SOME STUDIES ON THE PLASMA POLYMERIZATION OF ETHANE IN THE GAS PHASE

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ABSTRACT

Plasma polymer of ethane prepared by use of 13.56 MHz. r.f. in tubular reactor is examined. Tube pressure and power levels are found to effect rate of deposition inversely or linearly, respectively; at 40 cc.STP per min. flow rate. Products obtained at various locations in the reactor are found to have some differences in structure for some runs, and some characteristics of products are summarized.

1. INTRODUCTION

It is known that in a glow discharge (or plasma) produced in the gas phase, the electrode surfaces and molecules are bombarded with high energy ions and electrons which usually leads to polymeric products. A variety of compounds, including unusual saturated (1,2), unsaturated (3,4) and organometallic compounds have allready been polymerized by plasma to a thin, uniform film or liquid and powder products.

In this communication, some results on gas phase plasma polymerization of ethane will be presented, which are supplementary to those presented before (1).

2. EXPERIMENTAL

Ethane is plasma polymerized in a plug-flow type reactor by application of r.f. power at 13.56 MHz. The reactor used contained inner electrodes which are capacitively coupled to r.f. to form a gas flow with uniform cross-section, teflon inserts are placed before and after the electrodes. Further details of the reactor and system is presented before (1).

Types of polymeric products as well as their deposition rates are known to be effected by experimental conditions (3,6). To help to simplify the complex nature of experiments, all polymerization parameters except three (r.f. power applied; monomer flow rate and reactor pressure) are fixed.

Either surface cleaned aluminium foil or NaCl optic crystals, both placed on cooled electrode; are used to follow rate of deposition and during I.R. studies, respectively.

C.P. Grade Ethane (Mattheson Gas Co. product) is used as received.

3. RESULTS

Characteristic map for plasma polymerization of ethane in tubular reactor is presented before (1). Although much higher power levels are employed in this work, a similar characteristic map is obtained as in previous publications (1,5); which is not presented here.

The amounts of film plasma polymer (p.p.) ethane deposited per unit substrate area versus plasma durations -at different reactor pressures and at a fixed monomer flow rate- are presented in the first Figure. As seen in the figure, either the increase of tube pressure or decrease of power levels decreases amount of polymer deposited (as well as rate of deposition). Both of these effects are somewhat expected and explainable (2,b; 3,a).

I.R. characteristics of polymers obtained at different locations in the reactor is of interest to check the chemical homogeinity of the product, which showed several differences. This group of studies are made by placing optic crystals equispaced one next to the other, on teflon insert next to the entrance (before plasma) $-T_1,T_2-$ and on cooled electrode surface $-E_1,E_2,E_3-$ with the smallest number corresponding to the one closest to monomer entrance. Results are shown in Figure 2 for (four) different plasma conditions. All of the products examined are film, except the last one (D) in the figure, which clearly shows that the product obtained may show same variations with the location. The p.p. ethane produced by different plasma conditions at the same location on electrode(s) also show same differences, as summarized in the following table:

Table- Some characteristics of p.p. ethane.

Reaction Cond.	Phys.State of p.p.	Product(H/C) mole/mole cooled electrode	hot electrode
100 watt, 1 torr, 3.5 cc.STP/min	film	1.407	-
100 watt, 3 torr, 6 cc.STP/min.	film	1.135	-
100 watt, 2 torr, 3.54 cc.STP/min.	film	1.573	-
100 watt, 2 torr 20.1 cc.STP/min.	film	1.857	-
50 watt, 2 torr, 60.5 cc.STP/min.	film	1.737	1.979
75 watt, 2 torr 61 cc.STP/min.	film	1.581	1.896
100 watt, 0.5 torr, 2.1 cc.STP/min.	powder	1.014	1.033
100 watt, 1 torr, 19.6 cc.STP/min.	powder+film	1.326	-
ethane gas, as reference (monomer)		3.00	-

Some aging characteristics of these films will also be included in the discussion.

