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A CHROMATOGRAPHIC ANALYSIS OF THE
RESPONSE OF POLYMERIC FIRE-DETECTION
DEVICES TO COMBUSTION PRODUCTS

Stephen D. Senturia

FINAL REPORT

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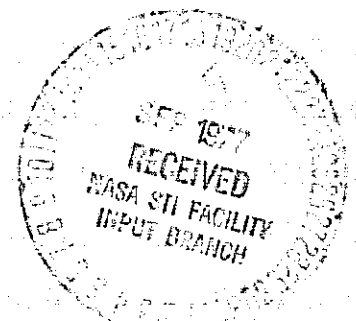


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I. SUMMARY

This grant had as its goals the use of gas-chromatography/IR spectroscopy to correlate the spectrum of combustion products produced by smouldering samples with the electrical responses of polymeric early-warning fire detection devices, and to make improvements in device design and performance.

The most significant success was in the device area, in which successful fabrication of charge-flow transistor structures, similar to what was originally proposed, made possible a realistic, low-cost, miniaturized sensor design. These devices work very well, and have attracted widespread industry interest.

Also successful was improved documentation of polymer responses to a variety of smouldering sources, including cellulose, acrylic, urethane, poly (vinyl chloride), and wool. The polymers selected for application as fire detectors respond well to all of these materials.

Much less successful was the chromatography part of the program, first, because the planned use of on-line infrared spectroscopy using a Digilab GC/IR interface to a Digilab FTS-14 spectrometer turned out to be unfeasible (primarily because of defects in the interface), and secondly, because of severe technical problems encountered in sampling the combustion materials.

Because of the relative success of device work relative to the chromatography work, the effort during the grant period increasingly shifted over to the device portion of the program. This work was the Master's thesis responsibility of Mr. Carl Sechen. His thesis, which as a separate document will provide the major documentation of results under this grant, also has led to the publication of one paper and the presentation of two additional

papers at recent meetings.

Work under this grant has also lead to discoveries about the charge-flow transistor device for which patents are being sought

II. RESEARCH REPORT

A. Device Research

The device research aspects of this program were carried out primarily by Mr. Carl M. Sechen, a Graduate Research Assistant in the Department of Electrical Engineering and Computer Science, under the supervision of Professor Senturia, and with technical consultations with Professor Fonstad. Mr. Sechen's Master's Thesis, which will be completed in June 1977, will be issued as a separate technical report under this grant. Therefore, only a brief summary of this work will be presented here.

The most prominent result of this effort has been the successful fabrication of the charge-flow transistor and the elucidation of its operating principles. The basic concept is described in the previous Thesis of John A. Wishneusky, and in two articles, one of which is included in this report as an Appendix. This Appendix describes the device, its operating principles, reports the fabrication of specific devices and experimental data illustrating device operation, and also illustrates the use of these devices in fire-sensing applications.

We also have discovered an important feature of operation of these devices, leading to significant improvements over Wishneusky's concept. A patent application covering these new ideas has been filed. A number of industrial firms have indicated interest in exploiting this new technology.

The charge-flow transistors tested in this particular program were coated with several different polymers. The polymer PAPA was used because of its strong and well documented humidity response. Two additional polymers, PFI and PSB, were used because of their relative insensitivity to humidity

variations and their strong responses to smouldering fires. These polymers were tested on conventional lock-and-key devices with samples of smouldering wool, cotton, poly(vinyl chloride), acrylic, and urethanes. Both polymers responded well to all fires. The Appendix illustrates some of the response obtained.

Long-term monitoring of lock-and-key devices was carried out during the five-month period August-December 1975. Devices coated with PAPA, PTMP, PEP, PFPA, PEF, PSB, and PFI were monitored in laboratory ambients and device currents correlated with temperature and relative humidity variations. At constant relative humidity and temperature, the first four polymers showed large drifts, while the latter three were relatively drift-free.

B. Chromatographic Analysis

The chromatographic analysis aspects of this program were carried out primarily by Ms. Deborah Samkoff, a student in the Department of Chemistry, under the supervision of Professor Senturia. In that portion of the program involving interfacing with the infrared spectrometer, Mr. Charles Clahassey, a Graduate Research Assistant in the Department of Physics, supervised by Professor Kastner, participated.

This program was severely disrupted, first, by the long delay in the acquisition of the GC/IR interface required to interface the gas chromatograph eluent with the on-line infrared spectrometer, and, second, by the failure of that interface to perform properly. The net result of these disruptions was a major shift of emphasis away from the proposed chromatography portion of the program and extra effort of the more successful device

research portion. NASA was apprised of this shift in emphasis as it evolved. The activities in the chromatographic area are described in the following paragraphs.

A Hewlett Packard 5712A dual column gas chromatograph with dual thermal conductivity detector and temperature programmable oven was purchased in May 1975 for use in this program. The chromatograph came equipped with columns packed with UCW-982 (10%) on a Chromasorb W support. Standard operation with test specimens indicated proper operation of the system. A sample chromatograph of a solution of C₁₄, C₁₅, and C₁₆ hydrocarbons in hexane is shown in Figure 1. These same columns were used in much of the remaining work, as they were adequate for our needs.

A Digilab GC/IR interface for on-line spectroscopic measurements of the GC eluent was ordered in May 1975, but was not delivered until almost a year later. This delay severely hampered our ability to proceed with the planned experimental program. The interface was installed in June 1976, but significant problems with its operation were noted within the first month, and were never properly addressed by the manufacturer. The problems included poor purge quality with the interface in place (produced, we believe by outgassing of H₂O and CO₂ from the ceramic materials in the interface), and a failure to operate well in the so-called trap mode, in which eluent is trapped in the IR cell for extended observation. The failures were of two types: an unexplained build-up of CO₂ in the cell in the trap mode, and a leak into the cell from the bypass line, which meant that spectra from large GC components (such as solvents) would interfere with the spectra of smaller samples (on the order of several micrograms) trapped in the cell. These

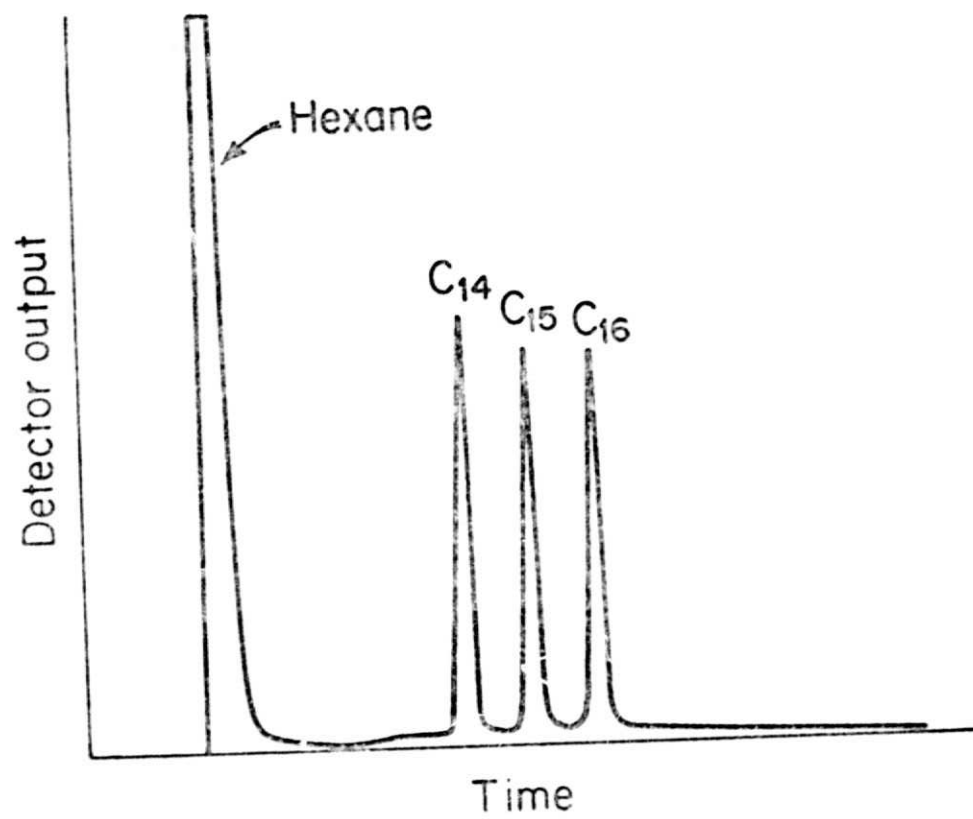


Figure 1: Test chromatogram

problems were called to the attention of Digilab during the period August-October 1976, but no satisfactory responses were received. As a result, a decision was made to cancel the order on the interface in November 1976, and the manufacturer was notified. Our unsatisfactory experience with this interface has prevented successful completion of the primary goal of the chromatographic portion of the proposed program of work.

