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ANISOTROPIC OPTICAL PROPERTIES OF PURE AND DOPED POLYACETYLENE. (U)  
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⑥ Anisotropic Optical Properties of Pure and Doped Polyacetylene

by  
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**Anisotropic Optical Properties of Pure and Doped Polyacetylene\***

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**Abstract**

Optical absorption and reflection measurements have been carried out on pure and doped polyacetylene films. The absorption data are consistent with a model of  $(\text{CH})_x$  as a direct gap quasi-1d semiconductor; doping with  $\text{AsF}_6$  or iodine introduces absorption within the gap, but appears to leave the interband transition intact. The anisotropic reflectance from partially aligned films and the increased optical anisotropy upon doping provide evidence of quasi-1d behavior of the semiconducting pure polymer and the metallic doped polymer.

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\* Work at the University of Pennsylvania supported by the Office of Naval Research.

As a result of the extended  $\pi$ -electron system in long chain polyenes, an extensive literature<sup>1</sup> has developed devoted to the experimental and theoretical study of these model systems. In parallel with this, interest has grown in recent years in the area of quasi-one-dimensional (1d) conductors.<sup>2</sup> Although work in this area<sup>2</sup> focused initially on charge transfer molecular crystals with anisotropic structures, extension to appropriate long chain polymers is of clear importance. Recent studies<sup>3-9</sup> of polyacetylene,  $(\text{CH})_x$ , have provided a point of contact for these two problems. Polyacetylene is the simplest of linear polyenes and provides the physical example of extrapolation to very long (infinite) chain length. The chain-like structure with one  $\pi$ -electron per carbon atom suggests that  $(\text{CH})_x$  may be the first example of a covalent organic polymer in what has grown to become a wide class of quasi-(1d) conductors.

An anisotropic electronic structure is implied by comparison of the intrachain and interchain electronic overlap. The intrachain coupling is determined by the strong  $\pi$ -electron overlap along the chain where the c-c distance of  $\sim 1.4 \text{ \AA}$  leads to a transfer integral  $\beta \approx 2 - 2.5 \text{ eV}$  and an overall bandwidth,  $W$ , of order 8 to 10 eV ( $W = 2z\beta$  in tight binding theory with  $z$  the number of nearest neighbors). In contrast, the interchain distance is approximately  $3.8 \text{ \AA}$ ,<sup>10</sup> even greater than the intermolecular distances in molecular crystals such as TTF-TCNQ<sup>2</sup>, implying a narrow transverse bandwidth with magnitude no greater than a few tenths of an eV.<sup>2</sup>

Extensive dc transport measurements<sup>3-9</sup> have demonstrated that semiconducting  $(\text{CH})_x$  can be chemically doped<sup>3</sup> with donors or acceptors to yield n-type or p-type material,<sup>7</sup> that the two kinds of dopants can compensate one another,<sup>7</sup> and that a p-n junction can be formed.<sup>7</sup> Through careful control of the dopant concentration,<sup>3-8</sup> we have shown that the electrical conductivity can be systematically varied over twelve orders of magnitude<sup>3-8</sup> covering the full range from insulator to semiconductor to metal. Transport and infrared transmission studies indicate a semiconductor-to-metal transition<sup>4</sup> at a dopant concentration near 1%. The room temperature electrical conductivity of partially oriented films of  $[\text{CH}(\text{AsF}_6)_{0.10}]_x$  is in excess of  $2000 (\Omega\text{-cm})^{-1}$  with anisotropy  $\sigma_{||}/\sigma_{\perp} \approx 15$ .<sup>8</sup>

In this paper we report our initial studies of the optical properties of pure and doped polyacetylene.<sup>11, 12</sup> The absorption spectra are consistent with a strong interband transition characteristic of a direct gap semiconductor with a quasi-(1d) density of states. Doping with  $\text{AsF}_6$  or iodine introduces additional absorption in the energy gap, but appears to leave the principal interband transition intact suggesting little change in the basic band structure. Polarized reflection spectra from stretch oriented films reveal significant optical anisotropy. The  $R_{||}$  and  $R_{\perp}$  results ( $||$  and  $\perp$  refer to light polarized parallel and perpendicular to the orientation direction) imply that the interband transition observed in absorption is polarized along the chain axis. Reflectance measurements from stretch-oriented

films heavily doped with  $\text{AsF}_5$  are consistent with quasi-(1d) metallic behavior.

