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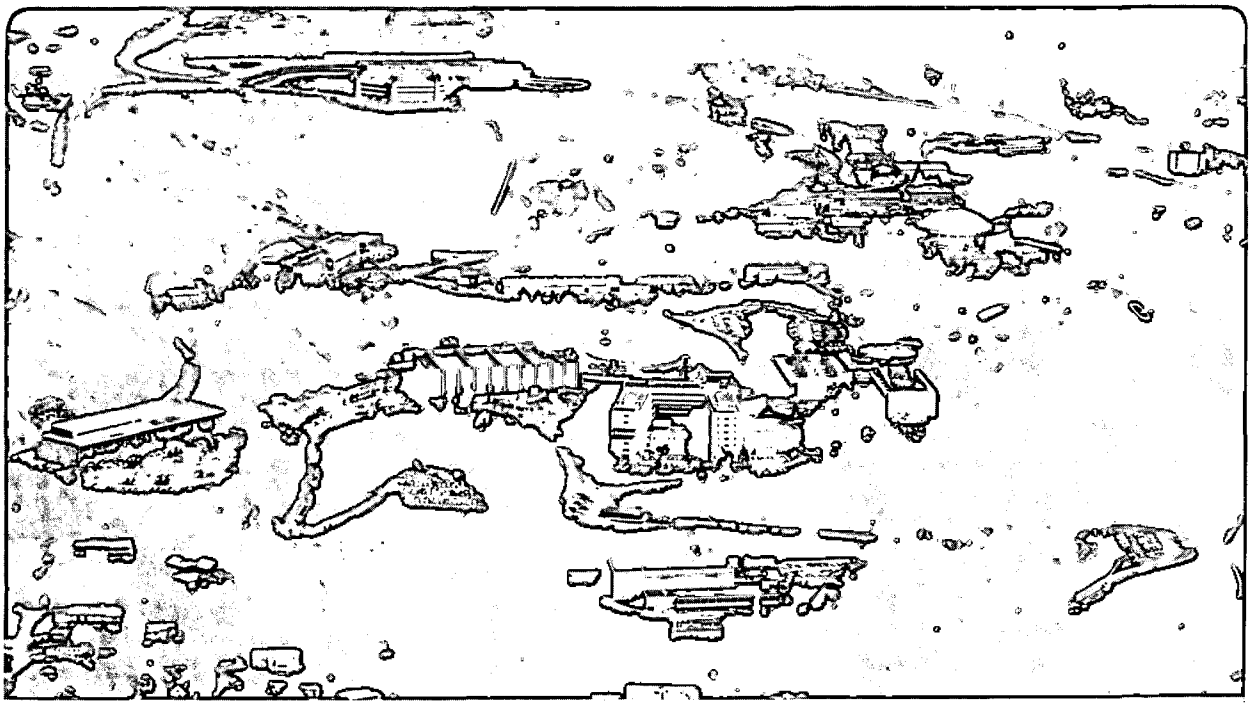

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Abstract

Thin film metal-oxide-silicon (MOS) devices were evaluated as detectors for microparticle impacts. The devices were fabricated with a range of thickness of oxide so that the sensitivity of the devices could be determined as a function of oxide thickness and bias voltage. Thin films of silicon dioxide (125 to 10000 Å) were thermally grown on phosphorus-doped silicon wafers and overlaid with Al (200 Å) or Ti/W (200 Å) and the integrity of the oxide film was evaluated by measuring device leakage current as a function of bias voltage. Devices characterized by a small leakage current, were subsequently bombarded with test particles which induced dielectric breakdown in devices with an oxide layer thinner than 500 Å. The nature of the breakdown was momentary, thus creating current pulses, but adequately energetic to destroy catastrophically the device at the point of impact. The catastrophic events vaporized a crater in the Al film surrounding the impact spot, rendering the spot electrically isolated and permanently inactive, while adjacent areas of the detector retained function. MOS devices fabricated with oxide layers ≤ 500 Å and coated with Al registered the impact of sonic velocity glass particles at least as small as 2 μm in diameter. Devices fabricated with Ti/W metallization did not register particle impacts but simply shorted permanently when bombarded with particles, presumably due to the lack of metal vaporization at the point where discharge through the oxide occurred.