Some progress was made in the other part of the chromatography program, namely, the design of a suitable trapping system for combustion products. The sampling system design was predicated on the requirements of achieving microgram-sized separated fractions for infrared analysis, as required by the sensitivity of the Digilab system. This meant that some form of concentration of combustion products was needed. Based on surveys of the literature, we selected Tenax GC as an absorber, and arranged to pass the combustion gases from a smouldering 1 gram cotton sample through a glass injection port liner packed with Tenax GC. This liner was then inserted into the GC injection port, and the injection port was then brought quickly to 250°C. Then the column temperature was programmed from 40°C to 200°C at 8°C/min. The chromatographs obtained from such a method showed many poorly resolved peaks and in addition showed strong tailing from the water vapor in the sample. However, with a properly operating interface, we were hopeful of being able to subtract the water vapor spectrum digitally using the on-line computer and examine the composition of the other constituents of the eluent peaks using infrared spectroscopy.

As the problems with the interface were encountered, we attempted to trap out the water vapor using molecular sieve. Contrary to what is suggested

in the literature, we found that trapping of the water vapor also trapped most of the volatile combustion products. We also tried CaSO_4 with the same result.

To illustrate these results, we present a sequence of chromatograms. Figure 2 shows a superposition of a chromatogram taken from the adsorbed products of combustion from a 1 gram sample of smouldering cotton, together with a blank run (no fire) with normal laboratory air. The total area under the combustion products test corresponds to several milligrams of combustion products at the sensitivity settings used for this test. When molecular seive is inserted ahead of the Tenax GC, the chromatogram of Figure 3 results. Note the almost complete disappearance of combustion products. Similar results are obtained when CaSO_4 is used instead of the molecular seive, as shown in Figure 4 which contains a blank together with the chromatogram from a smouldering cotton sample. There is no useful difference between the two traces.

To summarize, the chromatographic portion of the program was not a success, in large part because of technical difficulties with the proposed GC/IR method of analysis. As increasing difficulties were encountered, more and more of the program effort was directed into the actively fruitful device work, with what we feel has been significant and useful results.

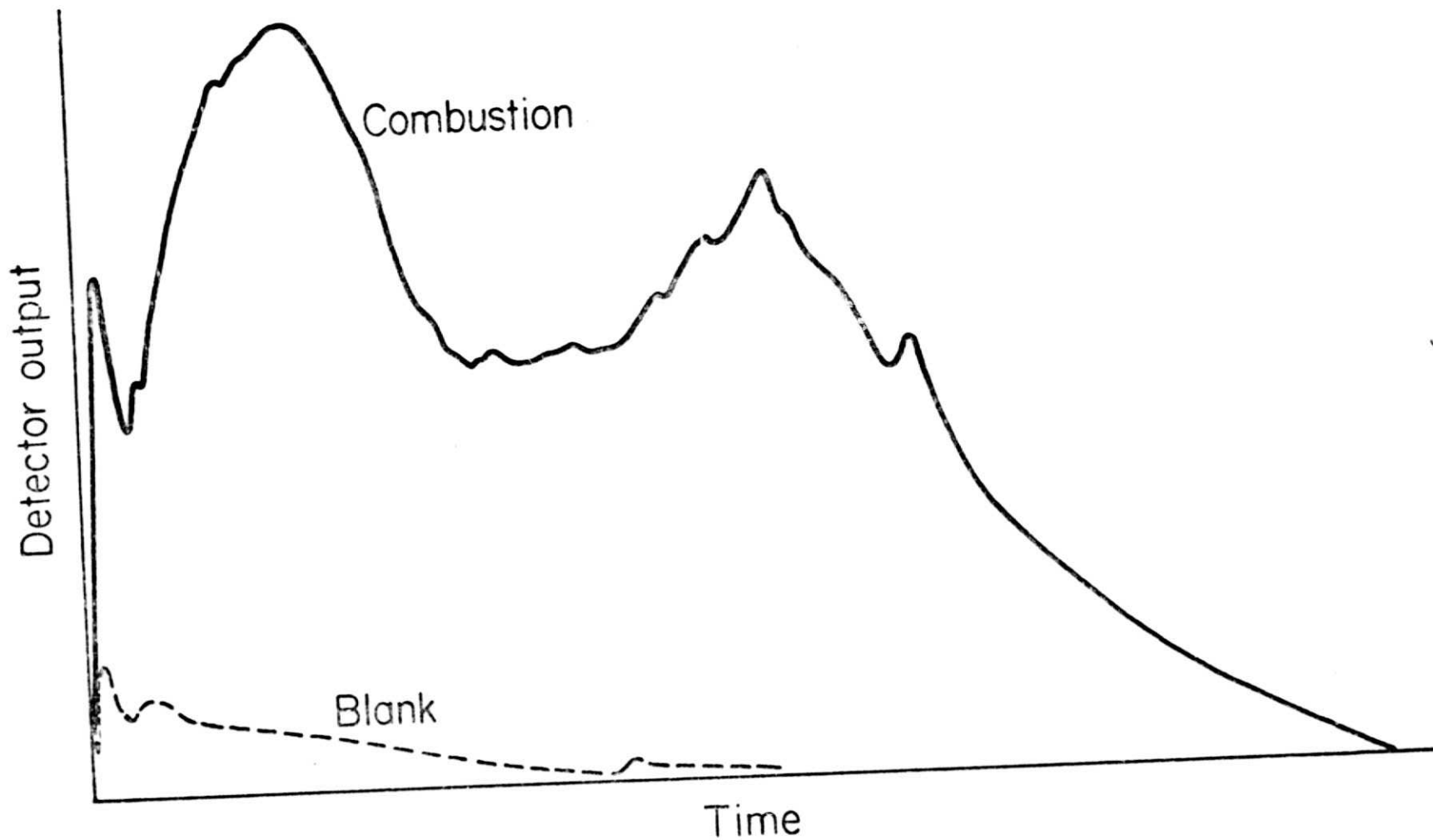


Figure 2: Chromatograms of combustion products from a one gram sample of smouldering cotton adsorbed on Tenax GC, and from a blank run (no combustion) with normal laboratory air.

