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ORGANIC METALS AND SEMICONDUCTORS: POLYACETYLENE, (CH)<sub>x</sub>, AND IT--ETC(U)  
OCT 79 A G MACDIARMID; A J HEEGER N00014-75-C-0962  
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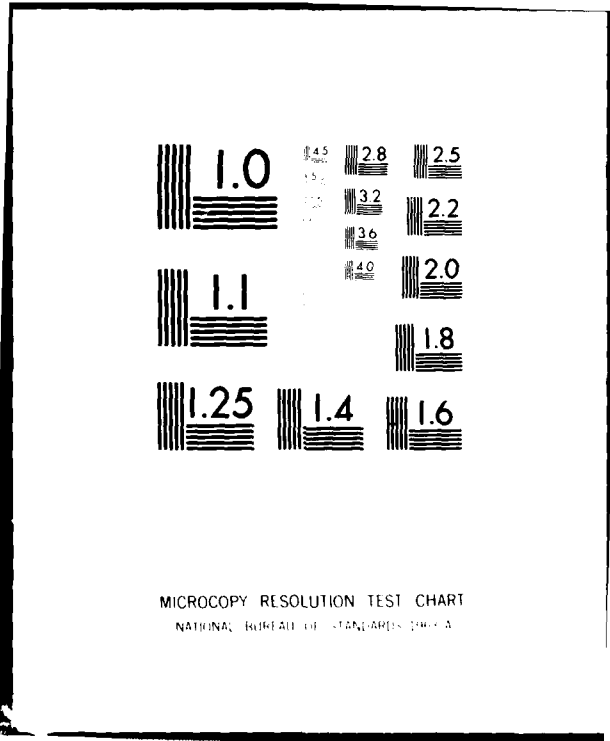
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Polyacetylene; cis-trans content; Ziegler catalyst; cis-trans isomerization; electron microscopy; n-type and p-type dopants; polycarbonium ion; electrical conductivity; semiconductor-metal transition; stretch oriented (CH)<sub>x</sub>; p-n junctions; Schottky barrier junctions; thermoelectric power measurements; photoelectron spectroscopy; photovoltaic effect

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  
Polyacetylene, (CH)<sub>x</sub>, is the simplest organic polymer. Both the cis- and trans- forms may be prepared<sup>x</sup> as silvery, flexible, polycrystalline, semiconducting films. The cis-films can be stretched over three times their original length with partial alignment of the (CH)<sub>x</sub> fibrils. Through chemical or electrochemical doping, the electrical conductivity of the films can be increased over twelve orders of magnitude with properties ranging from insulator ( $\sigma < 10^{-10}$  ohm<sup>-1</sup>cm<sup>-1</sup>), to semiconductor to metal ( $\sigma > 10^3$  ohm<sup>-1</sup>cm<sup>-1</sup>). By the use of donors or acceptors n-type or p-type polymer, respectively, is produced. When doped, the partly aligned (CH)<sub>x</sub> films

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20. show marked anisotropic electrical and optical properties. Studies have shown that rectifying p-n junctions as well as Schottky barrier junctions can readily be fabricated from  $(CH)_x$ .

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<sup>6</sup> Organic Metals and Semiconductors:  
Polyacetylene,  $(CH)_x$ , and its Derivatives •

by

<sup>10</sup> Alan C. MacDiarmid and Alan J. Heeger

Prepared for publication in Organic Coatings and  
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ORGANIC METALS AND SEMICONDUCTORS:  
POLYACETYLENE, (CH)<sub>n</sub>, AND ITS DERIVATIVES

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Polyacetylene, (CH)<sub>n</sub> is the simplest possible conjugated organic polymer and is therefore of special fundamental interest. In a series of studies, Shirakawa *et al.* (1-4) succeeded in synthesizing high quality polycrystalline films of (CH)<sub>n</sub> and developed techniques for controlling the cis-trans content. (Figure 1).

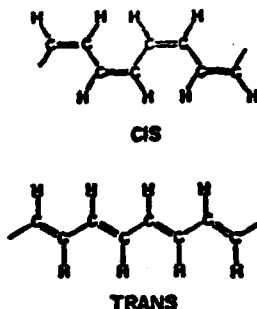


Figure 1: Cis- and trans- polyacetylene, (CH)<sub>n</sub>

The (CH)<sub>n</sub> films have a lustrous silvery appearance; they are flexible and appear to have excellent mechanical properties. Films can be made free standing, or on substrates such as glass or metal. Films have been made with thickness varying from 10<sup>-5</sup> cm to 0.5 cm.