Introduction

The detection of individual particles impacting a surface could provide a way to transform aerosol impactors into particle counters and an approach to study small particles with time-of-flight techniques. Such a detector could be used to detect particles as they are collected on the stages of a cascade impactor. A detector of this semblance also could be used as a "stop" detector to register particles after they travel through a flight tube. The real-time detection of individual microparticle impacts generated with a typical aerosol impactor has not been reported extensively in the literature,[1] perhaps because of the absence of appropriate detectors. Particle beam or mass spectrometry systems[2,3,4,5,6,7,8,9] sometimes used to study individual particles in vacuum would likely benefit from the development of such a detector. A common approach which circumvents the lack of detectors for measuring individual microparticle impacts is to measure the accumulation of particles on an impactor surface by measuring the current transferred to the surface while a continuous flow of particles is sampled.[10] If particles have unit charge, a pA corresponds to the collection of about 10^7 particles per second. Another approach is to impact particles onto an oscillating piezoelectric crystal and detect the accumulated mass by measuring the change in frequency of the oscillating crystal.[11,12] These two measurement approaches provide real time signals but do not respond to individual submicron-sized particles. To detect individual particle impacts, impact velocities much greater than those typically attained in an aerosol impactor must be

provided. Several devices designed to detect hypervelocity (velocity > 1 km/sec) particles have been reported. The devices detect particle impacts by impact ionization,[13,14] piezoelectrically,[15] and by film depolarization.[16] Such hypervelocity impact detection schemes work only for energetic impacts such as those produced by high voltage particle accelerators[17,18,19,20] or when cosmic dust impacts[21] onto detectors carried by spacecraft.

The work reported here addresses an experimental requirement we have to detect very large deoxyribonucleic acid (DNA) molecules after they are segregated according to mass/charge ratio in a mass spectrometer. Large DNA molecules in the gas phase might alternatively be viewed as microparticles. For reference, a 300,000 Da molecule of DNA (456 base pairs) weighs 5×10^{-19} g and, if collapsed into a ball, has a diameter of 0.01 μm. Molecules of this size and larger ones can be separated by gel electrophoresis but the analysis of such molecules by time-of-flight techniques is attractive because of potential time savings. Our desire to detect large DNA molecules extends to complete chromosomes but for our immediate application, the upper size requirement is to detect molecules or particles as large as 0.05 μm in diameter. This regime, corresponding to particles from 0.05 μm down to about 0.01 μm diameter, is currently very difficult, if not impossible, to detect in real time in reduced pressure environments such as in the vacuum environment of a mass spectrometer. The field of semiconductor fabrication would also benefit from the development of detectors for particles in this size regime. Particles commonly referred to as contamination particles[22] are generated in reduced pressure operations used to process silicon wafers,[23,24] and many are invisible to current particle detectors.

The present constraints for detecting particles in the 0.01 to 0.05 μm diameter size range in reduced pressure environments pertain to the way current particle detector technologies function. Below about 0.05 μm diameter, individual particles do not scatter light well and are therefore difficult, if not impossible, to detect in real-time via light scatter from a laser beam. If we assume that the smallest individual particle detectable via laser light scatter is 0.05 μm in diameter,[25] then only those DNA molecules larger than 3.1×10^6 Da are detectable by this approach. This excludes light scattering approaches for time-of-flight detection methodologies, except for extremely large DNA molecules. A second approach based on electrostatic acceleration could be considered but can be shown to be inappropriate for particles in this size range. Above about 0.01 μm diameter, charged particles or ions are too heavy to be accelerated to velocities that create secondary electrons upon impact with a surface. Electron multipliers or microchannel plates function only if ion impacts exceed a velocity threshold which is difficult to exceed with acceleration voltages used in most types of mass spectrometers.