All measurements reported here were carried out with trans- $(\text{CH})_x$  synthesized using techniques described in detail by Ito et al.<sup>10, 13</sup> The stretch orientation was obtained with the procedure developed by Shirakawa and Ikeda.<sup>14</sup>

Visible absorption measurements utilized thin films polymerized on the inner surface of a glass reactor apparatus. The film was kept under vacuum during initial measurements; doping into the metallic range was carried out by subsequently introducing iodine (or  $\text{AsF}_5$ ) vapor. Electron micrographs taken after completion of the optical measurements indicated an approximate film thickness of 0.1  $\mu\text{m}$ . For the reflection studies, pieces of stretched  $(\text{CH})_x$  film ( $l/l_0 = 2.94$ ) with thickness approximately 0.1 mm were mounted (in a dry box) on a glass support, which was then inserted into a glass sample cell for measurement. Large area PbS and Si photodetectors were used (with phase sensitive detection) in a near-normal incidence single beam method. In the middle of the measurement range, the reproducibility of absolute values was about 2% of  $R = 1$ ; relative values were reproducible to about 1% of the measured value. To dope the reflectance samples,  $\text{AsF}_5$  gas was introduced into the sample cell gradually, over two hours; then the excess was pumped off. After completion of the reflectance measurements, leads were



attached and  $\sigma_{dc}$  measured as a check on the doping. All reflectance measurements were taken from the smooth lustrous surface of the films.

For the undoped polymer, the absorption curve (Figure 1) is in qualitative agreement with the earlier results of Shirakawa et al.<sup>15</sup> The absorption edge is quite sharp, rising much more rapidly than the typical three-dimensional (3d) joint density of states, which increases from the gap edge as  $(\mathcal{E} - E_g)^{\frac{1}{2}}$ . In contrast, if we assume weak interchain coupling as argued above, the 1d joint density of states has the well-known  $(\mathcal{E} - E_g)^{-\frac{1}{2}}$  singularity at the gap edge with a correspondingly steep absorption edge. Actual attempts to fit with a Gaussian broadened 1d density of states resulted in good agreement with the data near the gap edge. The magnitude of the absorption maximum ( $\alpha \sim 3 \times 10^5 \text{ cm}^{-1}$ ) is comparable to that for interband transitions in more common direct gap semiconductors.<sup>16</sup>

Since the interband transition in the  $(\text{CH})_x$  polymer is the infinite chain analogue of the lowest  $\pi-\pi^*$  transition of short chain polyenes, it is useful to compare Fig. 1 with the results obtained from the latter.<sup>17</sup> The lineshape of the absorption spectrum is indeed similar to that of other polyenes, and the energy of maximum absorption ( $E_{\text{max}} \approx 1.9 \text{ eV}$  for  $(\text{CH})_x$ ) is in reasonable agreement with extrapolation from the short chain systems.<sup>1</sup> A precise value for the energy gap requires a detailed theoretical model. If we assume a 1d band structure with broadened 1d density of states,  $E_g = E_{\text{max}} = 1.9 \text{ eV}$ . Using the more

conventional definition of the onset of absorption, one estimates

$$E_g \approx 1.4 - 1.6 \text{ eV.}$$

These values are considerably larger than the activation energy obtained from temperature dependent resistivity studies of trans-(CH)<sub>x</sub> (0.3 eV).<sup>18</sup> This is consistent with the transport in undoped (CH)<sub>x</sub> being dominated by trace impurities or defects as discussed earlier.<sup>4</sup>