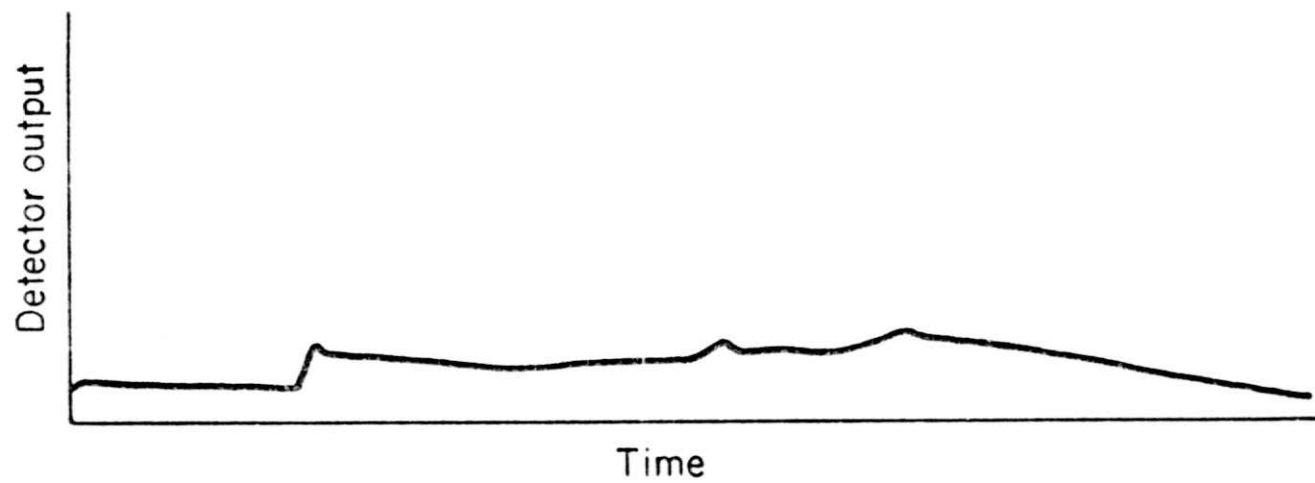


Figure 3: Chromatogram of combustion products from a one gram sample of smoldering cotton, but with molecular seive ahead of the Tenax GC absorber. The scales are the same as Figure 2.

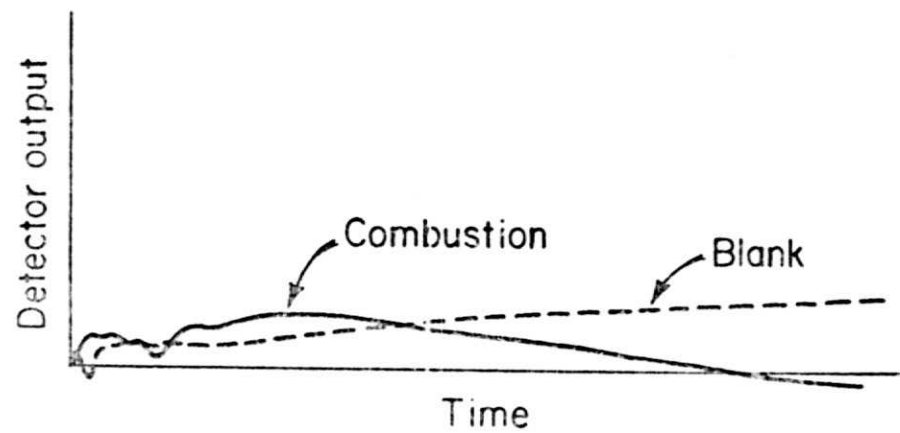


Figure 4: Chromatograms similar to Figure 2, but with a calcium sulfate absorber ahead of the Tenax GC. The scales are the same as Figure 2.

III. PUBLICATIONS, PAPERS, THESES AND PATENTS

This grant has led to the publication of one paper, the presentation of two additional papers at international device meetings, and the filing of a major patent application. In addition, the grant has supported the S.M. Thesis work of Carl M. Sechen, a Graduate Research Assistant in the Department of Electrical Engineering and Computer Science. The various publications, papers, and patents are listed below:

1. "The Charge-Flow Transistor: A New MOS Device", S.D. Senturia, C.M. Sechen, and J. A. Wishneusky, Journal of Applied Physics, 30 106 (1977).
2. "The Charge-Flow Transistor", S.D. Senturia, C.M. Sechen, and J.A. Wishneusky, paper presented at the IEEE Intranational Electron Device Meeting, Washington, D.C., December 7, 1976. A copy of this paper is included as the Appendix to this report.
3. "The Use of the Charge-Flow Transistor to Distinguish Surface and Bulk Components of Thin-Film Sheet Resistance", S.D. Senturia and C.M. Sechen, to be presented at the 1977 Device Research Conference, Cornell University, 27-29 June, 1977.
4. "Charge-Flow Structures as Polymeric Early-Warning Fire-Alarm Devices", C.M. Sechen, S.M. Thesis, MIT Department of Electrical Engineering and Computer Science, May 1977 (also issued as Technical Report No. 1 under NASA Grant NSG-3061).
5. "A Charge-Flow Transistor and Instrument Embodying the Same", S.D. Senturia, patent application filed 22 April 1977 (assigned to MIT).

APPENDIX

"The Charge-Flow Transistor"

The Charge-Flow Transistor*

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Paper presented at
IEEE International
Electron Device Meeting
Washington, DC
Dec 7, 1976
Paper 12.8

ABSTRACT

The Charge-Flow Transistor (CFT) is a new MOS device that we have developed for applications (such as humidity, gas, and fire detection) in which one wishes to monitor the transverse or sheet resistance of a thin film. The CFT is similar to an enhancement-mode MOSFET, but with a gap in the gate metallization above the center of the channel region. The gap is coated with the thin-film material of interest. When a gate-to-source voltage greater than the FET threshold voltage is applied, charge must flow in the thin film before a complete channel is induced in the substrate. The time delay between the application of the gate-to-source voltage and the inversion of the entire channel region is sensitively dependent on the sheet resistance of the thin film.

This paper reports the successful fabrication of CFTs coated with several different polymeric thin films. Experimental results on delay times as functions of device geometry and polymer resistivity are shown to be in good agreement with a simple theory of device behavior. Finally, we report the first use of suitably coated CFT's for the detection of smoldering fires, one of the important applications for this type of device.

*Work supported in part by the National Science Foundation and by the NASA/Lewis Research Center.

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I. Introduction

The Charge-Flow Transistor (CFT) is a new MOS device that we have developed¹⁻³ for applications (such as humidity,⁴ gas, and fire detection⁵⁻⁸) in which one wishes to monitor the transverse or sheet resistance of a thin film. The usual method has been to apply the thin film to a substrate carrying an interdigitated-electrode pair, referred to here as a lock-and-key device (Figure 1). A major problem with this approach is that the film resistance is often very high. The working currents are in the nanoampere to picoampere range, requiring elaborate detection electronics and careful shielding and guarding of components.

Our new device permits the film resistance to be monitored with voltage and current levels normally used in MOS circuits, and, in addition, offers the following advantages:

1. The CFT is based on MOS technology. The fabrication process is such that all silicon-wafer processing, including metallization and passivation, is completed before the deposition of the film. Thus, films that would be unable to survive MOS processing steps can nevertheless be incorporated into the CFT.

2. The CFT can be fabricated as part of an MOS integrated circuit. The detection circuitry can thus be integrated with the sensor element, a factor of major importance in ultimately reducing system size and cost.

3. No guarding or shielding of components is required, even for films with very high resistivities.

This paper outlines the principles of operation of the CFT and presents two kinds of experimental results: Basic measurements of device properties, and the use of the device to detect smoldering fires.

II. Device Principles

The principle of operation of the CFT is most easily described with reference to a simpler structure, the "charge-flow capacitor" (Figure 2). The substrate is conducting, and the two portions of the upper electrode are presumed to be connected together. The thin film of resistive material (called the polymer in Figure 2) is applied to a gap in the upper electrode. The insulator can be any material that is much more resistive than the polymer. When a voltage is suddenly applied between the electrode and the substrate, the capacitor charges in two steps. First, the electrodes charge very rapidly to the applied voltage, yielding the situation depicted in Figure 2b. Second, charge gradually flows through the resistive polymer film (Figure 2c) until, finally, a state is reached (Figure 2d) where the polymer-film portion of the capacitor is uniformly charged to the applied voltage. The time required for this charging process depends on the sheet resistance of the film (the resistivity divided by the film thickness), the width of the gap, and the dielectric constant and thickness of the insulator.