An impact velocity threshold of 2×10^6 cm/sec is currently accepted as the lowest velocity that will produce at least one secondary electron for each incident particle.[26] When the regime of particulate matter between 0.01 μm and 0.05 μm is reconsidered, the application of secondary electron formation as an approach for detecting impacting particles is daunting. For example, a 0.01 μm diameter particle with a mass-to-charge (m/z, expressed as Da per number of charges) ratio of 8×10^5, charged to the electron limit of 44 electrons[27] and accelerated through 10^4 volts, attains a terminal velocity of only 2.5×10^5 cm/sec in vacuum, which is less than the threshold for secondary electron formation. In this example, a value of 10^4 volts was used because this is within the range of acceleration voltages of mass spectrometers. Larger accelerating voltages could be used but will not satisfactorily solve the velocity deficit problem because high voltage engineering and associated safety considerations make the high voltage approach undesirable for voltages in excess of 100,000 volts. The application of secondary electron detection appears more plausible if electron multipliers are used as the impactor surface. Zimmermann, et al.[28] recently detected singly

charged 2.5 x 10^6 Da Cesium atom clusters (m/z=2.5 x 10^6 , 0.017 μm diameter if a cluster density of 1.9 is assumed) with microchannel plates; however they did not report the detection efficiency of the large clusters, which could be small. Five hundred and ten thousand (510,000) Da singly-charged protein molecules, with an equivalent diameter of about 0.01 μm, have also been detected mass spectrometrically,[29] but again, this size of molecule is not detected efficiently with electron multipliers. Hopefully, these comments show that the constraints to the detection of particles using conventional electrostatic acceleration and impact detection via secondary electron formation are inadequate for detecting ions or particles in the 0.01-0.05 μm diameter range.

Metal-oxide-silicon (MOS) devices consist of a film of silicon dioxide, grown on a silicon substrate overlaid with a metal film. Thick film MOS devices, with oxide layers several μm thick, have been developed for the detection of hypervelocity particle impacts[30] and were used to generate information about the nature and origin of cosmic dust particles. The National Aeronautics and Space Administration and the European Space Agency launched these devices as part of the instrumentation packages of several space probes. The Long Duration Exposure Facility (LDEF) satellite was equipped with several MOS particle impact detectors.[31] Numerous particle impacts were recorded with MOS particle detectors as the Giotto spacecraft encountered the coma of Comet Halley.[32,33] The sensitivity of these devices was determined by bombarding them with hypervelocity particles produced with van de Graaff accelerators specially modified to accelerate electrically charged microparticles.

In the original work reported by Kassel,[30] MOS devices were fabricated with an oxide thickness of 4000 Å or 10,000 Å and an overcoat of 1000 Å of aluminum. Bombardment of these devices with high velocity particles (1 - 9 km/sec) generated in a van de Graaff accelerator, indicated that for equal bias voltage, the 4000 Å oxide device was more sensitive to lower velocity microparticle impacts than the thicker oxide device. From their velocity threshold data for 4000 and 10,000 Å devices, we determined that the limit of detection for glass microparticles with the 4000 Å devices can be described with the following equation:

$$\log Dp = -1.81 \log V + \log 2.6$$

in which Dp = particle diameter in microns, V = particle velocity in km/sec and 2.6 is the minimum particle diameter (in microns) detected when V = 1 km/sec. For a given velocity, this equation predicts that particles ≥ Dp will be detected. From their data we also determined that a similar equation describes the limit of detection for glass microparticles with the 10,000 Å oxide devices as follows:

$$\log Dp = -1.81 \log V + \log 5.2$$

in which Dp and V are as indicated before and 5.3 is the minimum diameter (in microns) particle detected when the impact velocity is 1 km/sec. These two equations describe particle diameter and impact velocity relationships which when plotted on log-log graph paper are parallel. We extrapolated their data to smaller particles by reducing the minimum size particle detectable at 1 km/sec by a factor of 2 for each factor of 2.5 decrease in the oxide thickness. For example, the minimum size particle impacting at 1 km/sec detected with the 10,000 Å oxide detector is 5.2 μm. The minimum size decreases to 2.6 μm for 1 km/sec particles impacting a 4000 Å oxide device. We stepwise decremated the minimum size by 1/2 for each subsequent decrement by 1/ 2.5 of oxide thickness. In this fashion, we estimated that the minimum particle detected a 1 km/sec for a 1600 Å oxide MOS device would be 1.3 μm. Continuing in this way, a 100 Å oxide/ 1000 Å

aluminum device could register the impact of a 0.18 μm particle traveling at 1 km/sec. MOS devices with thinner aluminum layers than they used is expected to improve sensitivity further. This extrapolation is not part of the original work and its validity is questionable.