(1) SYNTHESIS OF (CH)<sub>n</sub> FILMS

Polyacetylene films may be prepared by simply wetting the inside walls of a glass reactor vessel with a toluene solution of (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Al and (n-C<sub>4</sub>H<sub>9</sub>O)<sub>2</sub>Ti Ziegler catalyst and then immediately admitting acetylene gas at any pressure from a few centimeters up to ca 1 atmosphere pressure. The cohesive film grows during a few seconds to 1 hour, depending on the pressure of acetylene and temperature employed, on all surfaces which have been wet by the catalyst solution. If a polymerization temperature of ca-78°C is used, the film is formed almost completely as the cis-isomer; if a temperature of 150°C is used (decane solvent) the film is formed as the trans-isomer. With room temperature polymerization the film is approximately 80% cis- and 20% trans-isomer. The cis-isomer may be conveniently converted to the trans-isomer (the thermodynamically stable form) by heating at ca 200° for ca 1 hour. (1-4). Dark red gels of (CH)<sub>n</sub> (5) may be prepared using lower catalyst concentrations. These may be freeze-dried to yield highly porous, very low density, "foam-like" (CH)<sub>n</sub> (5).

## (2) STRUCTURAL PROPERTIES OF (CH)<sub>x</sub> FILMS

Electron microscopy studies show that the as formed (CH)<sub>x</sub> films consist of randomly oriented fibrils (typical fibril diameter ca 200Å). The bulk density is ca 0.4 gm/cm<sup>3</sup> compared with 1.2 gm/cm<sup>3</sup> as obtained by flotation techniques. This shows that the polymer fibrils fill only about one third of the total volume. X-ray studies show that the films are polycrystalline with interchain spacing of approximately 3.8Å (1-4).

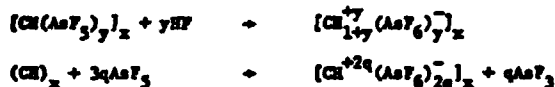
## (3) DOPING OF (CH)<sub>x</sub> FILMS

When either *cis* or *trans* (CH)<sub>x</sub> films are exposed to electron attracting substances such as Br<sub>2</sub>, I<sub>2</sub>, AsF<sub>5</sub>, H<sub>2</sub>SO<sub>4</sub>, HClO<sub>4</sub>, etc. vapor, they become "doped" with the species and their electrical and optical properties change markedly (See Table I) (6,7). They may also be readily doped electrochemically to yield compositions such as [CHI<sub>0.07</sub>]<sub>x</sub>, [CH(ClO<sub>4</sub>)<sub>0.0645</sub>]<sub>x</sub>, [CH(AsF<sub>6</sub>)<sub>0.077</sub>]<sub>x</sub>, etc. (8). Salts containing the (NO)<sup>+</sup> or (NO<sub>2</sub>)<sup>+</sup> ions, such as (NO<sub>2</sub>)<sup>+</sup>(SbF<sub>6</sub>)<sup>-</sup>, also act as good dopants (7) e.g.



All these dopants are electron-attracting species and may be considered as "p-type" dopants.

Most of the physical studies on doped (CH)<sub>x</sub> films have been carried out using I<sub>2</sub> or AsF<sub>5</sub> as dopants. Raman studies have shown that the iodine is present as the I<sub>3</sub><sup>-</sup> ion (9,10). This implies that the (CH)<sub>x</sub> chain must therefore exist as a polycarbonium ion. The nature of the AsF<sub>5</sub> species is still somewhat uncertain. Elemental analyses (Table 2) (6,11) have given different results, possibly because of slight differences in doping procedure or purity of the AsF<sub>5</sub>, which frequently contains HF. If the dopant species contains arsenic and fluorine in the ratio of 1:5, then it may be present as the diamagnetic (As<sub>2</sub>F<sub>10</sub>)<sup>-2</sup> ion (12). If it contains the elements in the ratio of 1:6, then it is presumably present as the (AsF<sub>6</sub>)<sup>-</sup> ion. Photoelectron spectroscopy (12) shows the principal species has the empirical formula AsF<sub>6</sub>, whereas X-ray absorption (13) and I/R data (13) indicate the presence of the (AsF<sub>6</sub>)<sup>-</sup> ion. The latter ion could arise through one (or both) of the two possible reactions below:



Electron-donating, i.e. "n-type" dopants, may also be introduced into (CH)<sub>x</sub> films (6,14) (See Table I) simply by immersing the film in a THF solution of e.g. sodium naphthalide, viz.



