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MASS ANALYSIS OF NEUTRAL PARTICLES AND IONS RELEASED DURING
ELECTRICAL BREAKDOWNS ON SPACECRAFT SURFACES

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MASS ANALYSIS OF NEUTRAL PARTICLES AND IONS RELEASED DURING
ELECTRICAL BREAKDOWNS ON SPACECRAFT SURFACES

1. ABSTRACT

Charging of insulating surfaces and the resulting electrical breakdowns are known to have been responsible for numerous malfunctions of orbiting spacecraft. Both theoretical and experimental studies of charged-particle fluxes from breakdown events have been made in several laboratories.

The present research broadens the attack on this problem to include neutral particles (molecules and molecular clusters) and specifically to develop methods of measuring the mass spectra and total emitted flux of such particles.

The design and construction of the specialized mass spectrometer required for this work was completed at the end of January 1981. Tests at Penn State were entirely satisfactory but after transfer to the Tenney vacuum chamber at NASA Lewis Research Center an immediate and progressive deterioration of its performance occurred, probably caused by oil vapor contamination in the Tenney chamber. The mass spectrometer was subsequently restored to normal operation by extensive cleaning and rebuilding. The reassembled instrument was then operated satisfactorily for the remainder of the project in its original test chamber at Penn State which was adapted to include the necessary apparatus for electrical breakdown studies on polymer films.

Electrical breakdowns were initiated by a movable blunt contact touching the insulating surface. A high-voltage ramp was applied to the contact through an energy-storing line until breakdown occurred. The resulting surface damage sites could be made similar in size and shape to those produced by a high voltage electron beam system operating at similar discharge energies. The contact

discharge apparatus was used for final development of two different high-speed recording systems and for measurements of the composition of the materials given off by the discharge. An additional advantage of this technique was that, by progressively moving the sample relative to the contact, the observed gas bursts could be correlated with actual discharge sites left behind on the sample. The sample could then be removed and observed under an optical or electron microscope.

With this apparatus it was shown that intense instantaneous fluxes of neutral particles were released from the sites of electrical breakdown events. For Teflon FEP and PFA films of 50 and 75 microns thickness the material released consisted almost entirely of fluorocarbon fragments, some of them having masses greater than 350 amu (atomic mass units). The material released from a 50 micron Kapton or Mylar film consisted mainly of light hydrocarbons with masses at or below 44 amu, with additional carbon monoxide and carbon dioxide. Tefzel films released hydrogen, HF, hydrocarbons, carbon monoxide and carbon dioxide.

Additional studies were made with a Laser Micropulse Mass Analyzer, which showed that visible discolorations at breakdown sites were correlated with the presence of iron on the polymer side of the film, presumably caused by punch-through to the Inconel backing. There was also a considerable amount of sodium and potassium compounds, accompanied by hydrocarbons, on all samples, including Teflon samples which had been handled only with tweezers since removal from a bulk supply.

Further tests were done on Kapton samples which had been irradiated by an oxygen ion beam at NASA Lewis Research Center. The irradiated samples were free of surface hydrocarbon contamination but otherwise behaved in the same way as the Kapton samples tested earlier.

Total emitted flux measurements were made with a special fast ion gauge and recording system. Most of the samples released $1 - 2 \times 10^{-4}$ Torr liters of gaseous products per discharge.

Many of the observed phenomena could have significant effects on spacecraft surfaces. Jets of heavy polymer fragments from Teflon discharge sites could form insulating layers on adjacent electrodes, could act as triggers for gas discharges, and could change the secondary electron emission properties of distant surfaces. The much lighter fragments from Kapton may also be capable of triggering remote discharges. The ejection of material from the conducting backing of polymer films may result in metallic contamination of nearby insulation. Photon-induced and electron-induced desorption of gas from surfaces adjacent to a discharge site also occurs and adds to the intensity of the observed neutral-particle pulses.

Tefzel should probably be used with caution on spacecraft if there is a possibility of electrical discharge through it because of the possible production of HF.

The discovery of traces of both organic and inorganic molecules on the surfaces of untouched samples of polymer film suggests sources of contamination (possibly unavoidable) during production or packaging. Only the two samples exposed to oxygen ion bombardment were relatively clean. This indicates an additional variable that should be considered when testing spacecraft materials in the laboratory.

2. INTRODUCTION TO THE PROBLEM

Spacecraft charging in geomagnetic substorms often leads to large changes in potential of spacecraft surfaces relative to space plasma potential. An additional source of localized charging of spacecraft surfaces occurs in the

interaction of high-voltage solar arrays with the charge exchange plasma generated by ion thrusters. In either case the resultant potential differences may reach several kilovolts. The charging may lead to large potential gradients across dielectrics used on the spacecraft surfaces or used to insulate one part of the spacecraft from another. Brief but intense discharges may result.

Discharges across spacecraft dielectrics may produce spurious electromagnetic signals. Discharges may also degrade thermal protective films, contaminate optical surfaces, and cause direct structural damage to the dielectrics. Laboratory tests indicate the possibility of degrading other insulators if metallic vapors are emitted from the discharge, or of forming extremely thin insulating layers on conductors which can then charge enough to modify critical electrostatic field distributions near charged-particle detection equipment. In general there will be a large and almost instantaneous release of both neutral and charged particles from the site of a breakdown event.

Hundreds of charging-induced anomalies were identified in more than twenty spacecraft between 1971 and 1976.

Because of the absence of existing data on the composition of the neutral and ion fluxes from dielectric breakdown events, mass spectrometric analyses of these fluxes are particularly important.

Mass analysis of particles from an electrical breakdown event involves a particularly difficult set of constraints. The event occurs essentially at a point in space and at an instant in time. The exact position and timing of the event are not known in advance, although they can be influenced to some extent. The event produces a swarm of neutral molecules, molecular clusters and ions of different masses which presumably radiate from the breakdown site over a wide range of speeds and directions. At a distance greater than a few cm from the breakdown site the particle number density is likely to be quite low and falling rapidly because of both speed variations and angular dispersion. The expanding

gas and ion burst will pass any given point in a time much shorter than the time taken for any conventional mass spectrometer to scan once through its mass range.

For reasons discussed in the original proposal, it was decided that a time-of-flight mass spectrometer offered the most cost-effective approach to the study of this problem.

3. MASS SPECTROMETER

The time-of-flight mass spectrometer and the vacuum chamber used for its development are shown in Figure 1. The pulsed two-field ion source¹ is on the left. Incoming molecules are ionized by an electron beam inside the ion source and the resulting ions are accelerated in approximately monoenergetic bunches into the flight tube. The ions therefore reach the ion detector in ascending order of mass, according to the formula

$$t = s(m/2eV)^{1/2}$$

where t is the flight time through the flight tube, s is the length of the flight tube, m is the ion mass, e is the ion charge, and V is the potential difference through which the ions fall inside the source. With a 115cm flight tube and 300V accelerating potential, the flight time of an ion of mass 100 amu is approximately 47 μ sec.

A segmented cylindrical lens focuses the ion beam and centers it on the input of the ion detector. The flight tube is operated at ground potential, rather than at high potentials as in most time-of-flight mass spectrometers, in order to minimize electrostatic interactions with the sample charging apparatus. An electron multiplier ion detector is used for high sensitivity and fast response.

It follows from the equation for the flight time of an ion that the electrical signals leaving the ion detector represent a series of complete mass spectra, each one having the corresponding source pulse at its $t = 0$ point. Any number of successive spectra can be displayed, from a single spectrum up to as many as 100 spectra per millisecond. A typical mass spectrum of residual gases in the vacuum chamber is shown in Figure 2. Special techniques are needed for operation at repetition intervals faster than the ion flight times² and for displaying the rapidly-changing spectra.^{3,4}

The layout of the vacuum system, which is based on a Leybold TMP-350 turbomolecular pump, is shown in Figure 3.

4. OTHER APPARATUS

A. Direct-Contact Charging Apparatus

This apparatus is shown in Fig. 4. Electrons are fed onto the insulating surfaces from a smooth platinum contact. A slowly increasing negative potential from a high-impedance, low-capacitance source is applied to the contact until breakdown occurs. The sample is held in place on a perforated, rotatable 9 cm disc by a circumferential retaining ring.