We have attempted to extend the development of MOS particle detectors to the detection of smaller particles impacting at much lower velocities than previously reported, hoping to improve their sensitivity and apply them to the detection of massive molecular ions of DNA. The extension of impact particle detection to small particles and massive ions fills a niche of particle sizes not currently detectable in reduced pressure applications. This article is intended to be primarily of interest to experimentalists and presents descriptively a new approach to massive ion and small particle detection.

Device Fabrication

MOS devices were fabricated on silicon wafers using standard silicon planar processing techniques. Figures 1 and 2 show schematics of the device from top-view and cross-section perspectives. The thin oxide films were thermally grown on 500 μm thick phosphorus-doped silicon of 3-5 ohm-cm resistivity. The oxide under the 5 x 5 mm pad areas ranged from 125 Å to 10,000 Å thick, and were defined using standard photolithographic techniques. The pad areas were overlaid with 200 Å aluminum or Ti/W, while the bonding pad areas and guard ring were 5000 Å thick. The backside contact consisted of 1000 Å aluminum. The devices were diced into approximately 1 x 1 cm squares and the active area and guard ring were wire-bonded to a small IC socket for electrical contact, as shown in Figure 1a.

Testing Procedures

The leakage current was monitored during bias testing and particle bombardment using the simple voltage divider shown in Fig. 2. An oscilloscope was capacitively coupled, as shown, to detect pulses in the leakage current that corresponded to impact events. A 5 V transorb was incorporated in the circuit to prevent voltage excursions from exceeding the rating of the analog-to-digital converter (ADC) (National Instruments, LabPC board, sampling rate 4.8 k Hz). A second ADC recorded the bias voltage of the MOS detector. The digitized time record was stored in computer memory. The computer board also had several digital-to-analog channels, one of which was used to control the bias voltage across the MOS detector (negative voltage applied to the bottom metal layer). An aerosol impactor was constructed which could support a MOS detector at adjustable distances underneath a nozzle and used it to bombard MOS detectors with particles at near sonic velocity (~ 330 m/s) as estimated from the pressure drop across the impactor nozzle. The impactor nozzle consisted of a 0.5 mm id. x 5 cm long metal capillary tube and the MOS detector was located 1.5 nozzle diameters below the nozzle exit. The impactor was operated at an internal pressure of 1 cm Hg absolute, and at this pressure, free-jet expansion produces near sonic flow, thus accelerating the particles to high velocity. A flow stream of particles (2, 20 or 50 μm particles, Duke Scientific, Palo Alto, CA) was produced by nebulizing, and then drying, particles suspended in filtered deionized water or by directing a burst of gas from a micro-duster (Texwipe, Co.) towards a surface to resuspend a few dry particles off the surface. The nebulized flow or the gas burst blew particles into a vertically mounted cylinder that guided particles, as they settled in room air, towards the inlet of the impactor. Filtered air could also be sampled through the impactor to determine if pulses were induced by particle impacts or were intrinsic breakdown events. The response of the MOS devices to particle impacts was determined as a function of detector bias voltage and particle diameter.