Ovchinnikov<sup>1</sup> has argued that the energy gap extrapolated to infinite chain polyenes is too large to be accounted for by simple band theory of the bond-alternated chain, and he therefore concluded that Coulomb correlations play an important role. In a tight binding calculation, the band gap due to bond alternation would be  $\delta\beta = \delta x \left. \frac{\partial\beta}{\partial x} \right|_{x_0} = \frac{\delta x}{a} \beta(x_0)$  where  $\delta x$  is the difference in bond lengths and  $x_0$  the average bond length;  $\beta(x) = \beta_0 \exp(-x/a)$  is the transfer integral where  $a$  is a characteristic atomic distance ( $a \sim 0.7 \text{ \AA}$ ) describing the fall-off of the carbon  $2p_\pi$  wavefunction ( $\beta(x_0) \approx 2.5 \text{ eV}$ ). If we assume  $\delta x$  takes the maximum value, equal to the difference in bond lengths between a single bond (1.51  $\text{\AA}$  as in ethane) and a double bond (1.34  $\text{\AA}$  as in ethylene) we estimate  $E_g \approx 0.6 \text{ eV}$ . Certainly this question must be resolved with more detailed band calculations.<sup>19</sup> However, based on results obtained thus far in our transport<sup>3-9</sup> and magnetic<sup>20</sup> studies of (CH)<sub>x</sub>, we see no experimental evidence suggesting strong Coulomb correlations.

The absorption spectra after doping with iodine is shown in Fig. 1; similar results are obtained after doping with AsF<sub>6</sub>. There is relatively strong absorption at low frequencies within the gap as would

be expected for a heavily doped semiconductor. Detailed studies of the onset of absorption within the gap at lightest doping levels are in progress. More important in the context of this study is the observation that the strong interband transition persists even at the highest doping levels. Although this absorption in the doped polymer may be connected with the tri-iodide ion, the nearly identical shape and frequency compared to the undoped polymer suggest that the absorption before and after doping are of common origin. Further, the same absorption is evident in the  $\text{AsF}_6^-$  doped polymer. These data suggest that at least for these dopants the basic  $\pi$ -electron band structure of  $(\text{CH})_x$  remains intact in the doping process, consistent with the charge transfer doping model<sup>6</sup> with  $\text{A}^-$  species between chains and/or on the surface of the polymer fibres. An uninterrupted  $\pi$ -system is consistent with the excellent transport properties of metallic doped  $(\text{CH})_x$ . On the other hand recent studies<sup>21</sup> with bromine doping have revealed a major change in the absorption spectrum upon doping. This may be related to earlier observations<sup>6</sup> that bromine tends to add to the double bond (at least at high concentrations) with a corresponding decrease in conductivity. Studies must be carried out with a variety of dopants before general conclusions can be drawn.

Throughout the above discussion we have assumed that the absorption edge results from an interband transition with the creation of electron-hole pairs. The possibility of electron-hole bound states (excitons) on the chain must be considered since such exciton transitions can lead to a sharp absorption edge below the gap. However, the observation of this absorption even at the

highest doping levels argues against a transition to an exciton bound state, which would be screened in the metal. Moreover early photoconductivity measurements<sup>22</sup> on powder samples suggest a photoconductive edge consistent with the absorption edge.

Direct visual inspection of oriented  $(\text{CH})_x$  films reveals a silvery reflection, similar to Al foil, but somewhat darker. Through a polarizer, the reflection polarized parallel to the fibre and polymer chain orientation direction is silvery, but the reflection polarized perpendicular is pastel orange. Doping with  $\text{AsF}_5$  ( $\sigma_{\parallel} \geq 10^3$   $(\Omega\text{-cm})^{-1}$ ) produced no obvious change by direct vision on parallel polarization, but the reflection polarized perpendicular became much darker. This was stable for a week; the period of observation. The matte reverse side of the film appeared similar but duller, and a slight brassy color (visible with light polarized parallel to the fibres) developed in several days. Independent experiments have shown that wiping or scraping of the duller reverse side reveals the bright silvery polymer surface, and that the brassy color of the doped polymer comes off with similar treatment. We have also observed that if  $\text{AsF}_5$  doping is allowed to proceed too rapidly, the brassy color appears. Carefully controlled conditions which lead to the highest electrical conductivity result in the silvery doped films described above.