The discharge current waveforms, peak currents, and surface damage characteristics obtained with this apparatus were made similar to those produced by a high-voltage electron beam charging system by choosing a suitable length (about 45 cm) of RG-62 coaxial cable as an energy storage line. A useful feature was that, by progressively rotating the sample beneath the contact, the observed gas burst could be correlated with actual discharge sites left behind on the sample, which could then be removed and observed under an optical or electron microscope. Most discharges occurred within about 2mm of the contact, with occasional discharges up to 4 mm away. Few occurred directly beneath the contact. Breakdown voltages were similar to those obtained elsewhere with monoenergetic electron beam charging systems.

B. Miniaturized Electron Beam Charging System

A miniaturized electron beam charging system similar in principle to that used at NASA Lewis Research Center was built. It used a small commercial electron gun modified in our laboratory to allow operation at up to 20 Kv instead of the original 7 Kv.

Although the electron energies and current densities, sample materials and pulse detection circuit were essentially identical with those used by other experimenters, no large pulses were detected. The limited diagnostic techniques possible in such a small system showed a reasonably uniform beam distribution and surface potentials high enough for breakdown to occur. Test pulses of current were correctly recorded when passed through the sample support. Numerous very small current pulses were observed, with larger pulses occurring only during sudden reductions of beam energy or movements of the beam.

C. High-Speed Recording and Display Apparatus

High-resolution mass spectra were generated at such a rate in this experiment (up to 5×10^4 /sec) that even modern digital recorders were barely adequate for following complex events. Photographic techniques have been used almost exclusively to date. For semiquantitative measurements a simple intensity-modulated display was used, as described in the next section. When accurate quantitative measurements of peak heights were desired an offset raster display was used. Here successive conventional mass spectra were superimposed upon one another, after which the oscilloscope trace was moved upwards and to the right for display of the next group.

An improved signal-to-noise ratio would be possible if the superimposed spectra could be averaged in the mathematical sense. Digital circuits for achieving this have been designed.

D. Apparatus for Measuring Total Volumes of Evolved Gas

A fast-response nude hot-cathode ion gauge was used to detect the pressure transients following electrical breakdown of a sample. From knowledge of the system volume, the total amount of gas was determined. From the time constant of the near-exponential pressure drop following the peak, the approximate mean molecular weight of the evolved gases could be determined independently of the mass spectrum.

5. SAMPLES

The samples tested in the main series of direct-contact experiments were Teflon FEP and Kapton H films of 50 and 75 micron thicknesses. They were metalized on one side with silver overlaid by an Inconel protective coating. No adhesive backing was used. These materials are widely used in spacecraft applications.

A subsequent series of tests was carried out on 50 micron Mylar film with an aluminum backing, 125 micron Teflon film with silver/Inconel backing, 50 micron Tefzel and 50 micron Teflon PFA. The latter two samples were without backing.

A final run was made with three special 75 micron Kapton samples supplied by Dale Ferguson of NASA Lewis Research Center. Two of these samples had been exposed to an oxygen ion beam to simulate conditions in Shuttle orbit, and one was an unexposed reference sample from the same bulk supply.

6. RESULTS

A. Mass Spectra of Neutral Particles

Tests with Teflon FEP samples showed that an intense burst of neutral fragments was being released from each discharge. A large number of peaks representing

Teflon fragments of the form $C_xF_y^+$ could be seen in each mass spectrum.

The variation with time of the number density of these Teflon fragments was obtained by using the mass peak amplitude signals to intensify a cathode-ray oscilloscope trace, producing an array of dots. By deflecting this display downwards, a semiquantitative indication of the various changes in number densities was obtained. Such a display is shown in Figure 5 with the major peaks identified. A background spectrum is included for comparison. The most intense peak corresponds to CF_3^+ but ions up to and beyond $C_5F_9^+$ are present. Switching off the ionizing electron beam in the mass spectrometer ion source causes these peaks to disappear, showing that they are ionization products of even larger neutral fragments and not ions released directly from the discharge. Far more of these heavy ions were observed than are present in the mass spectrum of the heaviest fluorocarbons for which published data are available (C_6F_{14}), suggesting that very large neutral fragments, of mass much greater than 350 amu, were leaving the Teflon surface during the discharge. Later experiments with Teflon PFA samples showed similar results.

A 50 micron Kapton H film with metal backing was then installed in the apparatus and a new series of breakdown measurements was begun. The Kapton produced only light fragments, giving rise to mass spectra containing mainly masses 44, 28, and 15, as shown in the intensity-modulated spectrum of Figure 6. It appears that the mass 44 peak represents CO_2^+ and $C_3H_8^+$; mass 28 is CO^+ and $C_2H_4^+$, and mass 15 is CH_3^+ . It should be noted that Kapton contains a substantial amount of oxygen.

To confirm these findings and to show the relative amounts of the various ion fragments, the same data were displayed in an offset raster display (Fig. 7). For comparison, a background gas spectrum is shown in Fig. 8.

Samples of 50 μ metal-backed Mylar [Polyethylene Terephthalate] film were then tested in a similar way. Results, shown in Fig. 9, were very similar to those obtained with Kapton samples. Most of the evolved gas molecules had masses at or below 44 amu. Carbon monoxide, carbon dioxide, and light hydrocarbons were seen. The basic polymer unit of Mylar has some of its carbon atoms immediately adjacent to oxygen atoms. It is known that electron beams destroy or modify these oxygenated functional groups.

A sample of clear 50 μ Tefzel (ethylene/tetrafluoroethylene polymer) was then tested in the same way. The main gases evolved during breakdown were HF, H₂, CO₂, and CO plus C₂ and C₃ hydrocarbons (Fig. 10). The material appeared to release H₂O and C₂ hydrocarbons in small quantities during sample pumpdown, as determined by operating a cold finger near the sample and cyclically heating it.

The Kapton samples which had been exposed to an oxygen ion beam gave results generally similar to those already described for unmodified Kapton, except for greatly reduced hydrocarbon production, especially during the immediate pre-discharge period which probably represents removal of surface contaminants by surface currents.

B. Breakdown Voltages

In general breakdown voltages were slightly higher for a given thickness of Kapton than with Teflon, but there was otherwise little variation between samples. A typical 50 μ sample of Kapton broke down at about 16-18Kv, while the same thickness of Teflon usually broke down at 8-15Kv. Thicker films broke down at somewhat higher voltages, but even 125 μ Teflon often broke down before 20KV was reached. This suggests that defects in the films may play a major part in determining breakdown voltages.

C. Total Gas Evolution

The Kapton, Teflon and Mylar samples typically evolved about $1 - 2 \times 10^{-4}$ Torr liters of gas per discharge with the Teflon usually towards the low end of this range. The mean molecular weight of the evolved gas was much higher for the Teflon (by a factor of about two) so that the actual mass of the evolved material was probably similar for each type of sample. Tefzel was not tested in detail but appeared to behave similarly to Mylar.

Photon-induced desorption and electron-induced desorption of adsorbed gases from surfaces near discharge sites are also to be expected and have been observed. The effect is not directly linked with the presence of a polymer film, since any spark could supply the necessary photons and electrons. The effect appears to be about one order of magnitude smaller than the direct gas evolution from polymer film breakdowns.

D. Surface Changes After Testing

Removal of metal from the backing film was detected with Kapton samples. In some cases the Kapton film remained intact above the damage site. The effect is originally observable only under magnification but after several months in air the holes are easily visible with the naked eye because of local discoloration of the Kapton.

Some of the Teflon samples with metal backing also showed progressive discoloration over a period of months after removal from the test chamber. Reference samples stored under identical conditions showed no such effect. The yellow discoloration has the appearance of corrosion of the silver coating beneath the Teflon. There is a complex pattern of discoloration with little correlation with the sites of discharges.

Mylar and Tefzel samples showed no long-term deterioration after testing. Small areas of discoloration and cracking of the surface were visible close to some discharge sites, especially with Tefzel, but these did not change with time.

E. Laser Micropulse Mass Analyzer (LAMMA) Experiments

Phenomena such as the remote breakdown observed in some samples (up to 4 mm from the platinum contact), and the development of visible discolorations of samples during storage after breakdown tests, indicated the need for surface-sensitive studies.