Detection of Particle Impacts

The following presentation deals primarily with the preliminary evaluation of devices fabricated with a top metallization of aluminum. Results obtained with devices metallized with Ti/W are also presented but these are more brief. The flow of current through the oxide, i.e., the leakage current, indicates the integrity of the oxide in a MOS device. Oxide films with pin hole imperfections become electrically shorted when coated with a layer of metal because the metal contacts the underlying doped silicon. If non-shorting defects exist in the oxide, the oxide might still be an insulator but is not a high dielectric oxide. Defective areas or spots in the oxide conduct more current than a robust oxide and the presence of such defects is determined from leakage current vs. bias voltage measurements. A typical plot of leakage current vs. bias voltage for a 500 Å oxide device is shown in Fig. 3. A detectable leakage current (> 0.1 μA for this measurement system) is observed above 33 volts for this MOS detector. As the bias voltage is increased above 33 V for this device, the leakage current increases and momentary breakdown in the oxide is indicated as brief pulses rising above the steadily rising leakage current. The momentary breakdowns in the oxide observed when bias voltage is applied to a new detector for the first time are generally attributable to defects in the oxide. The transient nature of the breakdown event is assumed to be caused by a restorative process during which time the defect, i.e., the site of discharge, is removed by resistive heating which vaporizes a small hole in the Al film. The vaporization of Al disconnects the conduction path from electrical contact. This process is sometimes referred to as a "burning-in" process. The device tested in Fig. 3 eventually failed irreversibly at a bias voltage near 48 volts and became a device acting like an electrical resistor. Typically, the bias voltage of a MOS device is ramped up to "burn out" some of the defects but not biased so high as to permanently destroy it. Curves similar to that shown in Fig. 3 were obtained for MOS detectors fabricated with different thicknesses of oxide. As expected, the leakage current at a given bias voltage is higher for thinner oxides. In other words, the curve in Fig. 3 is shifted to the right for thicker oxides and to the left for thinner ones. The thinner oxide devices fail permanently at lower relative bias voltage than thicker layers. A number of MOS detectors with oxide layers 500 Å or less were defective from the start and acted like pure resistors with leakage currents of several μA below 5 volts. MOS detectors made with oxides thicker than 500 Å were more robust as suggested by the observation that fewer of them were defective from the start. This is not unexpected because thin robust oxides are more difficult to fabricate than thick ones.

In Fig. 4 the distribution of the onset of irreversible dielectric breakdown is shown as a function of oxide thickness. This data was collected by bias ramping the MOS devices in the manner described above. For each thickness of oxide tested, several devices were shorted from the start which are indicated by a near zero breakdown voltage. The average electric field strength for the oxides at breakdown for 100, 200, 500, 1000, and 10,000 Å oxide layers are 5.0×10^8, 4.8×10^8, 4.6×10^8, and 5.5×10^8 V/m, respectively. The intrinsic breakdown field strength is on the order of 10^9 V/m for silica. Our experimentally determined values for breakdown field strength compare favorably to attainable electric field strengths reported for silicon dioxide[34,35] and demonstrate that high quality oxide films were fabricated.

Some of the first devices were tested with particle bombardment by sprinkling 20 or 50 μm diameter glass particles above the inlet of the impactor. This generally proved disastrous because these particles, or clumps of these particles, rendered the detectors inoperable in a few seconds. Impacts of these large particles abraded a small spot in the Al layer of the MOS detector, creating a discontinuous Al film underneath the impaction jet. The abrasion destroyed the MOS capacitor at the site where particles impacted the MOS detector, yet the detector still functioned when particles

struck a fresh location on the detector. A few experiments demonstrated that reducing the jet velocity prevented abrasion yet allowed the impacts to be recorded. Following this observation, new detectors were bombarded with 2 μm particles traveling nearly at sonic velocity, knowing that the impacts of 20 or 50 μm diameter particles were easily detected. This choice allowed us to evaluate future refinements to the MOS device fabrication process that might lead to detectors that register even smaller particles.

A bias voltage as large as possible was applied to the MOS detectors in order to make the detectors sensitive to particle impacts. This approach carried the risk that the detectors would self-destruct due to intrinsic breakdown. Typically, a bias voltage in the range of 70-90% of the intrinsic breakdown field strength of the oxide was applied. Detectors fabricated with 100, 200 or 500 Å oxide showed similar sensitivity to particle impacts but the 100 Å oxide detectors were inherently more noisy because their leakage currents showed greater fluctuations. Sensitivity to particle impacts was greatly reduced for the 1000 Å oxide detectors and impacts were not detected with 10,000 Å oxide detectors. This measure of limited detector sensitivity was based on the observation that only a few impact events led to electrical pulses with 10,000 Å oxide device even though many particles were seen sticking to the impaction surface.