The reflectance results are shown in Figures 2 and 3. For the pristine sample,  $R_{\parallel}$  data show a broad maximum that corresponds to the

absorption peak near 2 eV.  $R_{\parallel}$  decreases in the infrared consistent with a semiconductor picture; extrapolation of the low energy data suggests a low frequency reflectance of 12 to 18% implying a dielectric constant,  $\epsilon_{\parallel}(0) \approx 5$ . The perpendicular reflectance is flat ( $\sim 4\%$ ,  $\epsilon_{\perp}(0) \approx 2$ ) at low frequencies, with a weak maximum centered at 1.7 eV. At higher frequencies  $R_{\perp}$  falls proportionately faster than  $R_{\parallel}$  suggesting that the observed structure in  $R_{\perp}$  is intrinsic and not the result of incomplete orientation. The optical anisotropy goes through a minimum of  $(R_{\parallel}/R_{\perp}) = 4.7$  at 1.65 eV, increases to  $(R_{\parallel}/R_{\perp}) \approx 10$  at 2.5 eV, then decreases at higher energy.

Heavy doping of the sample with  $\text{AsF}_6$  ( $\sigma_{\parallel} \geq 10^3$  ( $\Omega\text{-cm}$ ) $^{-1}$ ) increases  $R_{\parallel}$  below 1.4 eV (Fig. 3); the low frequency results are similar to the free carrier reflectance in heavily doped semiconductors.<sup>13</sup> The trans-(CH)<sub>x</sub> maximum near 2 eV remains after doping, consistent with the absorption results described earlier. However any estimate of energy shift or change in strength is difficult due to the onset of the lower energy reflectance. The 1.7 eV peak in  $R_{\perp}$  essentially disappears after doping, and there is a modest increase in reflection at both lower and higher energies. However, the overall result is an increase of the optical anisotropy at all energies below 2.5 eV.

A somewhat more quantitative comparison of the absorption and  $R_{\parallel}$  reflectance of the undoped polymer can be made by modeling the interband transition as a single Lorentz oscillator centered at

Thus, assuming

$$\epsilon(\omega) = 1 + \frac{\Omega_0^2}{\omega_g^2 - \omega^2 - i\omega\Gamma}$$

leads to  $E_g = \hbar\omega_g = 2$  eV,  $\hbar\Omega_0 = 4.0$  eV and  $\hbar\Gamma = 0.54$  eV; the latter two parameters being determined by fitting to  $\epsilon_{||}(\omega)$  and  $R_{||}(2$  eV). From these one estimates a peak absorption at 2 eV of  $\alpha \approx 5 \times 10^5$  cm<sup>-1</sup> in good agreement with Fig. 1. More detailed comparison must await a Kramers-Kronig analysis of the full reflectance spectrum; extension of the reflectance into the far ir is in progress. Note, however, that the implied quantitative agreement between the  $R_{||}$  data from partially aligned films of undoped (CH)<sub>x</sub> (Fig. 2) and the absorption by non-aligned films (Fig. 1) implies that the strong absorption is polarized along the chain direction. We therefore conclude that the anisotropy is intrinsic and is present on a single fibre scale in the non-oriented polymer. The large optical anisotropy is consistent with a quasi-(1d) band structure as described above.

The effect of doping on the reflectance is entirely consistent with the previous interpretation of a metal-semiconductor transition in doped polyacetylene.<sup>4</sup> The interband transition remains visible, but the reflectance begins to rise at lower frequencies due to the free carrier contribution to the dielectric function. Detailed Drude fits require extension of the data into the far ir, where preliminary experiments indicate metallic reflectivity.

In summary, optical absorption and reflection measurements have been carried out on pure and doped polyacetylene films. The absorption data are consistent with a model of  $(\text{CH})_x$  as a direct band gap quasi-(1d) semiconductor; doping with iodine (or  $\text{AsF}_6^-$ ) introduces absorption within the gap, but appears to leave the interband transition intact. The anisotropic reflectance from partially aligned films and the increased optical anisotropy upon doping provide evidence for microscopic anisotropy; i. e., quasi-(1d) behavior, in both the semiconducting state of the pure polymer and the metallic state of the doped polymer.