Arrangements were made, with the kind cooperation of Leybold-Heraeus, Inc., for analysis of several of our samples on one of their LAMMA 1000 instruments. No obvious surface defects could be found with the optical target finding system, and so the remote breakdown mechanism could not be studied. The visible discolorations at former breakdown sites were found to correlate with the presence of iron on the polymer side of the film, presumably caused by punch-through to the Inconel backing. Considerable amounts of sodium, potassium and hydrocarbons were present on all samples, including Teflon samples which had been handled only with tweezers since removal from a bulk supply. There is an implication that the surfaces may be picking up contaminants during manufacture or packaging.

7. DISCUSSION AND CONCLUSIONS

Figure 11 shows the chemical structure of Teflon FEP and Kapton H. The origins of many of the observed fragment ions (formed in the mass spectrometer ion source by electron bombardment of polymer fragments released by the discharge) are obvious.

It must be remembered that fragmentation occurs also in the process of ionization inside the mass spectrometer, so that the parent polymer fragments are always at least as big as the resulting ions and in some cases are very much bigger. Thus, in the case of the Teflon samples, which show a higher proportion of heavy ions than the heaviest fluorocarbon for which data could be found in the literature, it can be concluded that many of the released neutral fragments have masses above 350 amu. They probably form surface films after a few collisions with the system walls.

The Kapton samples showed mainly light ions in their mass spectra and the approximate independent check on molecular weight provided by the total-pressure waveform measurements confirmed that the parent neutrals were also light (at or below 44 amu). Such gases would be quickly desorbed from any surface they might hit except at low temperatures.

Figure 12 shows the chemical structures for Teflon PFA, Mylar and Tefzel. The former gave results similar to those of Teflon FEP. The latter two showed mainly light ions in their mass spectra during electrical breakdown, proving that the parent neutrals were also quite light. An important neutral fragment evolved from Tefzel appears to be HF, which is extremely reactive and could easily alter the work function of adjacent surfaces.

Items (1) - (3) of Figure 13 summarize the results of many of these experiments. The figure also shows the following additional phenomena which have been identified and studied:

Secondary discharges were seen on several occasions. Electrical breakdowns were triggered at distances up to 15cm from the site of a Teflon film breakdown. In some cases the metallic electrodes between which the secondary discharge occurred were operating at less than 65% of their normal breakdown potential difference. Triggering is presumably caused by the burst of neutral

and ionized material from the polymer breakdown site. The effect probably occurs also with other polymers, but this has not been confirmed. This phenomenon has been seen with both electron beam and direct contact charging of the sample.

Direct transfer of Teflon fragments is obviously likely because of the large fragments observed in the mass spectrum. It has been confirmed by the formation of insulating layers near the breakdown site and by instantaneous decreases in the secondary electron emission coefficient of dynode surfaces in electron multipliers up to 100 cm away. Partial recovery occurs over a period of several days. The effect does not appear to occur with the other polymers tested.

Indirect transfer of Teflon has similar effects. It appears to be the result of Teflon fragments striking an intervening surface and then being almost instantaneously re-emitted into areas which are not on a direct line of sight from the discharge.

Removal of metal backing and Induced desorption have been discussed previously.

Many of the phenomena listed in Figure 13 could have significant effects on spacecraft surfaces. Jets of heavy polymer fragments from Teflon discharge sites could form insulating layers on adjacent electrodes, could act as triggers for gas discharges, and could change the secondary electron emission properties of distant surfaces. The much lighter fragments from Kapton may also be capable of triggering remote discharges. The ejection of material from the conducting backing of polymer films may result in metallic contamination of nearby insulation. Photon-induced and electron-induced desorption of gas from surfaces adjacent to a discharge site also occurs and adds to the intensity of the observed neutral-particle pulses.

Tefzel should probably be used with caution on spacecraft if there is a possibility of electrical discharge through it because of the possible production of HF.

The discovery of traces of both organic and inorganic molecules on the surfaces of untouched samples of polymer film suggests sources of contamination (possibly unavoidable) during production or packaging. Only the two samples exposed to oxygen ion bombardment were relatively clean. This indicates an additional variable that should be considered when testing spacecraft materials in the laboratory.

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PRESENTED PAPERS

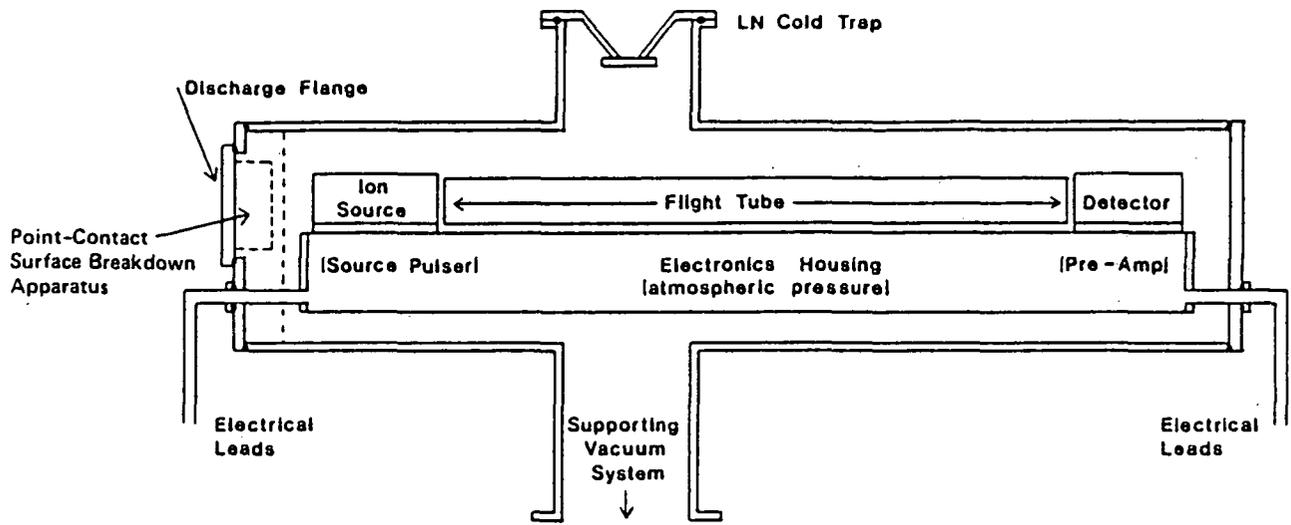
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1. S. M. Danziger, "Electron Gun Modeling with an Electrolytic Tank",
M. S. Thesis, March 1981.
2. P. Desai, "High-Frequency Integrated - Circuit Preamplifiers", M. S.
Thesis March 1983.
3. J. Dormer, "Recording Very Fast Changes in Gas Composition", M. S. Thesis
March 1983.

THESES IN PROGRESS

1. V. Bojan, "A Real-Time Spectrum Averager for Use with a Time-of-Flight Mass
Spectrometer."
2. V. Rohrer, "Mass Spectra of Neutral Particles Released During Electrical
Breakdown of Spacecraft Insulating Materials."



PHYSICAL LAYOUT OF THE TOFMS

Figure 1.