Low leakage current (< 2 μA at 80% of breakdown field strength) MOS detectors were subjected to particle bombardment using the aerosol impactor. By alternatively sampling particle-free or particle-laden air streams, the electrical response of a MOS detector could be attributed to particle impacts. In Fig. 5 a time record is displayed which shows the sampling, first of particle-free air, followed by air carrying 2 μm glass particles. The first 10 sec of the record shows that no pulses were produced while particle-free air was sampled through the aerosol impactor, but during the remaining part of the record, pulses were generated by particle impacts. The pulses are momentary and indicate the impact of individual particles. Examination of the metal surface of MOS detectors by electron microscopy revealed that impacts led to crater formation in the Al film. This type of crater formation has been correlated with localized electrical discharge of the MOS capacitor.[36]

Several related mechanistic explanations[37,38,39] help to clarify the physical processes that lead to electrical discharge through the oxide and create the type of pulses seen in Fig. 5. Compression of the oxide at the moment of impact causes the electrical field strength to increase leading to electrical discharge through the oxide at the point of compression. During compression, a few free charge carriers are accelerated in the relatively larger electric field created in the compressed oxide to an energy sufficient to generate secondary charges via electron impact ionization. The result is a growing cascade of charge flowing through the compressed oxide, culminating in discharge of the MOS capacitor through the compressed spot. The discharge is adequately energetic to heat locally the oxide and metal to a temperature high enough to vaporize a spot in the Al film. This process is self healing in that any metal surrounding the impact spot is removed, thus lowering the electrical field near the impact and ending the discharge. The result is the formation of a crater devoid of metal. These craters were approximately 10 μm in diameter and their centers frequently contained the particle that presumably initiated the discharge event. The formation of craters correlated with the type of pulses recorded in plots like that in Figs. 5 and 6. These pulses are nearly equal in magnitude suggesting that each event completely discharged the MOS capacitor. An alternative explanation for oxide breakdown during impact involves the concept of charge-trapping sites in the oxide. Such sites might be considered as a "cage" for an electron and the impact of a particle creates phonons which thermally stimulate the trapped electron to a slightly higher energy level, allowing it to escape from the trap. The freed electron is accelerated in the electric field, creating new ions along its course via electron impact ionization which eventually causes complete

breakdown. Regardless of which explanation better describes the oxide breakdown process, each mechanism points to the critical role played by the oxide layer. A detectable signal will result only if the particle transfers energy to the oxide. If the kinetic energy of the particle is dissipated in the metal layer, then it is unlikely that an impact will be registered.

The magnitude of the pulses resulting from discharge events increased as the bias voltage increased, an effect shown in Fig. 6, and is attributable to the increasing stored electrical charge. At the time of discharge, an amount of current determined by the bias voltage and the electrical capacitance of the MOS detector, flows through the voltage divider. As the bias voltage was ramped above 25 volts, 15 μA pulses appeared but at higher bias, voltage clipping in the measuring circuit limited peak height to 20 μA.

Discharge produced craters in the Al layer that were typically about 10 μm in diameter. These craters are inactive spots on the surface of a MOS device and subsequent impacts will produce more. Eventually the efficiency of the detector will be reduced because the inactive area becomes a significant fraction of the surface of the detector. The MOS devices were fabricated with 0.25 cm^2 active area which means that about 30,000 impacts would reduce the active area by 10 percent. Depending on the application, this could be an acceptable constraint for the operation of these detectors. The fabrication of devices with larger active areas or the fabrication of an array of detectors could solve this problem.

The MOS devices require further study to determine their useful range of operation. The smallest particles detectable with impact velocities resulting from jet expansion in an impactor needs to be determined. This information will lead to an assessment of the applicability of MOS detectors in impactors. Although the devices eventually are destroyed by the accumulation of craters, they may find application in low number concentration sampling conditions. A second mode of failure stems from chemical reactions that change the conductivity of the aluminum layer. Aluminum MOS devices, as fabricated for this study, have a limited shelf life. Devices which resided in a plastic storage box failed to respond to particle impacts when used for the first time after about three months of storage. Fabrication of a subsequent batch of detectors confirmed that the devices worked satisfactorily when they were new, but indeed failed after several months of storage at room conditions. The cause of failure was due to the oxidation of the aluminum layer which converted the aluminum into aluminum oxide, thus changing its electrical properties from a metal to a refractory insulator. In an attempt to solve the oxidation problem, MOS devices were fabricated with a Ti/W layer, instead of an aluminum layer. These devices were tested, first by measuring the leakage current vs. bias voltage, and then by bombarding them with particles. The Ti/W devices displayed similarly low leakage currents as did the devices fabricated with aluminum, but they did not produce many pulses when bombarded with 2 μm diameter glass particles. Instead, they simply failed as soon as particle bombardment started, even at very low bias voltage such as 4 volts. These observations led to the conclusion that oxide breakdown in these devices is not a reversible process and the only reason the aluminum devices worked was that the discharge vaporized the aluminum away from the point of discharge, and in essence, removed electrical contract from the place where discharge occurred through the oxide. Ti/W is a higher melting alloy than aluminum and the heat released during discharge was insufficient to vaporize the Ti/W at the point of discharge. Electron microscopy revealed that only a few craters, surrounded with semicircular blisters, formed on the Ti/W surface of a shorted device when it was operated at a bias voltage that would have produced many craters on an Al MOS device. The central craters were relatively small and probably allowed the Ti/W metal to remain in contract with the site of discharge. Whereas, the vaporization of aluminum allowed those devices to retain their insulating