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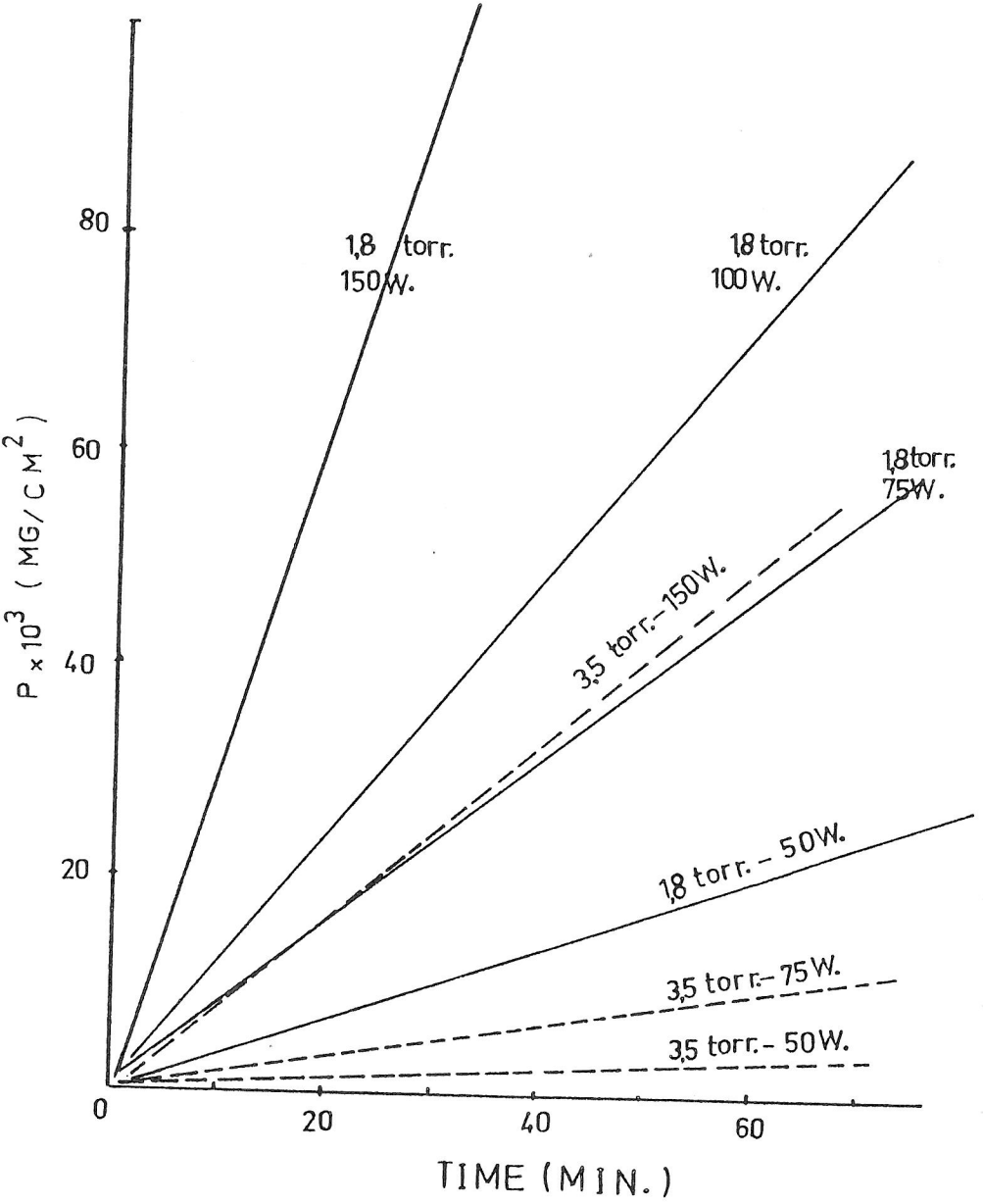

Figure.1- Deposition v.s. plasma duration at a fixed ethane feed rate of 40 cc.STP/min, at various pressure and power levels.

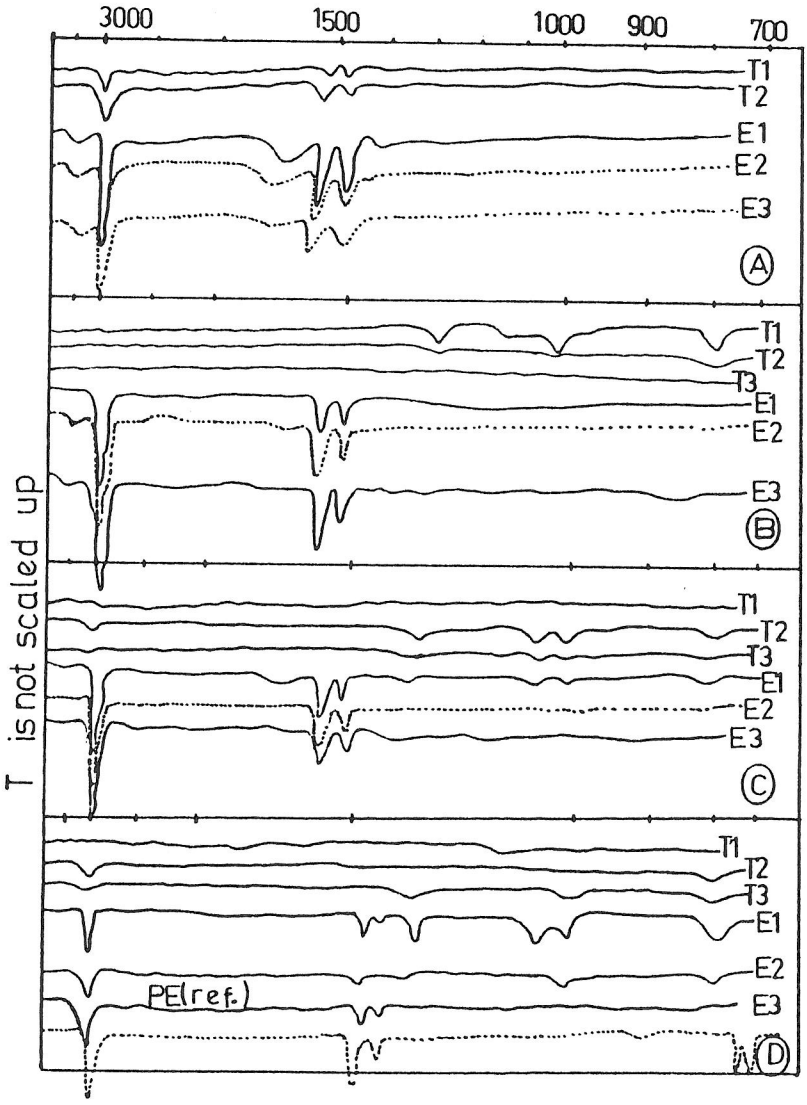

Figure.2- I.R. of various p.p.Ethane samples collected from various parts of reactor: T_1,T_2(on teflon insert) and E_1,E_2, E_3 (on electrode surface); through optic crystals. (A)100 watt, 1 torr, 3.5 cc.STP/min, film; (B) 100 watt, 3 torr, 6 cc.STP/min. film: (C) 100 watt, 2 torr, 20 cc.STP/min, film; (D) 100 watt, 1 torr, 19.6 cc.STP/min, powder. Monomer ethane flow rate: 40 cc.STP/min. for all examined.