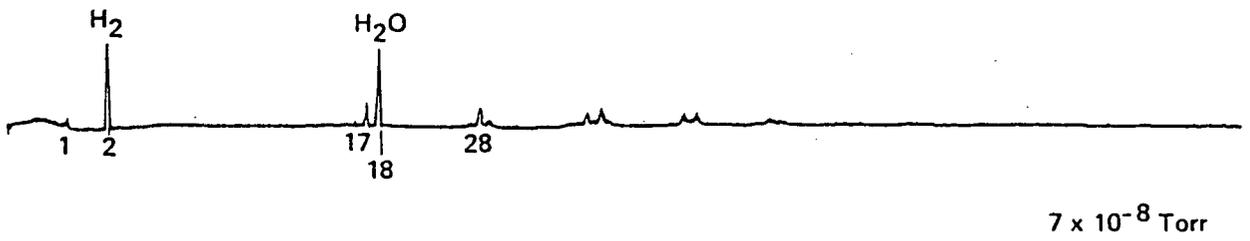


Figure 2. Mass Spectrum of Background Gases in Vacuum Chamber

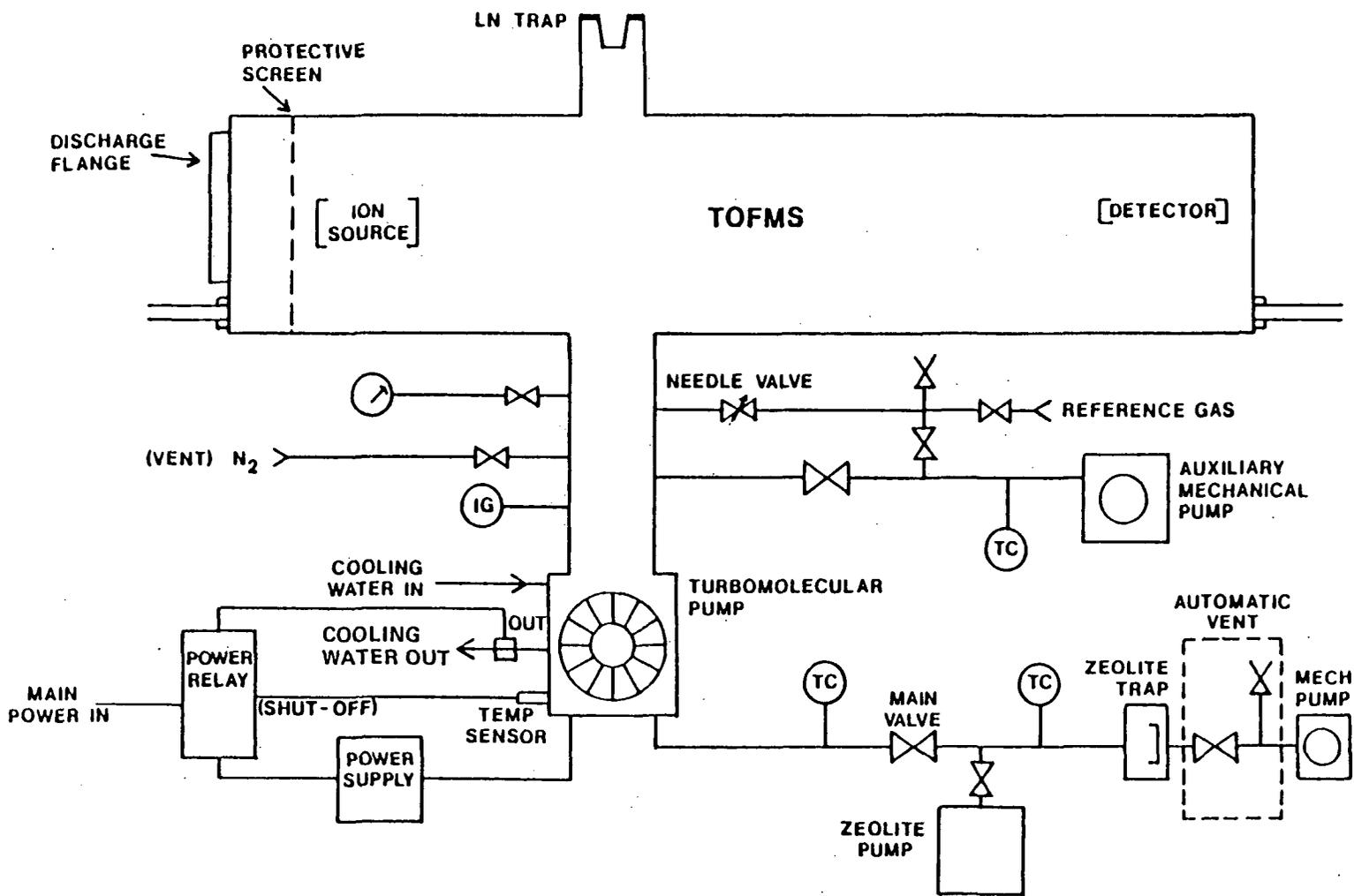


Figure 3. General Layout of Vacuum System

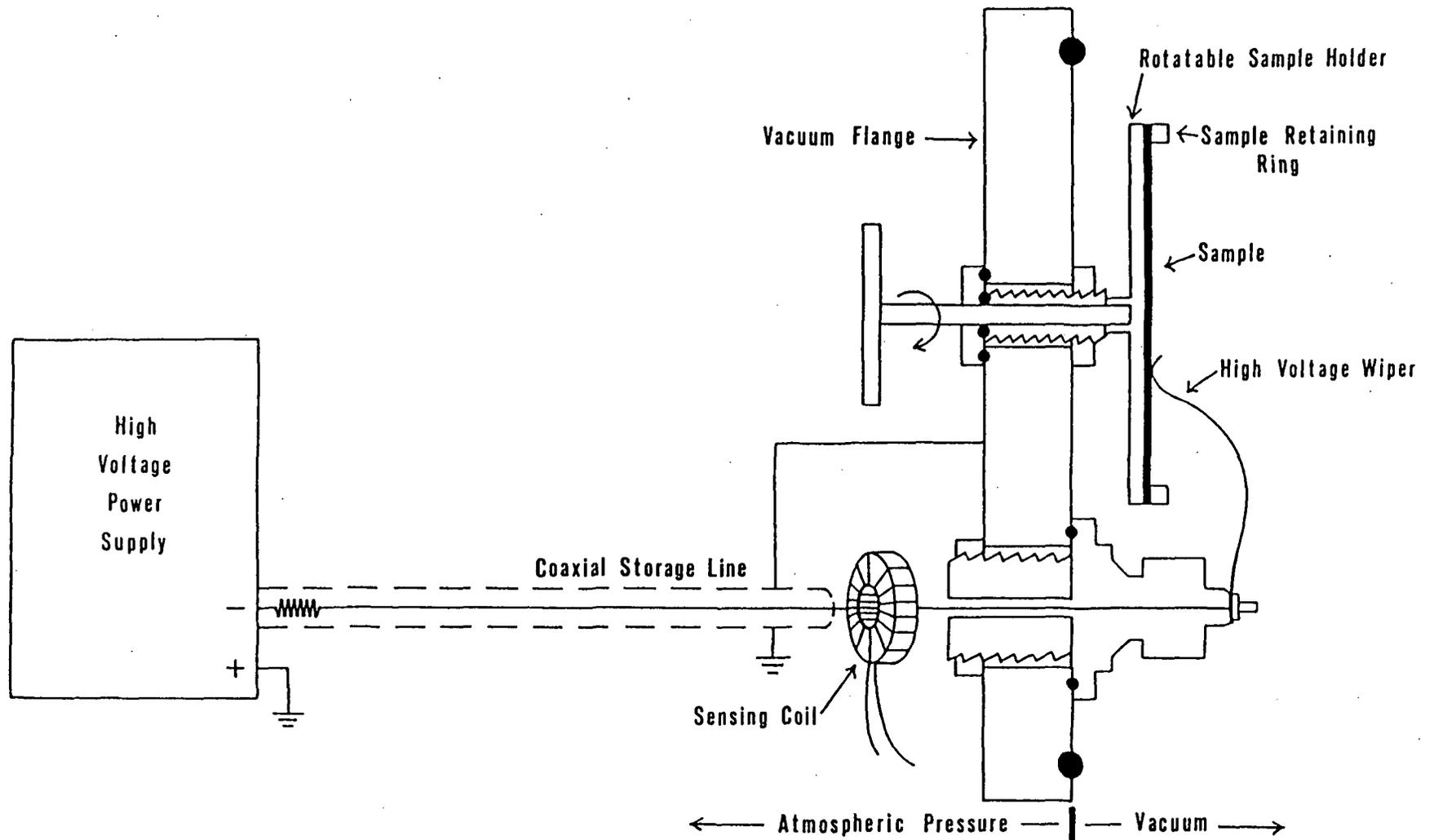


Figure 4. Direct-Contact Surface Breakdown Apparatus

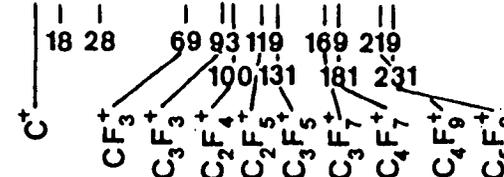
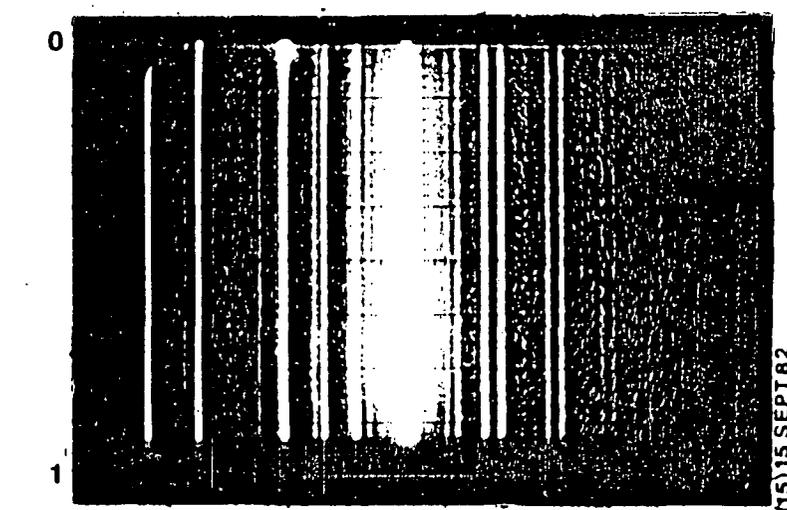
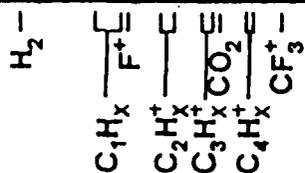
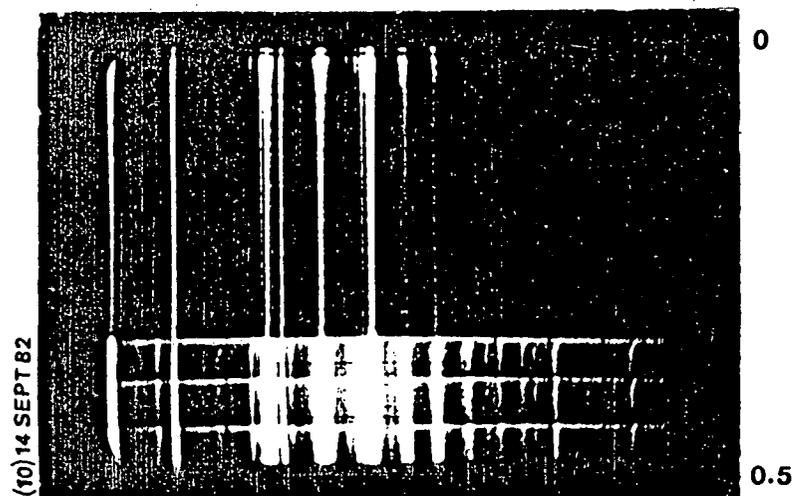
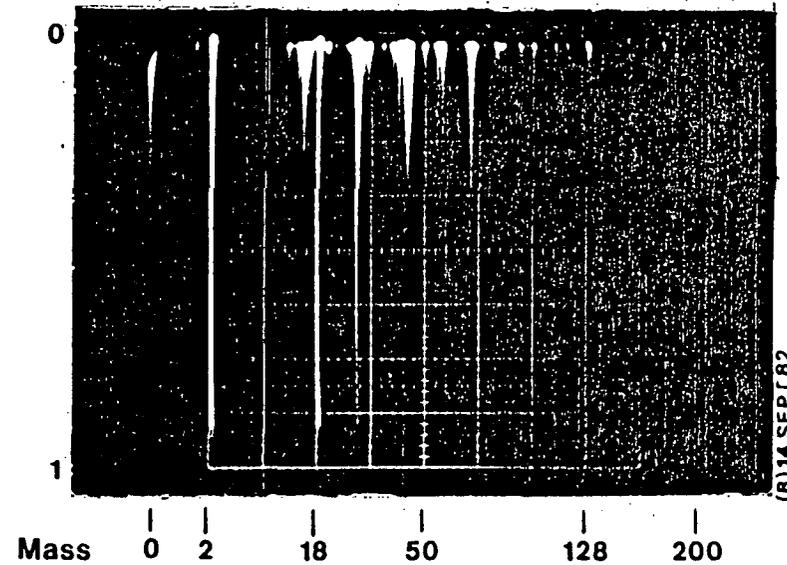