layer, the Ti/W devices became pure resistors. Aluminum is thus a key component in the MOS particle detector and we are now considering ways to extend the shelf life of these devices.

Conclusion

A 5 x 5 mm thin film MOS devices displayed a response to microparticle impacts and the tests demonstrated that this type of microparticle detector could be applied to the real time measurements of microparticle impacts generated with an aerosol impactor. The detectors registered the impact of particles at least as small as 2 μm in diameter traveling at near sonic velocity (330 m/s). The thinner oxide (≤ 500 Å) devices were the most sensitive with respect to particle detection, although several 1000 Å MOS detectors showed a limited response to microparticle impacts. In general, it was observed that devices fabricated with relatively thick oxides (1000-10,000 Å) were more robust and could be biased to higher absolute voltages before failure, but the thickest oxide detectors tested (10,000 Å) did not respond to particle impacts even with particles as large as 50 μm at sonic velocity. All thicknesses of oxide showed about the same maximum dielectric field strength before breakdown occurred. Particle impacts onto Al metallized MOS devices produced momentary, catastrophic electric breakdown of the oxide during particle bombardment when the electric field across the oxide approached the breakdown field strength for silicon oxide. The catastrophic events vaporized a crater in the metal film surrounding the impact spot, rendering the spot permanently inactive, but allowed undamaged areas of the detector to remain functional.

The results of this preliminary investigation indicate that MOS devices fabricated with an Al top layer function as particle impact detectors. The sensitivity of these devices is thought to be extendible to the detection of particles smaller than 2 μm in diameter and perhaps to massive ions. We plan to extend the testing of these detectors to include particles less than 2 μm and to improve the reliability and sensitivity of the MOS devices in an attempt to use them as detectors for large DNA molecules in time-of-flight mass spectrometers.

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[1] Lastow, O. (1994). Single Particle Detection by Impaction on a Piezoelectric Sensor, Proceedings of the Fourth International Aerosol Conference, p. 540, International Aerosol Research Assembly and the American Association for Aerosol Research, Los Angeles, CA

[2] Allen, J. and Gould, R.K. (1981). Mass Spectrometric Analyzer for Aerosol Particles, Rev. Sci. Instrum., 52, 804-809.

[3] Davis, W.D. (1977). Continuous Mass Spectrometric Analysis of Particulates by Use of Surface Ionization, Environ. Sci. Technol., 11 587-592.