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Figure Captions:

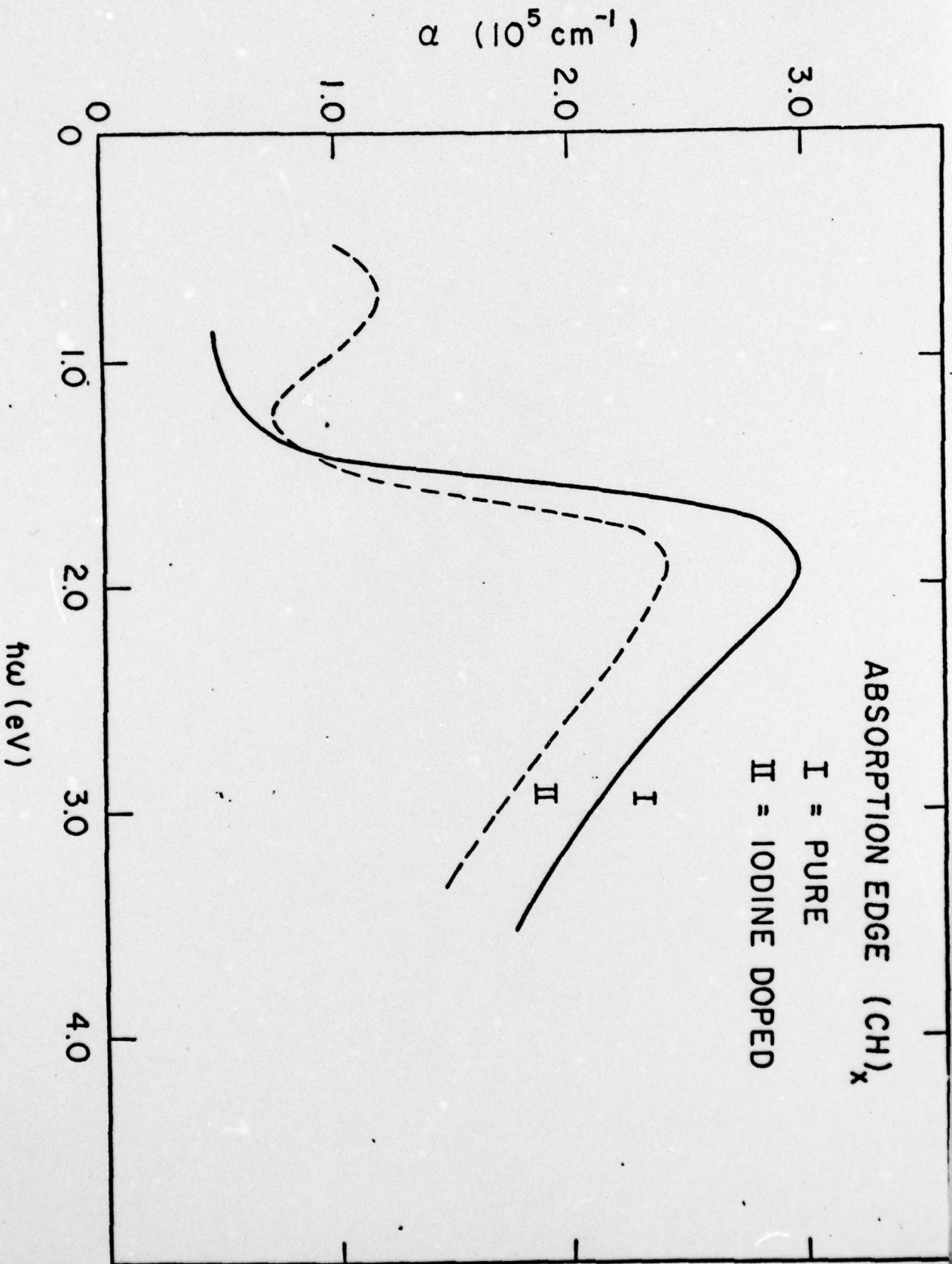
**Figure 1:** Absorption coefficient of trans-(CH)<sub>x</sub> before and after doping to saturation as a function of frequency; film thickness 0.1 μm.