During the charging process, the electric field between the polymer and the substrate gradually increases. The Charge-Flow Transistor exploits this effect by incorporating a charge-flow capacitor into the gate of an enhancement mode MOSFET (see Figure 3). Note the polymer-filled gap in the gate electrode. When a gate-to-substrate voltage larger than the FET threshold voltage is applied, the gate-to-substrate capacitor behaves like the charge-flow capacitor of Figure 2. A complete channel is not formed until the FET threshold voltage is exceeded at all points along the gap. Thus, there is a time delay between the application of the gate-to-source voltage and the appearance of drain current in

the completed channel. The CFT acts as a transducer, converting a sheet resistance measurement into the measurement of a delay time.

An approximate theory of operation of the CFT has been formulated by modeling the polymer-oxide-substrate region as an RC transmission line. The theory is based on the following assumptions: (1) the polymer thickness is uniform across the gap, (2) the dielectric constant of the polymer is unity, (3) the contacts to the polymer are ohmic, (4) the substrate-oxide interface is an equipotential, and (5) conduction in the oxide is negligible. Although none of these assumptions is strictly correct, the theory is reasonably tractable and does yield immediate insight into observed CFT behavior.

With the above assumptions, a theoretical expression for the voltage as a function of time and of the distance from the gate electrode, for steady state excitation by a unipolar square wave of amplitude V_0 and period $2T$ is obtained by standard solution of the diffusion equation for the RC transmission line. The result is:

$$v(x,t) = V_0 \left\{ \frac{1}{2} + \sum_{\text{odd } n} \left(\frac{2}{n\pi} \right) \operatorname{Im} \left\{ \frac{\cosh[\sqrt{n} \alpha (W - 2x)/2L] \exp(in\pi t/T)}{\cosh(\sqrt{n} \alpha W/2L)} \right\} \right\} \quad (1)$$

where

$$\frac{1}{L} = \left(\frac{\rho \epsilon \pi}{d_1 d_2 T} \right)^{\frac{1}{2}} \quad (2)$$

and where ρ is the polymer resistivity, d_1 the polymer thickness, ϵ the dielectric permittivity of the insulator, d_2 the insulator thickness, W the gap width, and $\alpha = (1 + i)/\sqrt{2}$. The theoretical delay time is defined as the earliest time in the "ON" half-cycle ($0 < t < T$) for which $v(x,t)$ exceeds the FET threshold voltage for all x . Equation 1 has been used to calculate delay times for comparison with experiment, as discussed in the following section.

III. Experimental Results

A. Device Behavior

We have fabricated p-channel CFT's with a gate-oxide thickness of 1000 Å (for which $\epsilon = 3.9$),⁹ and with gap widths that vary from zero to 1.5 mils in quarter-mil steps. Figure 4 shows the top view of a single CFT with a one-mil gap, and also shows the nine-transistor array (including three control devices with no gap in the gate electrode) that constitutes the basic replication unit in our fabrication process. Gate, drain, and source metallizations are clearly visible; the dark area in the gate gap is the active sensor region.

Some of the CFT's were spin-coated with a 5% solution of PFI¹⁰ in dimethyl formamide. PFI is a polymer for which we have sheet resistance data from spun-on films based on measurements with conventional lock-and-key devices; we have measured ρ/d_1 to be on the order of 5×10^{14} ohms/square in normal laboratory ambients. Other CFT's were spin coated with a 5% solution of PSB¹¹ in dimethyl formamide, a polymer with similar electrical properties to PFI.

The first measurements performed were to determine the variation of delay time with such parameters as gap width and applied gate-to-source voltage. We have used the standard threshold-voltage definition as that gate-to-source voltage needed to produce a drain current of 1 μ A. Measured threshold voltages for our CFT's are 3.9 ± 0.1 V. In the steady state with a unipolar square wave applied, we measure an experimental delay time between the beginning of the "ON" half-cycle of the square wave and the time at which the drain current reaches 1 μ A. Figure 5 illustrates the variation of delay time with gap width and applied voltage for PFI-coated devices for a unipolar square wave with half-period $T = 20$ seconds. As expected, there is a general increase in delay time with

increasing gap width, and except for the very short delay times (where the transmission line model can be expected to break down), there is a monotonic decrease in delay time with increasing applied voltage. As an illustration of the variation of delay time with gap width, the data at $V_0 = 8$ volts (twice threshold, and marked by the arrow in Figure 5) have been replotted in Figure 6 as a function of gap width. The solid curve is calculated from Equation 1 using a value of $L = 0.13$ mils. This value of L corresponds to a polymer sheet resistance of $\rho/d_1 = 1.7 \times 10^{15}$ ohms/square, in good agreement with the results obtained from the interdigitated-electrode devices.

In the above experiments, the square-wave period was chosen such that $2L$ and W were comparable in size. In this regime, the delay time is roughly proportional to W/L , and since $1/L$ is proportional to the square root of the polymer resistivity, the delay time should vary with $\rho^{1/2}$. The following set of experiments illustrates this variation of delay time with polymer resistance.

B. Fire Detection with CFTs

To illustrate the use of suitably coated CFTs for sensor applications, we present results of tests on the response of PSB- and PFI-coated CFTs to the emanations of a small smouldering fire. The chamber for these experiments is an 12-inch-long tube (2-inch diameter) containing at one end a heater coil into which a small combustible charge can be placed, and, at the other end, a mount for the device under test. A sufficient air-flow rate is maintained to prevent a build-up of combustion products in the tube. Combustible charges are in the range 6-10 mg of material, and the heater current is set to produce smouldering without flaming. Each combustible material has a characteristic profile of emission of detectible combustion products. The data presented below are for

samples of 8 mg of an acrylic fiber.

Figure 7 shows the response of a PSB-coated lock-and-key device to the smouldering fire. The device is driven with 60 volts; current is measured with an electrometer; the entire assembly is electrically shielded. Note the arrival of significant combustion products at about 120 seconds after heater turn-on, an extremely reproducible feature of all such tests with acrylic samples. Note also the increase in current through the device by a factor of 10 at the peak compared to the pre-fire level. The heater current is not turned off until after smouldering is complete, as evidenced by the peak in the PSB response. Not shown in this figure is the fact that the current does recover fully to its pre-fire value, and that a repetition of the test does produce reasonably repeatable results. Responses of PFI to acrylic are quite similar to the PSB responses.

The corresponding waveforms for a PSB-coated charge-flow capacitor are shown in Figure 8. Before the heater is turned on, alternate cycles of charging current and discharging current are evident. At about 110 seconds after the heater is turned on, the charging current shows the same sudden peak as was seen in the lock-and-key device, thus illustrating the charge-flow principle directly.

Finally, the results of fire tests on a PFI-coated CFT with acrylic are shown in Figure 9. Here, it is not possible to represent the continuous change of polymer resistivity, because one samples the delay time only once each period. Nevertheless, the trend is quite clear, and closely follows the results of Figures 7 and 8. The delay time falls from 17.6 seconds at heater turn-on to 5.6 seconds at about 120 seconds after turn-on, and then returns to its pre-fire value. The magnitude of the change in delay time is consistent with theory.

The polymer resistance, as measured by a lock-and-key device, decreases by a factor of 10. The corresponding CFT delay time drops by a factor of 3, very close to the value $\sqrt{10}$ that one estimates from the theory.

IV. Discussion

The above results demonstrate the principle of operation of the CFT, and illustrate its potential use in sensor applications.