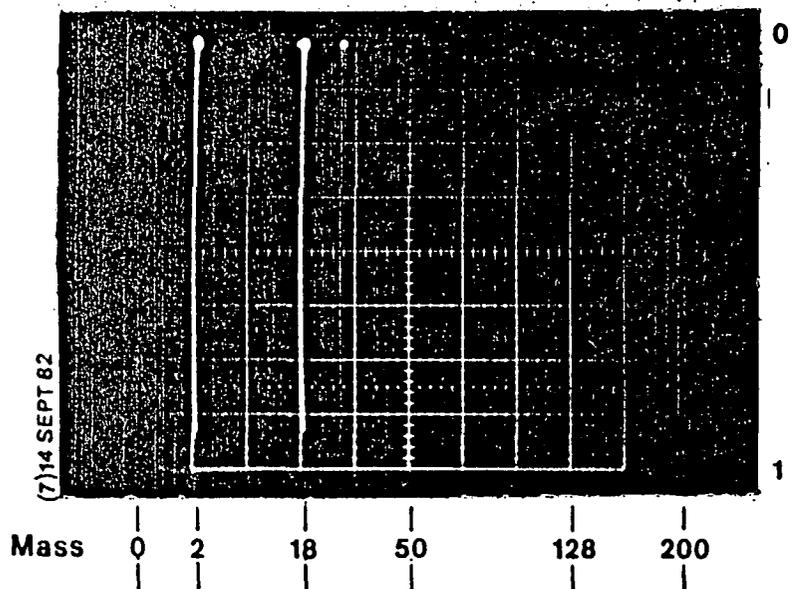


Figure 5. Variations with time of intensity - modulated mass spectrum associated with point-contact breakdown of 50μ Teflon FEP layer. Teflon PFA gives similar results.

Upper Left: Background spectrum, no breakdown. 9×10^{-8} Torr.

Upper Right: Breakdown at 13 KV. Approx. 0.01 joule.

Lower Left: Breakdown at 16KV. Multiple strike.

Lower Right: Continuous discharge through spark-damaged area of surface at 12-13KV.