The (CH)<sub>x</sub> chain in these materials may be considered as a polycarbonium ion.

## (4) ELECTRICAL CONDUCTIVITY OF DOPED (CH)<sub>x</sub> FILMS AND SEMICONDUCTOR-METAL TRANSITION

When pure polyacetylene is doped with a donor or an acceptor, the electrical conductivity increases sharply over many orders of magnitude at low concentration, then saturates at higher dopant levels, above approximately 1%

(6,14-19). The maximum conductivity we have reported to date at room temperature for non-aligned  $\text{cis}-(\text{CH}(\text{AsF}_6))_{0.10}$  is  $1200 \text{ ohm}^{-1} \text{ cm}^{-1}$ . Typical behavior for the conductivity as a function of dopant concentration ( $\gamma$ ) is shown in Figure 2. These conductivity studies suggest a change in behavior near 1% dopant concentration; a semiconductor to metal transition.

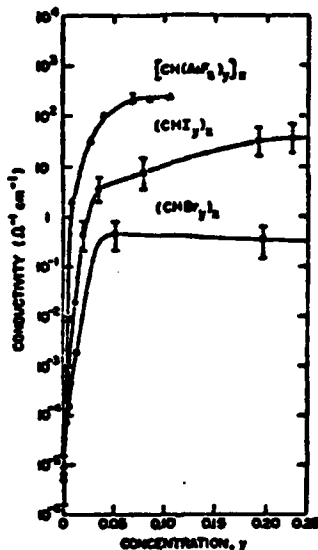


Figure 2: Electrical conductivity (room temperature) as a function of dopant concentration.

To verify the existence of the semiconductor-to-metal transition, far infrared transmission data were taken (16) on samples of varying concentrations of iodine and  $\text{AsF}_6$  (with qualitatively similar results). In the case of the 6% sample, there is no observable transmission throughout the I/R down to  $20 \text{ cm}^{-1}$ . This is consistent with metallic-type behavior. Thermoelectric power measurements (20) and conduction activation energies (17) also show the semiconductor-metal transition very clearly.

#### (5) PARTLY ORIENTED FILMS: ANISOTROPIC ELECTRICAL AND OPTICAL PROPERTIES

$\text{cis}$ -rich  $(\text{CH})_x$  films may be stretched to  $\approx 3$ -4 times their original length. This causes partial orientation of the  $(\text{CH})_x$  fibrils which results in a large increase in conductivity in the direction parallel to the stretch direction and a reduction in conductivity in the direction perpendicular to stretching (18). For example, conductivities in excess of  $2000 \text{ ohm}^{-1} \text{ cm}^{-1}$  have been observed with  $\text{AsF}_6$ -doped film (18). Optical studies using polarized light show marked anisotropic behavior of both the doped and undoped  $(\text{CH})_x$  films (19).

#### (6) SEMICONDUCTOR PHYSICS OF $(\text{CH})_x$ FILMS

A series of studies have been carried out which clearly demonstrate that rectifying p-n junctions as well as Schottky barrier junctions can readily be

fabricated from (CH)<sub>x</sub> (14,21). In addition, recent studies show that a good photo-voltaic effect may be observed, when, for example, parent (p-type) (CH)<sub>x</sub> is in contact with a crystal of (n-type) ZnS (21). Such systems may have potential use in low-cost terrestrial solar energy production.

(7) CONCLUSIONS *THE ILLUSTRATED*

As can be seen from Figure 3, which contains a list of conductivities of common substances, (CH)<sub>x</sub> is quite remarkable in that its conductivity can be readily modified to span an extraordinarily large range. Considering possible polyacetylene derivatives, replacement of some or all of the hydrogen atoms in (CH)<sub>x</sub> with organic or inorganic groups, copolymerization of acetylene with other acetylenes or olefins, and the use of different dopants should lead to the development of a large new class of conducting organic polymers with electrical properties that can be controlled over the full range from insulator to semiconductor to metal.