One caution is that because the CFT sensor cannot be fully encapsulated, it will be more susceptible to long-term contamination than encapsulated MOS devices, and deposition of suitable passivation layers over the gate oxide and/or over the entire chip may be required. Thus far, we have not observed threshold voltage drift on a time scale of months with the devices operated in normal ambients at room temperature.

Even if passivation layers are added to the device, the application of the sensing film is the final processing step. Our particular polymer films are spun on from solution but the CFT could also be made with evaporated, sputtered, or even sprayed-on films. Thus MOS-compatible and MOS-integrable sensing devices can now be made from materials which previously could only be used in discrete-device form.

The authors wish to thank Dr. Dan Smythe of the MIT Lincoln Laboratory Microelectronics Laboratory, for access to pattern-generation facilities used in making the masks for our devices.

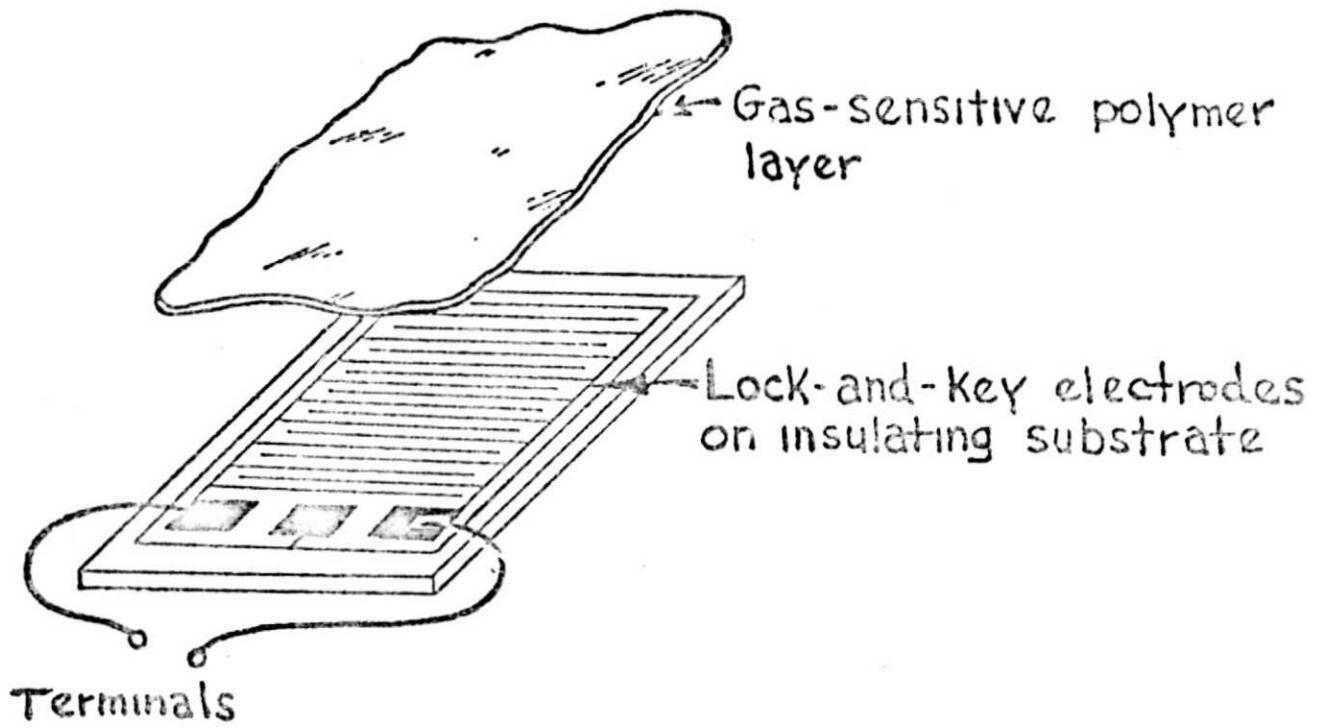
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10. poly(imidazole) from 1,4-bis(phenylglyoxyloyl) benzene and ferrocene -1,1'-dicarboxaldehyde. See Reference 7 for details.

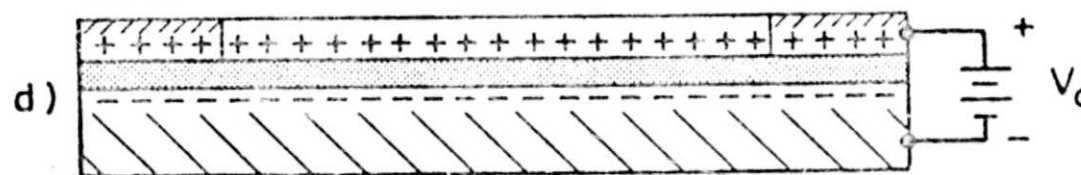
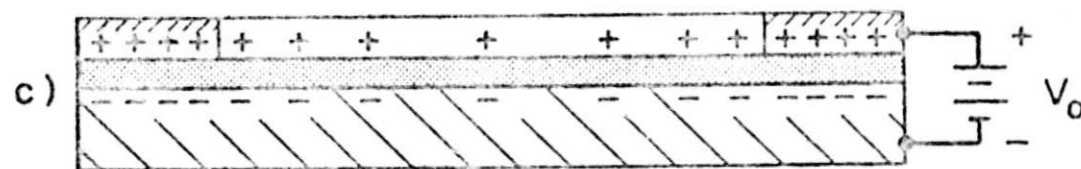
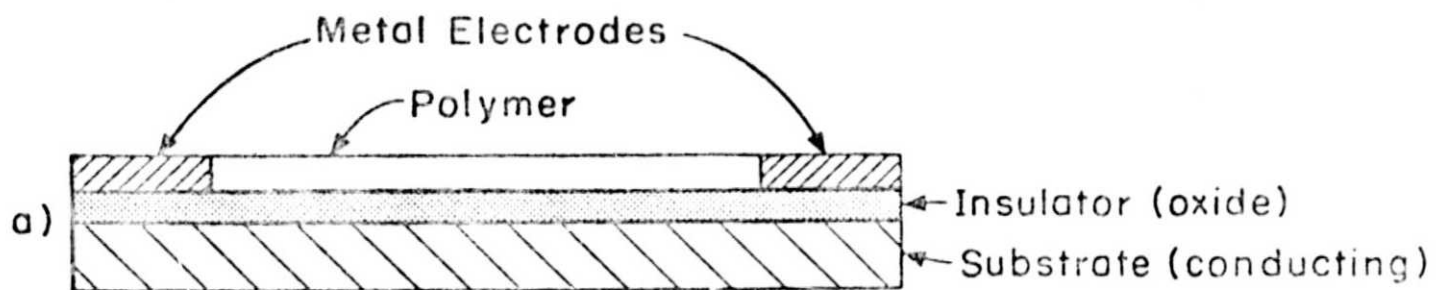
11. poly(Schiff's base) from p-phenylene diamine and thiophene -2,5-dicarboxaldehyde. See Reference 7 for details.