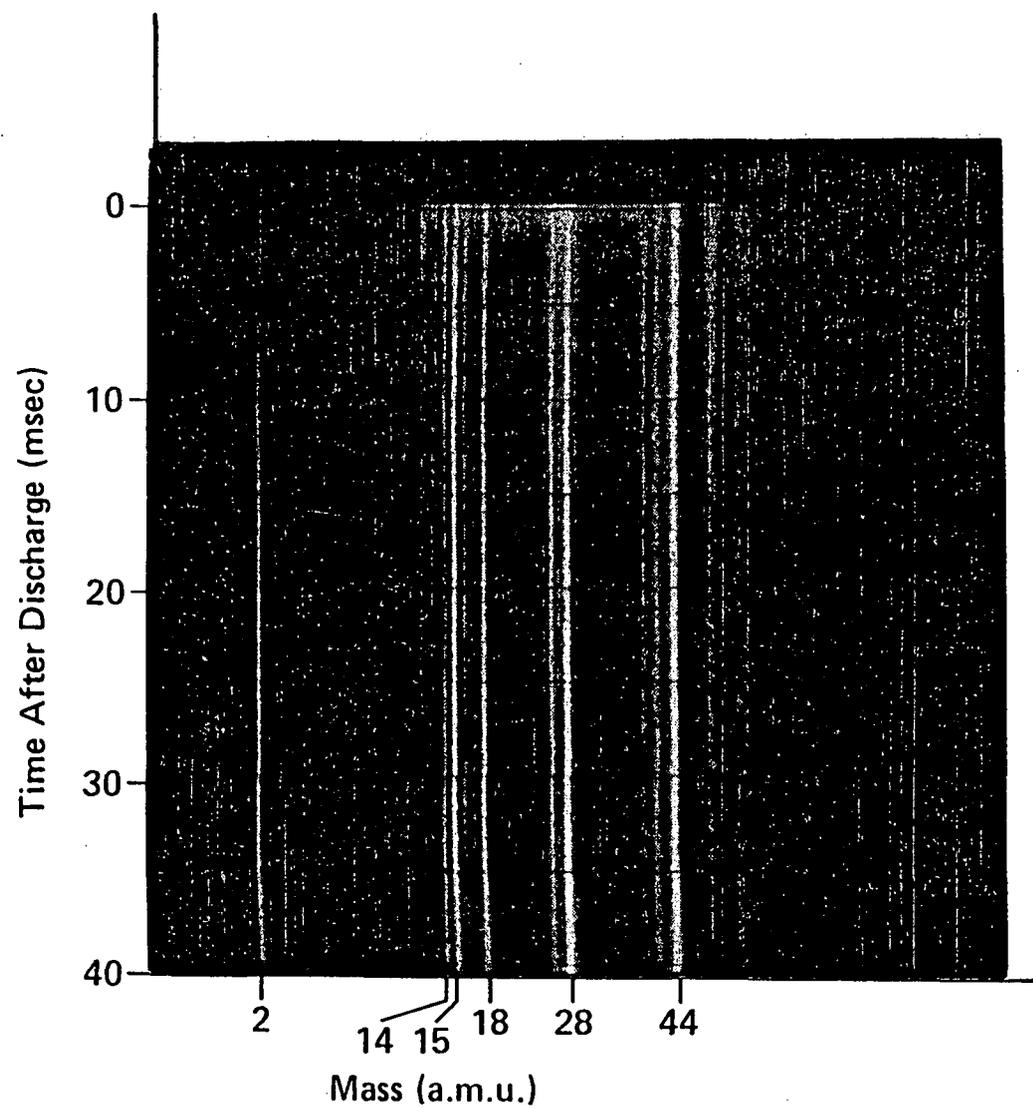


Figure 6. Intensity-Modulated Raster Display for Breakdown of 50 micron Kapton Film at 18 KV.
Main Peaks 44 (CO_2^+ , C_3H_8^+), 28 (CO^+ , C_2H_4^+), 15 (CH_3^+).

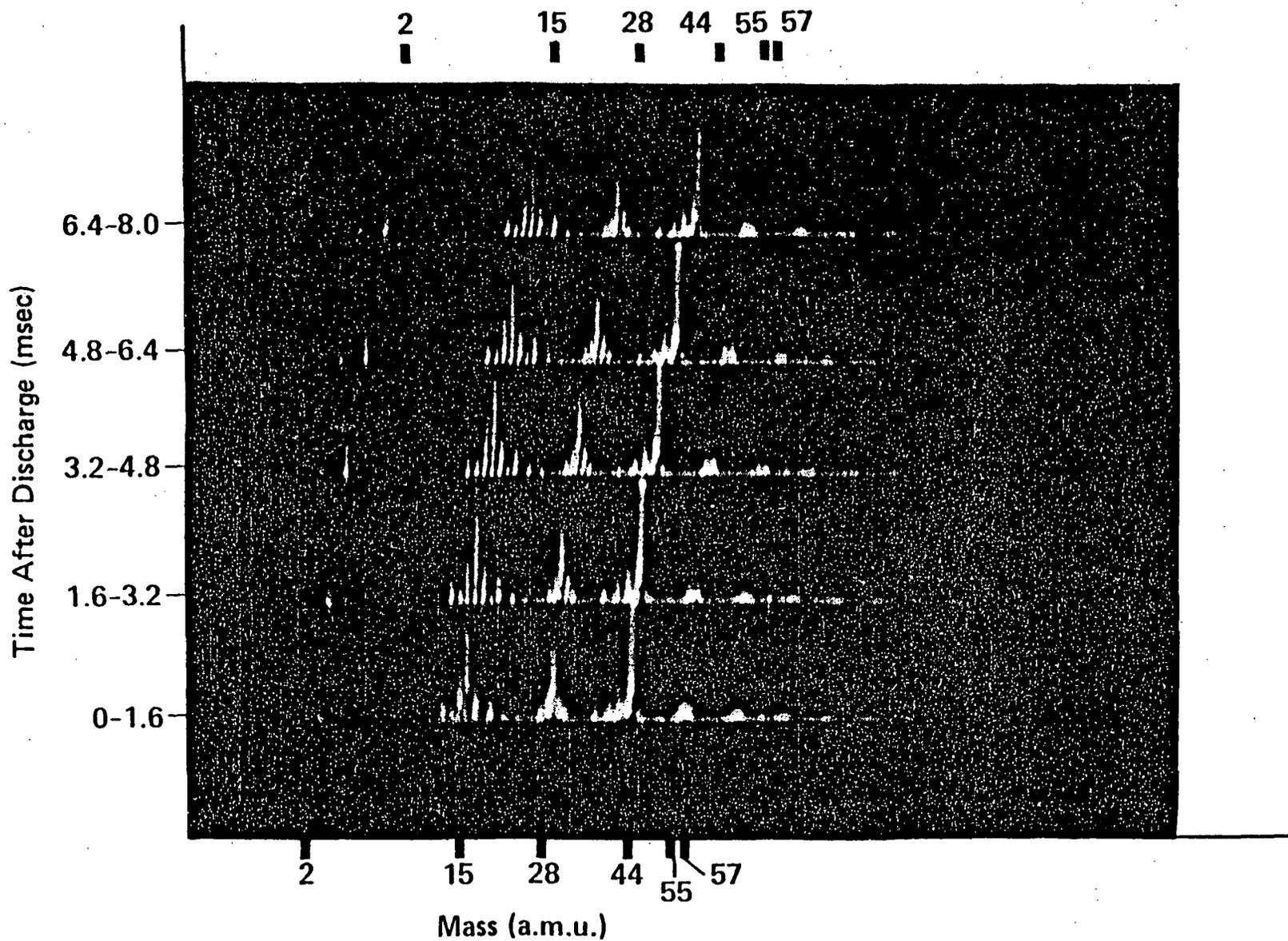


Figure 7. Contact Discharge Through 50μ Kapton Film at 16 KV. Offset Multitrace Raster Display, 16 Spectra per Trace.

