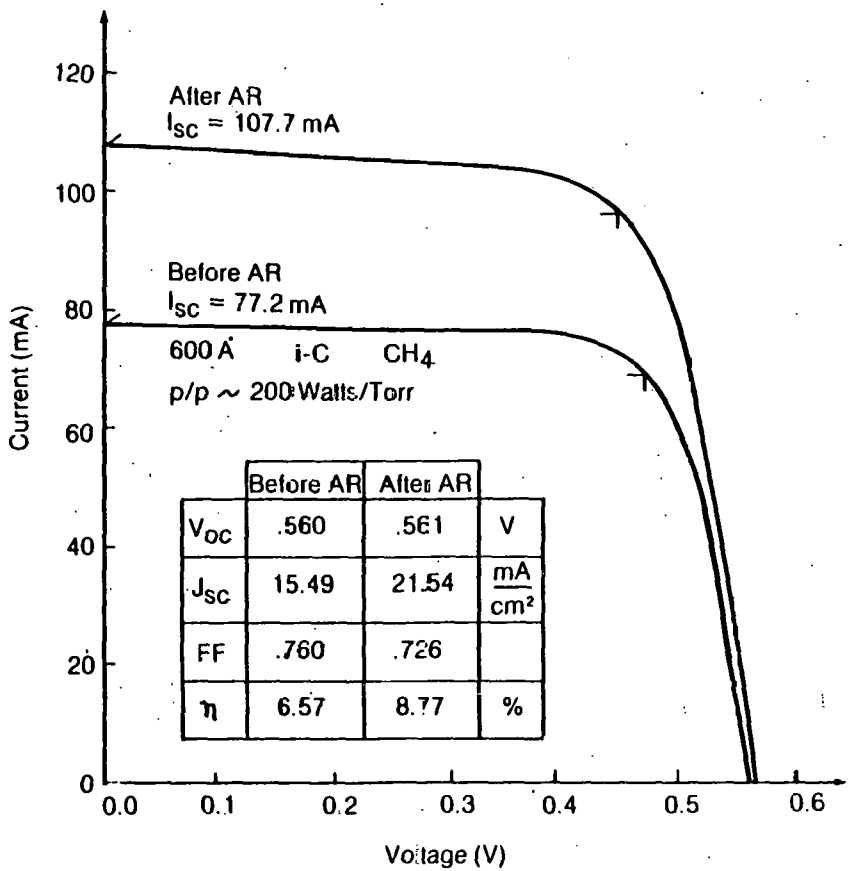


Figure 12. Current-voltage relationship of single-crystal silicon cell before and after AR coating demonstrating 31% increase with AR coating.



	Before AR	After AR	
V_{oc}	.596	.601	V
J_{sc}	20.04	27.10	$\frac{mA}{cm^2}$
FF	.641	.563	
η	7.66	9.17	%

Figure 13. Current-voltage relationship of a single-crystal silicon cell before and after AR coating by i-C C_3H_8 demonstrating a 35% increase with AR coating.



	Before AR	After AR	
V_{oc}	.560	.561	V
J_{sc}	15.49	21.54	$\frac{mA}{cm^2}$
FF	.760	.726	
η	6.57	8.77	%

Figure 14. Current-voltage relationship of a silicon-on-ceramic (SOC) cell before and after AR coating.

SECTION VII
CONCLUSIONS AND RECOMMENDATIONS

We have produced many i-C films from butane, propane, ethane, and methane by plasma decomposition and studied the optical properties versus process parameters. It is possible to vary the absorption k in the films over a broad range of p/p ratios and refractive index n over a narrow range. The lower absorbing films have more hydrogen in them as evidenced by the SIMS data, but this hydrogen is not bonded to the carbon atoms so as to exhibit infrared fundamental absorption. The refractive index at p/p < 1000 W/torr is of the correct value to obtain an efficient layer coating on Si. Solar cells have been coated that have shown an increase of up to 40% in efficiency over uncoated cells.

As part of another program partially funded by Honeywell, transmission electron microscopy was used to investigate the microstructure of the i-C films. This investigation of thin diamond-like carbon films prepared by ion beam and RF plasma techniques indicates that these films are predominantly amorphous. The degree of crystallinity of the films of both types was observed to vary with the deposition parameter in a manner that is not clearly understood at present. Although many optical and mechanical properties of these films are similar to those of diamond,[8] no conclusive evidence of significant formation of cubic diamond in films of either type, or of films of one type being more crystalline than the other, was obtained in this work. The results of this work have been published in the Journal of Applied Physics.[11]

In an effort to increase the degree of crystallinity of these films, they were annealed in ultra-high vacuum at temperatures of up to 600°C. There appeared to be some increase in crystallinity on annealing to 200°C; however, the films remained predominantly amorphous over the range of annealing temperatures used. Electron spectroscopy of these films, employing Auger, ESCA, and electron energy loss techniques, has also revealed that films made by

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11 Vora, H. and Moravec, T.J., "Structural Investigation of Thin Films of i-C (Diamond-like) Carbon," J. Appl. Phys. 52, 615 (1981).

both methods are strikingly similar, and that they resemble amorphous carbon more than they do graphite or diamond.[8]

Attempts to assign the crystalline diffraction patterns of the i-C films to commonly known forms of carbon were unsuccessful. Except possibly for one case, attempts to associate these diffraction patterns to probable contaminants originating from substrates and various components of RF plasma or ion-beam deposition systems were also unsuccessful. All the crystalline diffraction patterns that were obtained from thin i-C films were attributable to cubic phases. In this context, it is of interest to note that single-crystal hexagonal graphite can be converted irreversibly to a cubic phase of lattice parameter 5.45Å by exposing it to high pressure (150 to 200 kbar).[12] This transformation is believed to result from cross-linking of graphite planes.

There is no clear indication in the present work or in other programs as to the cause of the optical absorption in the i-C films, since clear signs of graphite are lacking. The films seem to be composed of a mixture of different phases of carbon (combined with hydrogen in some unknown fashion). The visible optical absorption could be an inherent feature of these new phases. We are very encouraged by the good enhancement of efficiency by using i-C AR coatings. The anticipated advantage in the use of i-C films is the environmental durability of these films. We recommend that a future program be initiated to optimize the deposition process and to study the durability of i-C AR-coated solar modules without further protection. It would be interesting to know the degree to which these coatings provide protection against rain, hail, intense UV, sand, and other environmentally severe conditions. Preliminary qualitative tests show that these coatings are extremely difficult to remove from silicon.

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