Figure Captions

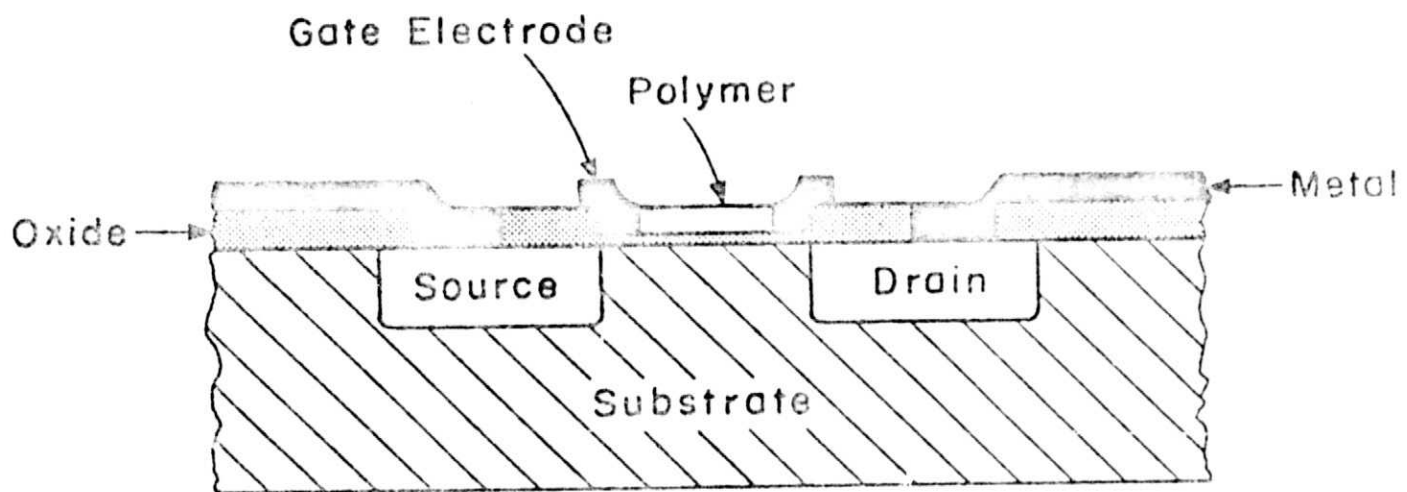
- Figure 1: Exploded view of a lock-and-key device.
- Figure 2: Cross sectional view of the charge-flow transistor. (a) Structure; (b) Just after application of a voltage; (c) Partially charged; (d) Fully charged.
- Figure 3: Cross sectional view of the charge-flow transistor.
- Figure 4: Top view photographs of charge-flow transistors. The gapped gate metallization lies between drain and source metallizations. The nine-transistor array contains three control transistors (no gap) and six transistors with gap widths of 0.25, 0.5, 0.75, 1.00, 1.25, and 1.50 mils.
- Figure 5: Variation of CFT delay time with gap width and applied unipolar-square-wave voltage. The threshold voltage of the devices is 3.9 ± 0.1 V.
- Figure 6: Comparison of experimental delay times from Figure 5 with theoretical delay times calculated from Equation 1.
- Figure 7: Fire test of a PSB-coated lock-and-key device with a smouldering 8 mg acrylic sample.
- Figure 8: Fire test of a PSB-coated charge-flow capacitor with a smouldering 8 mg acrylic sample.
- Figure 9: Fire test of PFI-coated charge flow transistor with a smouldering 8 mg acrylic sample.