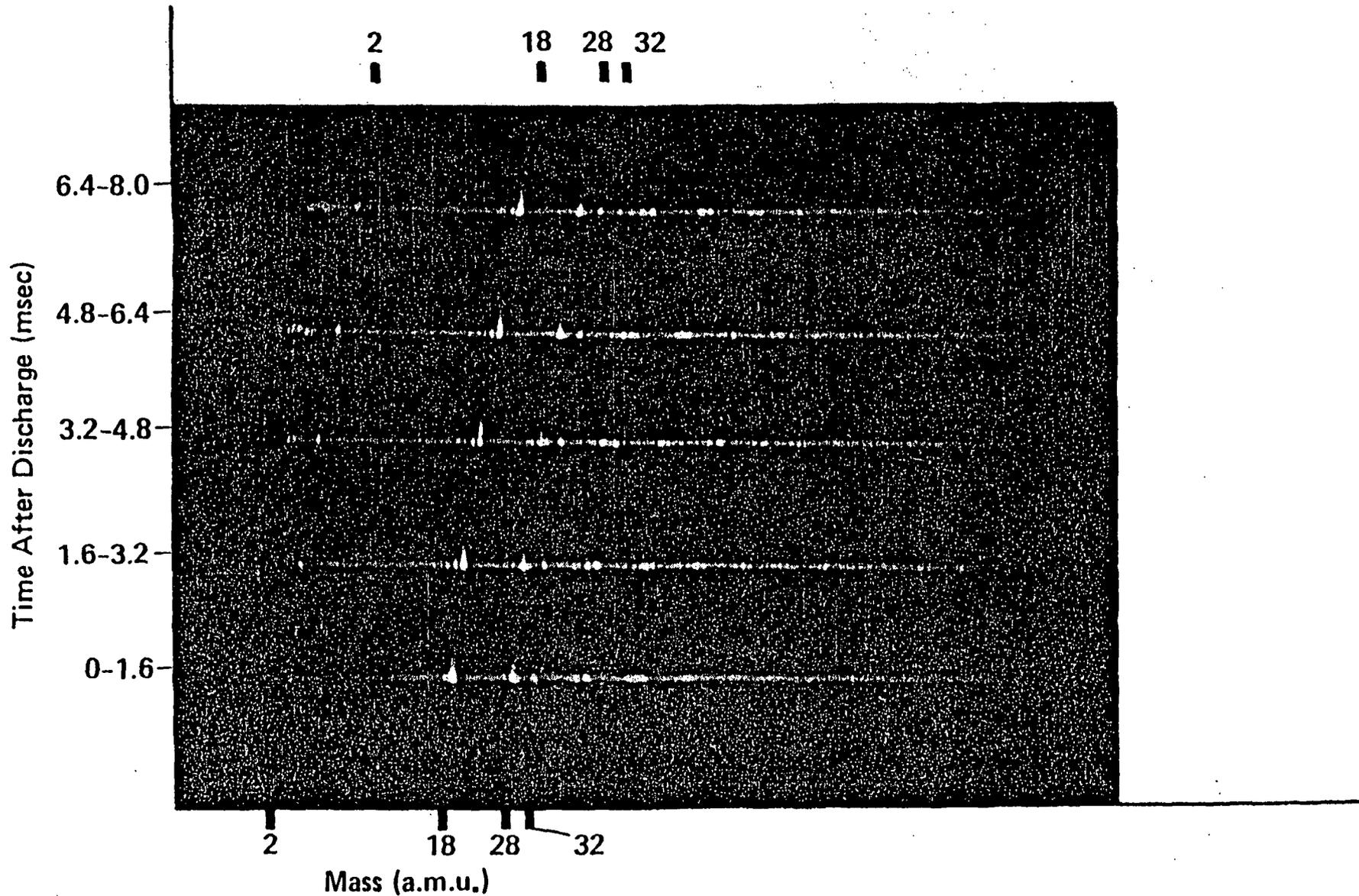


Figure 8. Background Gas Spectrum (No Discharge). Offset Multitrace Raster Display, 16 Spectra per Trace.

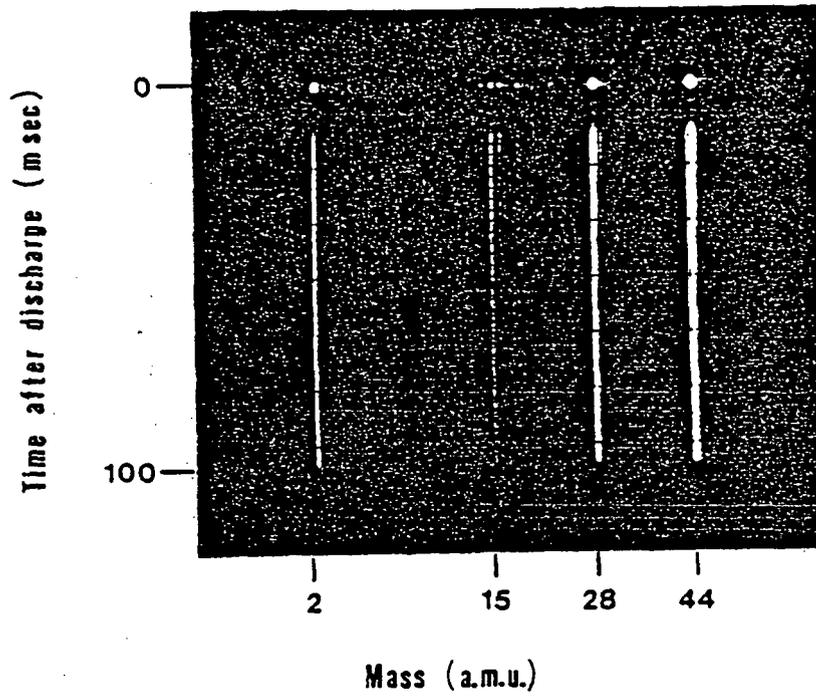


Figure 9. Intensity-modulated raster display of neutral mass spectra for breakdown of 50 micron Mylar film at 13 Kv. Main peaks 44 (CO_2^+ , C_3H_8^+), 28 (CO^+ , C_2H_4^+), 15 (CH_3^+) and 2 (H_2^+). Small peaks at masses 12, 14, 15, 16 appeared just before actual breakdown.

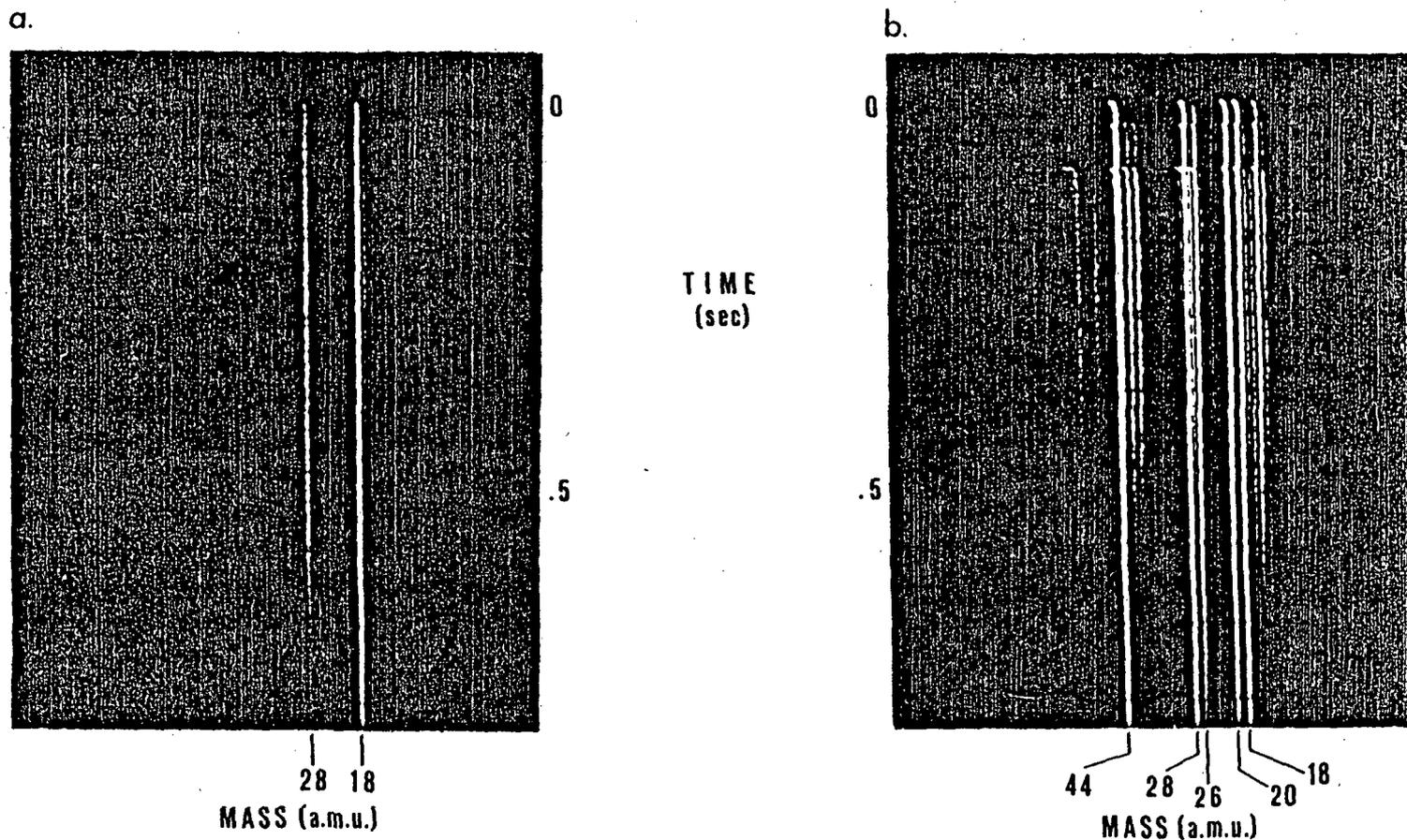
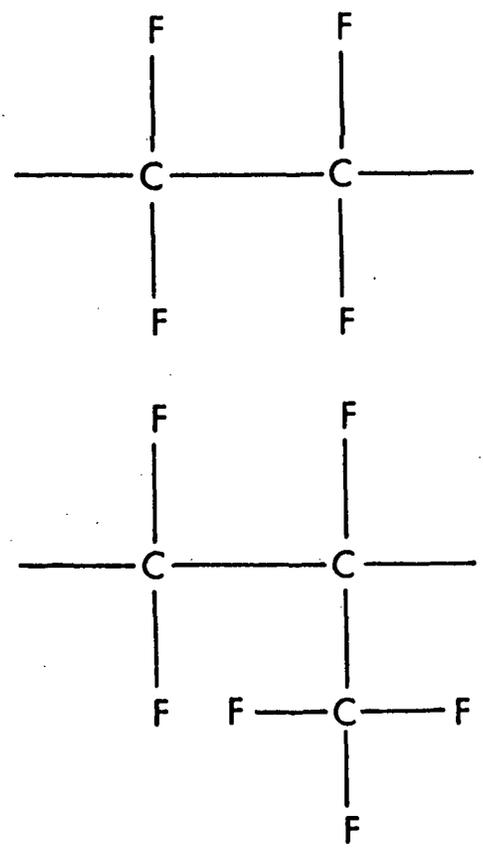
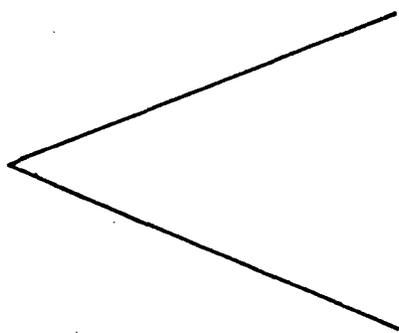