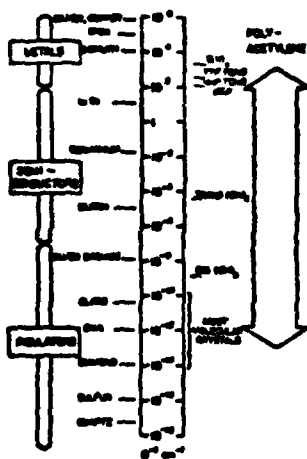


FIGURE 3: List of conductivities of common substances

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TABLE I  
DOPANTS FOR  $(CH)_x$

	Conductivity ( $\text{ohm}^{-1} \text{cm}^{-1}$ ) 25°C)
cis- $(CH)_x$	$1.7 \times 10^{-9}$
trans- $(CH)_x$	$4.4 \times 10^{-5}$
A. p-type (electron-attracting dopants)	
trans- $[(CH)(HBr)_{0.04}]_x$	$7 \times 10^{-4}$
trans- $[(CH)(Cl)_{0.02}]_x$	$1 \times 10^{-4}$
trans- $[(CH)(Br)_{0.05}]_x$	$3 \times 10^{-1}$
trans- $[(CH)(Br)_{0.23}]_x$	$4 \times 10^{-1}$
cis- $[(CH)(Cl)_{0.14}]_x$	$5 \times 10^1$
cis- $[(CH)_{0.30}]_x$	$3.5 \times 10^2$
trans- $[(CH)_{0.22}]_x$	$3.0 \times 10^1$
trans- $[(CH)_{0.20}]_x$	$1.6 \times 10^2$
cis- $[(CH)(I)_{0.15}]_x$	$4.0 \times 10^2$
trans- $[(CH)(AsF_3)_{0.03}]_x$	$7 \times 10^1$
trans- $[(CH)(AsF_3)_{0.10}]_x$	$4.0 \times 10^2$
cis- $[(CH)(AsF_3)_{0.10}]_x$	$1.2 \times 10^3$

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TABLE I (CONTINUED)

cis-[CH(H <sub>2</sub> SO <sub>4</sub> ) <sub>0.106</sub> (H <sub>2</sub> O) <sub>0.070</sub> ] <sub>x</sub>	1.2 x 10 <sup>3</sup>
cis-[CH(HClO <sub>4</sub> ) <sub>0.127</sub> (H <sub>2</sub> O) <sub>0.297</sub> ] <sub>x</sub>	1.2 x 10 <sup>3</sup>
cis-[CH(SbF <sub>6</sub> ) <sub>0.050</sub> ] <sub>x</sub>	4.0 x 10 <sup>2</sup>
cis-[CH(ClO <sub>4</sub> ) <sub>0.0645</sub> ] <sub>x</sub>	9.7 x 10 <sup>2</sup>
cis-[CH(AsF <sub>6</sub> ) <sub>0.077</sub> ] <sub>x</sub>	2.0 x 10 <sup>2</sup>
B. n-type (electron-donating dopants)	
cis-[Li <sub>0.30</sub> (CH)] <sub>x</sub>	2.0 x 10 <sup>2</sup>
cis-[Na <sub>0.21</sub> (CH)] <sub>x</sub>	25
cis-[K <sub>0.16</sub> (CH)] <sub>x</sub>	50
trans-[Na <sub>0.28</sub> (CH)] <sub>x</sub>	80

\*"cis" or "trans" refers to the principal isomeric composition before doping

TABLE II  
ANALYSES OF AsF<sub>6</sub>-DOPED (CH)<sub>x</sub> FILMS

	% C	% H	% As	% F	Total
Calc. for [CH(AsF <sub>6</sub> ) <sub>0.1</sub> ] <sub>x</sub>	37.64	3.17	23.48	35.71	100.00
Calc. for [CH <sub>1.1</sub> (AsF <sub>6</sub> ) <sub>0.1</sub> ] <sub>x</sub>	37.53	3.46	23.40	35.61	100.00
Found	37.67	3.69	23.73	35.81	100.90
Calc. for [CH(AsF <sub>3</sub> ) <sub>0.099</sub> ] <sub>x</sub>	40.15	3.61	24.80	31.44	100.00
Found	39.86	3.75	24.69	31.48	99.78

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