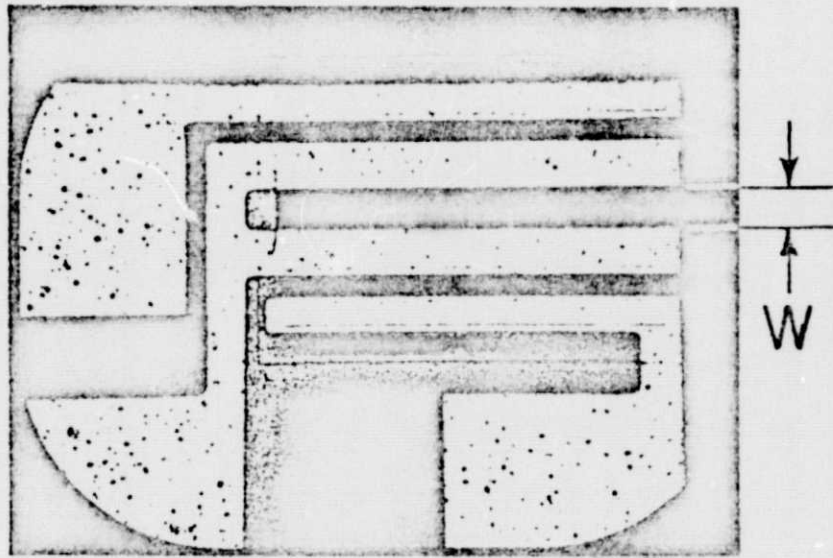
Lock-and-key Device



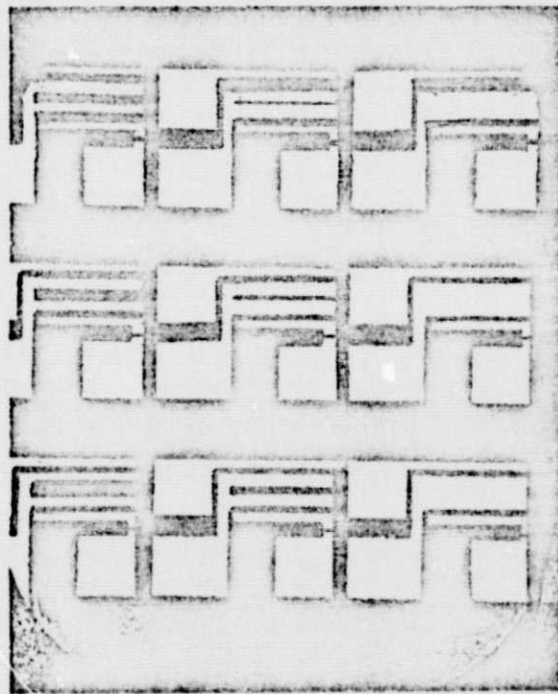
The Charge-Flow Capacitor



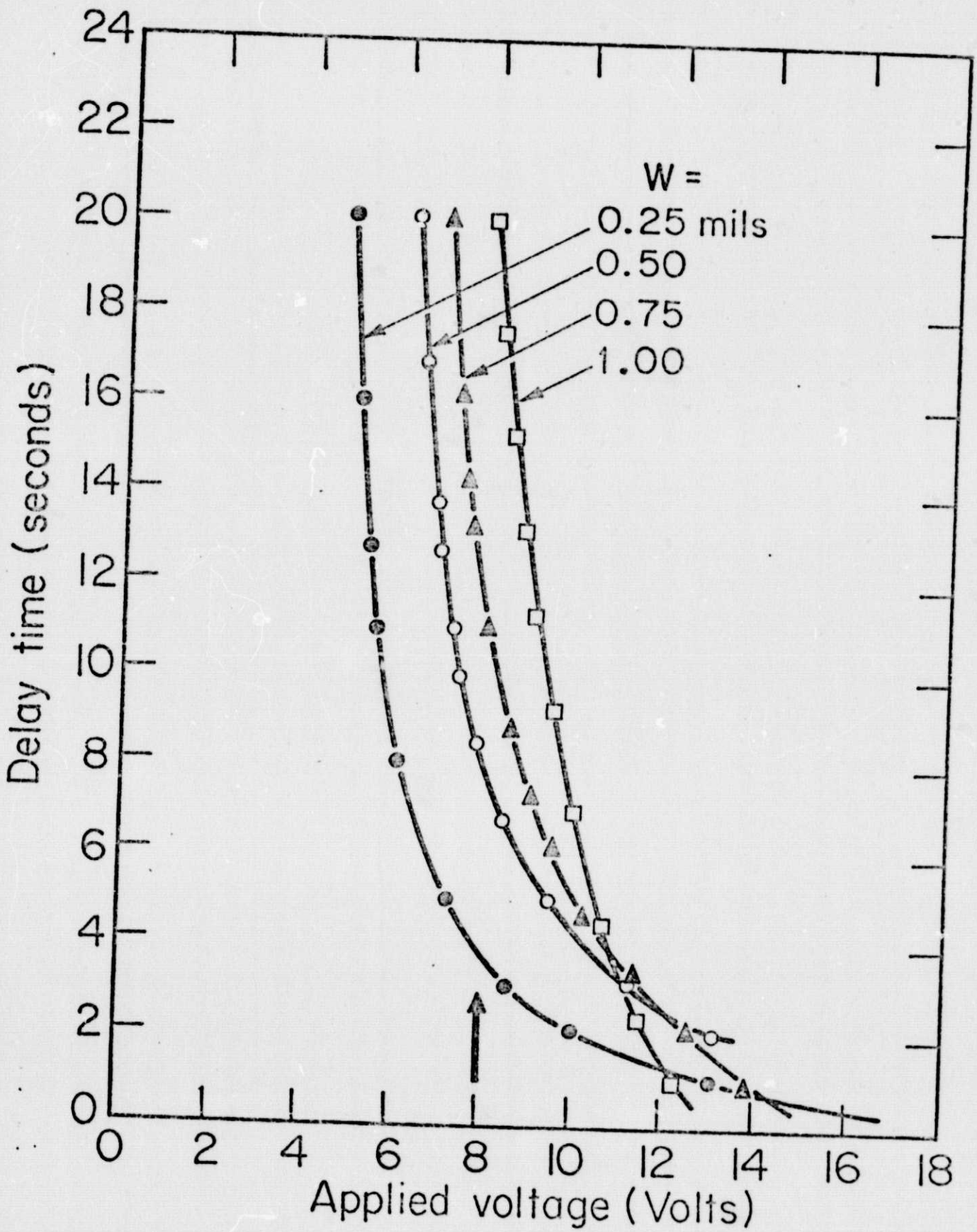
The charge-flow transistor

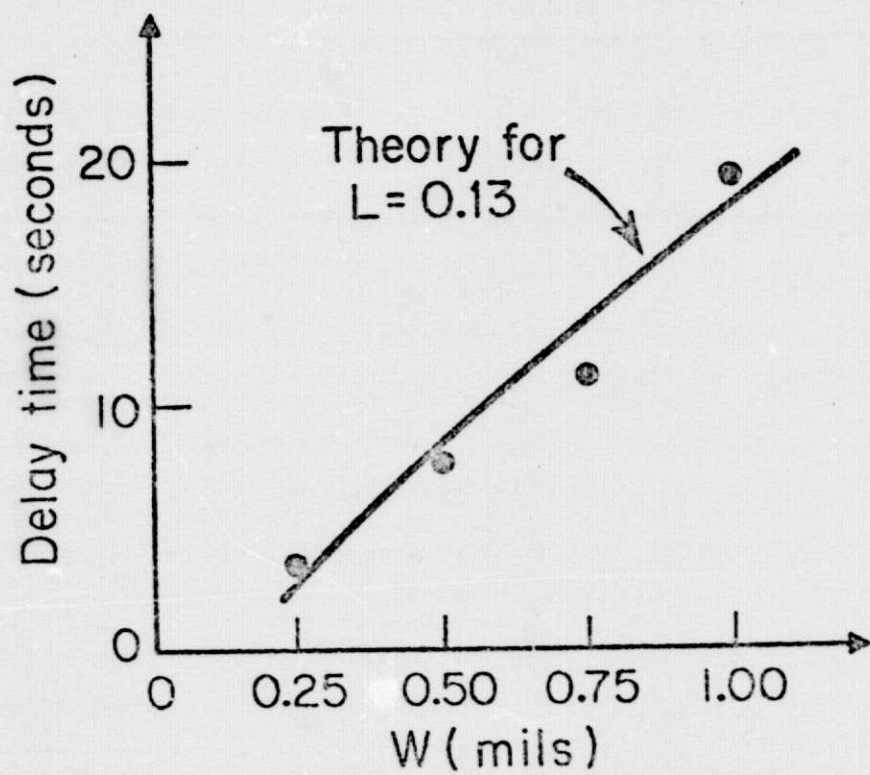


TOP VIEW
Charge Flow Transistor
($W = 1 \text{ mil}$)