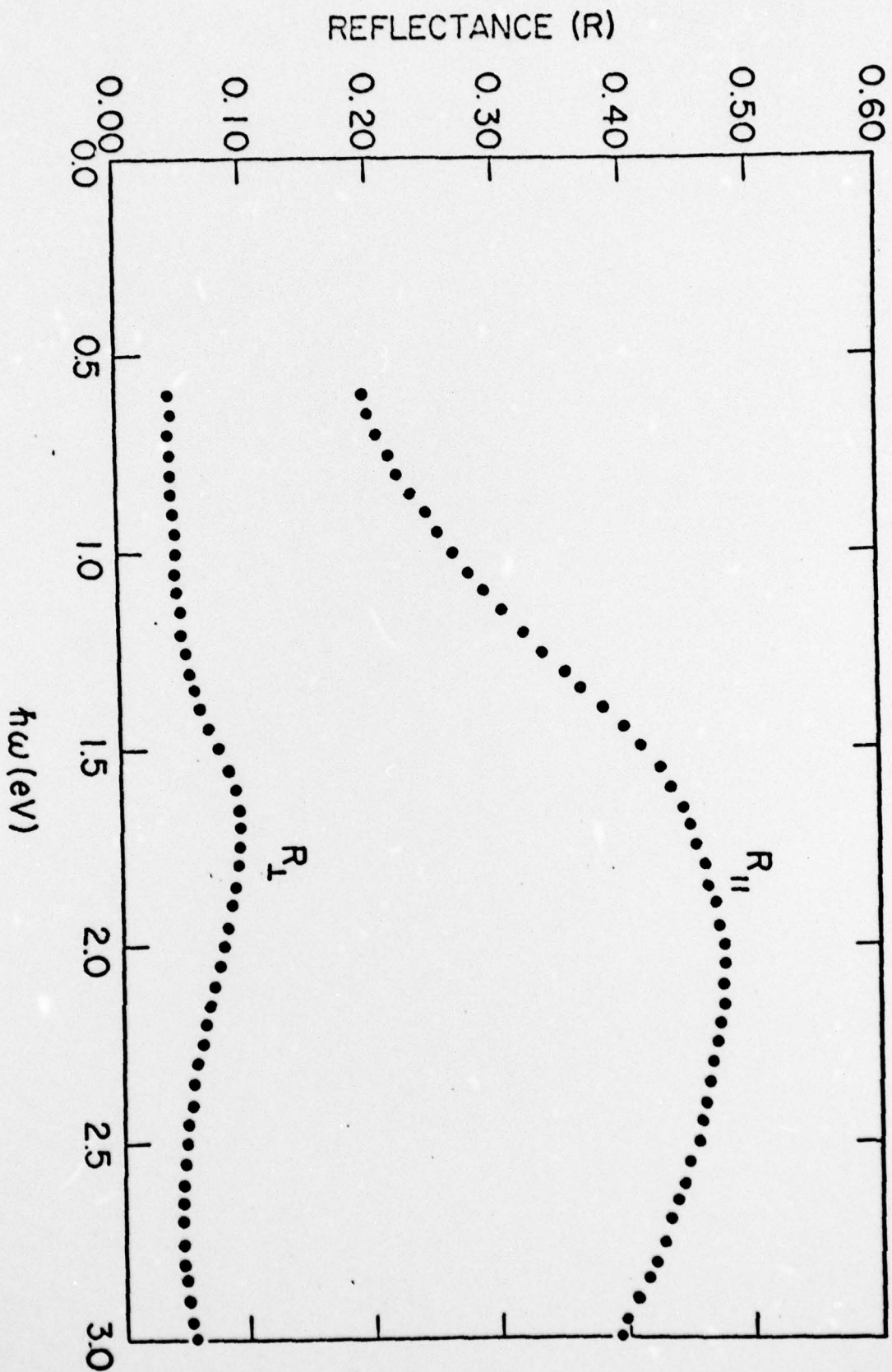
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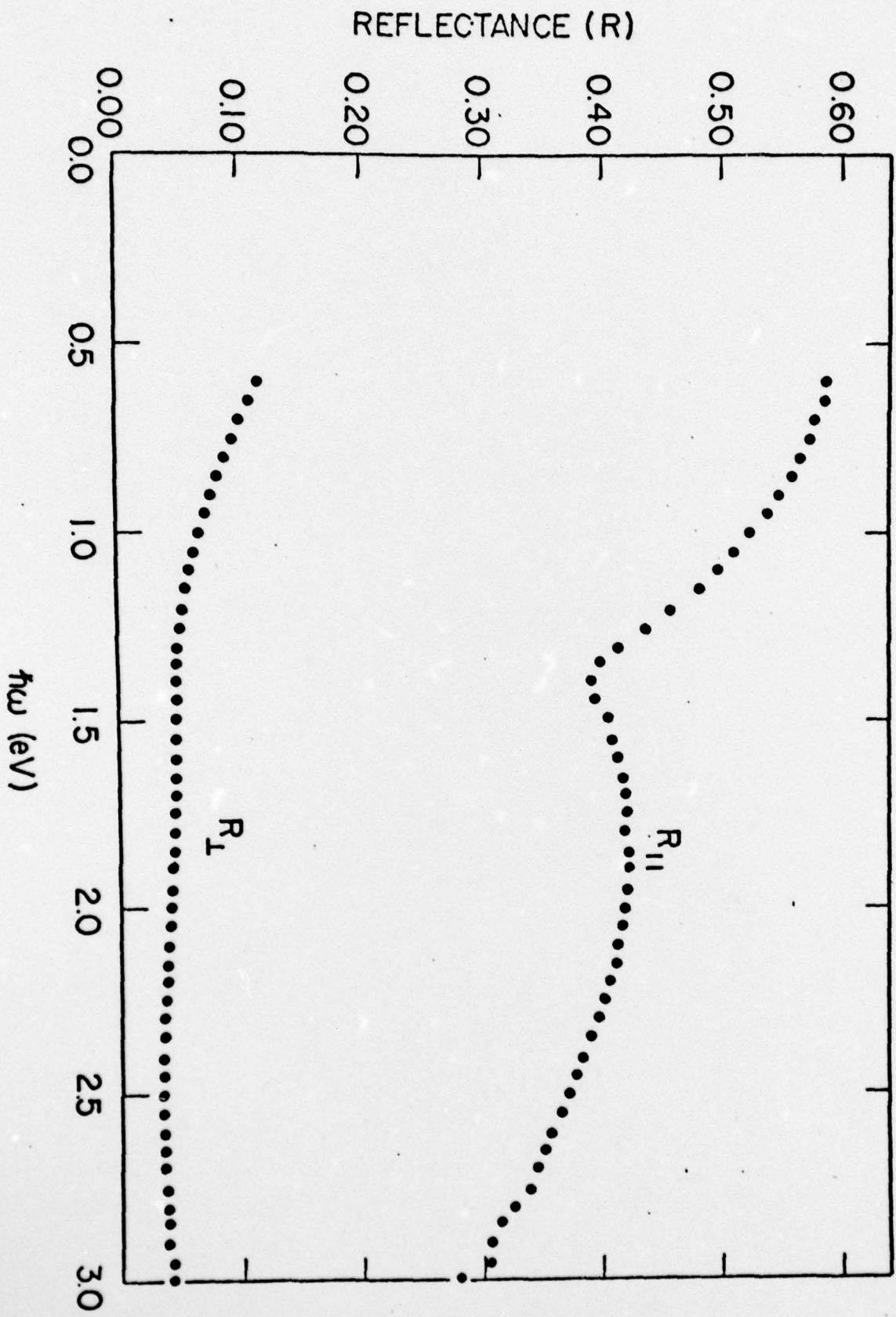
----- doped with iodine (similar results are obtained after doping with AsF<sub>5</sub>)

**Figure 2:** Anisotropic reflectance from partially oriented film of (CH)<sub>x</sub> ( $l/l_0 = 2.94$ ).

**Figure 3:** Anisotropic reflectance from partially oriented film of (CH)<sub>x</sub> ( $l/l_0 = 2.94$ ) after doping with AsF<sub>5</sub> ( $\sigma_{||} = 10^3 (\Omega\text{-cm})^{-1}$ ).







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