Figure 10. Intensity-modulated raster display of a.) background gas at 2×10^{-7} Torr and b.) breakdown of 50 micron Tefzel film at 18KV with main peaks at 44(CO_2^+ , C_3H_8^+), 26(C_2H_2^+), 20(HF^+). Mass 2 (H_2^+) present during breakdown but off scale.

TEFLON FEP: COPOLYMER



KAPTON H

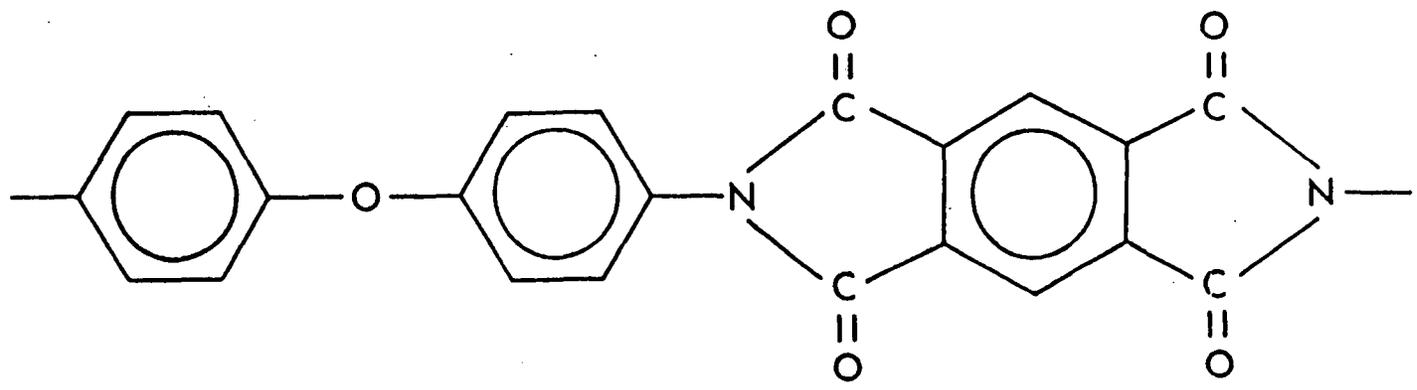
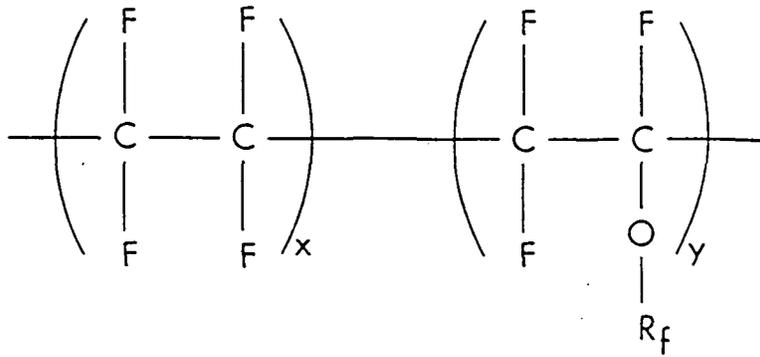
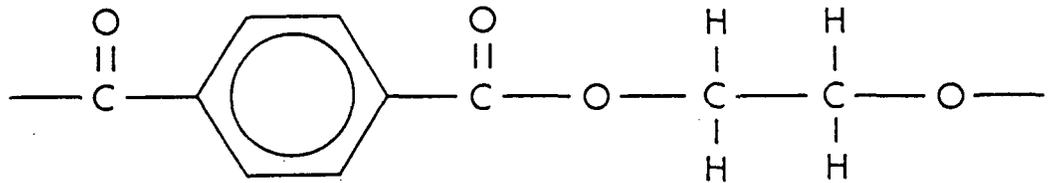


Figure 11. Polymer Structures. Hydrogen Bonds Omitted in Kapton Structure for Clarity.

TEFLON PFA



MYLAR



TEFZEL

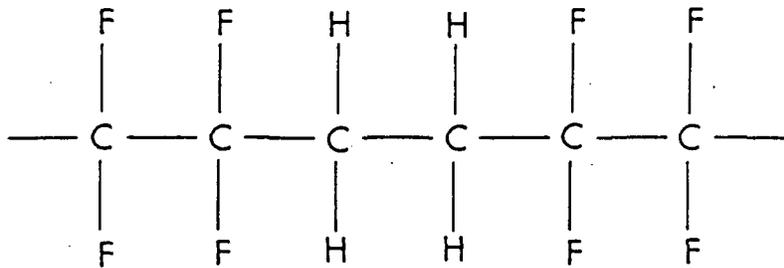


Figure 12. Polymer Structures. The OR_f in Teflon PFA represents a perfluoroalkoxy group.

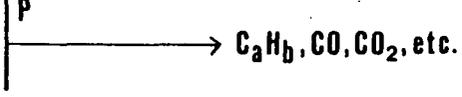
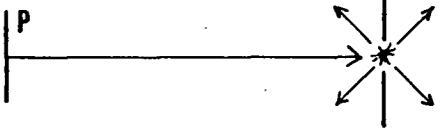
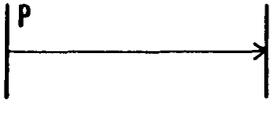
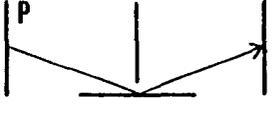
PHENOMENON	POLYMER	SYMBOLIC REPRESENTATION	COMMENTS
1. Intense neutral-particle pulse	Teflon Kapton Mylar Tefzel		Easily detectable by fast ion gauge.
2. Emission of heavy fragments	Teflon		> 350 amu
3. Emission of light fragments	Kapton Mylar Tefzel		≤ 44 amu
4. Secondary discharge	Teflon		Gases emitted from electrode surfaces.
5. Direct material transfer	Teflon		Change in secondary emission characteristics.
6. Indirect material transfer	Teflon		As for (5).
7. Removal of metal backing	Kapton		
8. Photon-induced desorption Electron-induced desorption	—		CO ₂ , CO, H ₂ , H ₂ O, CH ₄

Fig. 13 Neutral-particle phenomena observed during electrical breakdown of polymer films (contact charging).
Insulating side of metal-backed polymer films indicated by (P).