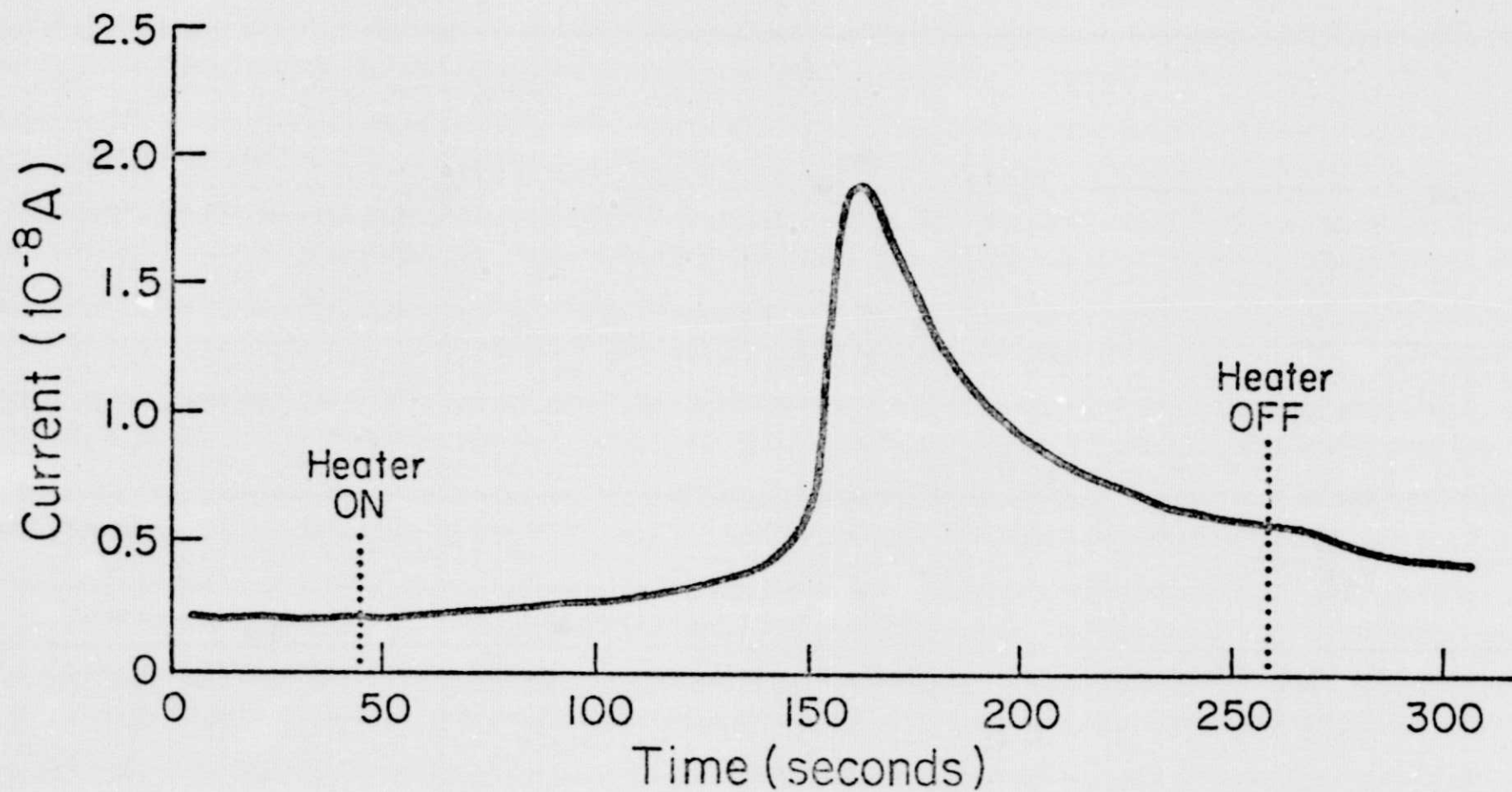


Nine-Transistor Array

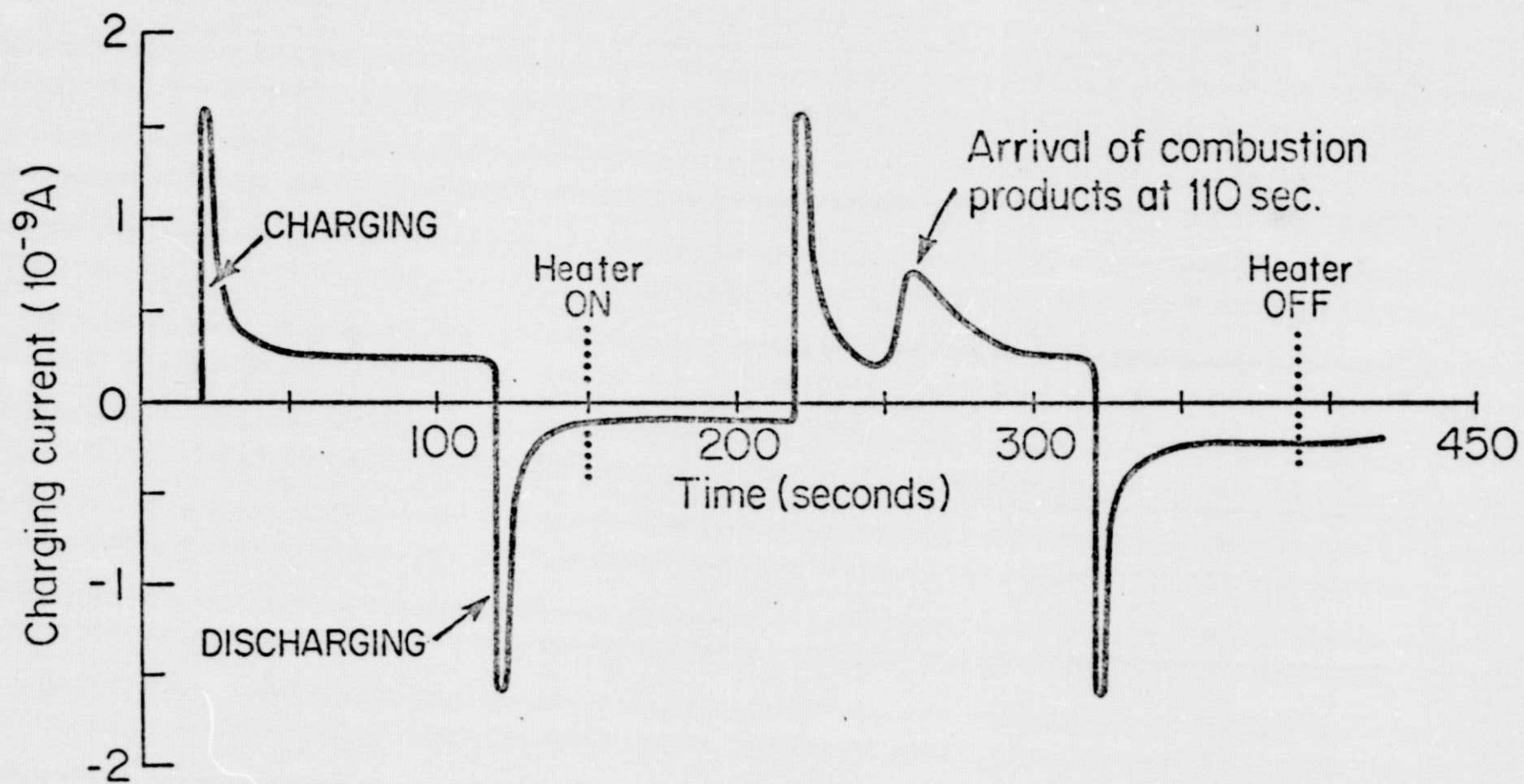




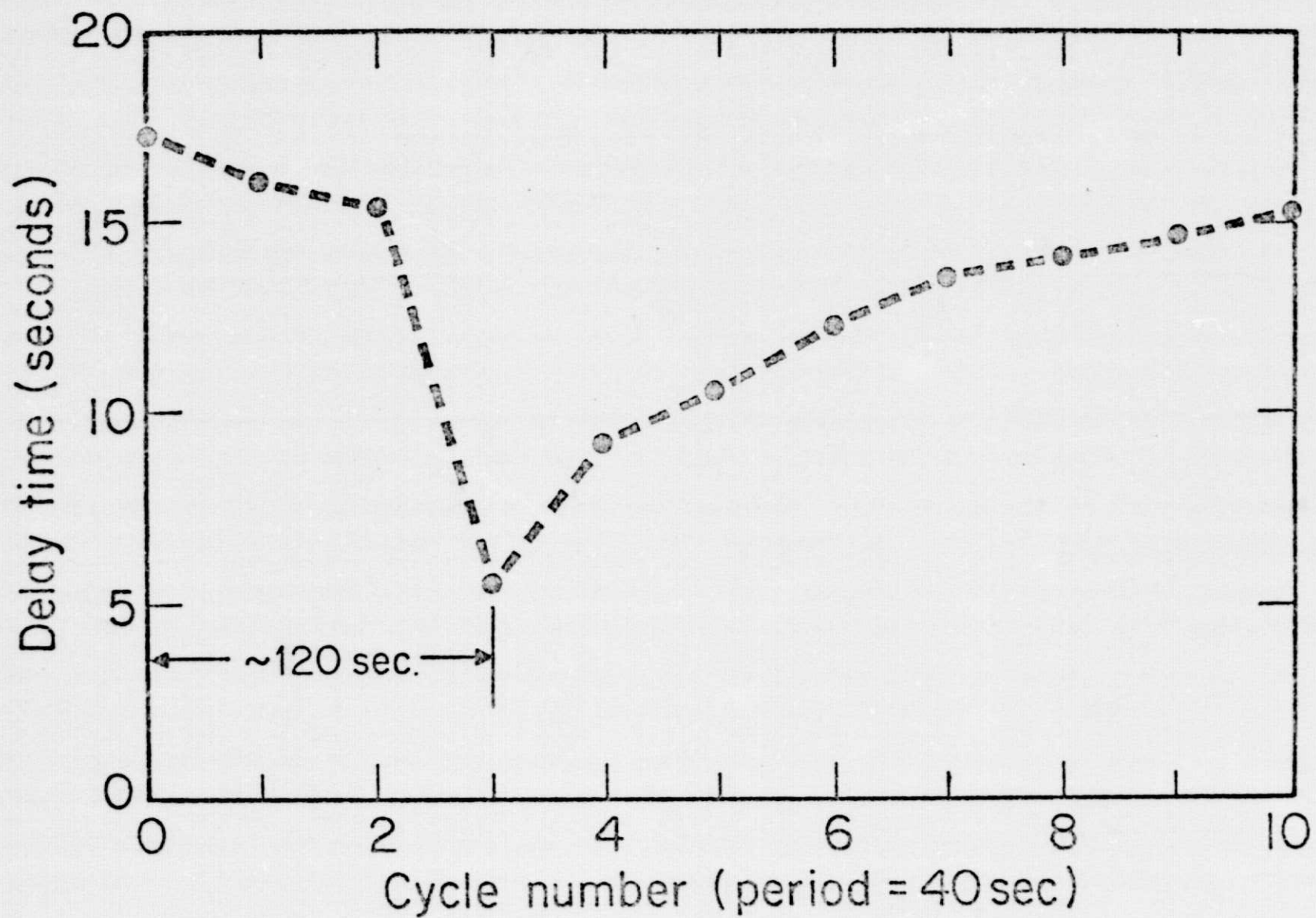
Delay Time vs W for $V_0 = 8$ Volts



Lock-and-Key PSB with Acrylic



Charge-Flow Capacitor PSB with Acrylic



Charge-Flow Transistor (PFI with acrylic)