[4] Davis, W.D. (1977) Continuous Mass Spectrometric Determination of Concentrations of Particulate Impurities in Air by Use of Surface Ionization, Environ. Sci. Technol., 11 593-596.
[5] Sinha, M.P., Giffin, C.E., Norris, D.D., Esters, T.J., Vilker, V.L., and Friedlander, S.K. (1985). Particle Analysis by Mass Spectrometry, J. Colloid and Interface Sci., 87, 140-153, 1982.
[6] Sinha, M.P., and Friedlander, S.K. Anal. Chem., 57, 1880-1883.
[7] Murphy, D.M. and Thompson, D.S. (1995). Laser Ionization Mass Spectroscopy of Single Aerosol Particles, Aerosol Sci. and Technol., 22, 237-249.
[8] Stoffels, J.J. and Lagergren, C.R., (1981). On the Real-time Measurement of Particulates in Air by Direct-inlet Surface-ionization Mass Spectrometry, Int. J. Mass Spectrom. Ion Phys., 40, 243-254.
[9] Prather, K.A., Nordmeyer, T. and Salt, K. (1990). Real-time Characterization of Individual Aerosol Particles Using Time-of-flight Mass Spectrometry, Anal. Chem., 66, 1403 -1407, 1994.
[10] Fernandez de la Mora, J., Hering, S.V., Rao, N. and McMurray, P.H. (1990). Hypersonic impaction of ultrafine particles, J. Aerosol Science, 21, 169-187.
[11] Lundgen, D.A., Carter, L.D., Daley, P.S. (1976). Aerosol Mass Measurement Using Piezoelectric Crystal Sensors, In Fine Particles, ed. B.Y.H. Liu, New York, Academic Press 485-510.
[12] Ward, D.M. and Buttry, D.A. (1990). In Situ Interfacial Mass Detection with Piezoelectric Transducers, Science, 249, 1000-1007.
[13] Adams, N. G., and Smith, D. (1971). Studies of Microparticle Impact Phenomena Leading to the Development of a Highly Sensitive Micrometeoroid Detector, Planet. Space Sci., 19, 195-204.
[14] Iglseder, H., Igenbergs, E. (1987). Measured Charge Generation by Small Mass Impact at Velocities Between 1 and 45 km/s, Int. J. Impact Engng., 5, 381-388.
[15] McDonnell, J.A.M. (1987). The Giotto Dust Impact Detection System, J. Phys., E., 20, 741-758.
[16] Tozzolino, A.J. (1992). PVDF Copolymer Dust Detectors: Particle Response And Penetration Characteristics, Nuclear Instrum. Meth. in Phys. Res., A316, 223-237.
[17] Slattery, J.C., Becker, D.G., Hammermesh, B. and Roy, N.L. (1973). A Linear Accelerator for Simulated Micrometers, Rev. Sci. Inst., 44, 755-762.
[18] Friichtenicht, J.F. (1962). Two-million-volt Electrostatic Accelerator for Hypervelocity Research, Rev. Sci. Inst., 33, 209-212.
[19] Shelton, H., Hendricks, C.D., Jr. and Wuerker, R.F. (1960). Electrostatic Acceleration of Microparticles to Hypervelocity, J. Appl. Phys., 31, 1243-1246.
[20] Keaton, P.W., Idsorek, G.C., Rowton, L.J., Sr., Seagrave, J.D., Stradling, G.L., Bergeson, S.D., Collopy, M.T., Curling, H.L., Jr., McColl, D.B. and Smith, J.D. (1989). A Hypervelocity-mircoparticle-impacts Laboratory with 100-km/sec Projectiles, Los Alamos National Laboratory report LA-UR-89-3952, Los Alamos National Laboratory, Los Alamos, NM.
[21] See a special issue dedicated to this study, Nature, 15 May 1986
[22]Clark L.A., Hastie, T., Psotakelty, L.A., Sinclair, J.D. and others (1992). Sources of Particle Contamination in an IC Manufacturing Environment, Aerosol Science and Technology, 43-50.
[23] Vander Wood, T.B. (1985). Identification of Particulate Contaminants in IC Manufacture, Solid State Technology, 177-182.
[24] Xu, Y., Bae, Y.K., Beuhler, R.J. and Friedman, L. (1993) Secondary Electron Analysis of Polymeric Ions Generated by an Electrospray Ion Source, J. Phys. Chem., 97, 11883-11886.
[25] K.A. Prather, Fergenson, D.P., Morical, B.D., Noble, C.A., Nordmeyer, T. and Salt, K. (1995). Analysis of Ambient Aerosol Particles using Aerosol Time-of-flight Mass Spectrometry, presented at the 1995 American Aerosol Annual Meeting, Pittsburgh, PA.
[26] Beuhler, R.J. and Friedman, L. (1980). Threshold Studies of Secondary Electron Emission Induced by Macro-Ion Impact on Solid Surfaces, Nuclear Instrum. Meth., 170, 309-315.
[27] Hinds, W.C. (1982). Aerosol Technology, John Wiley and Sons, New York, NY.
[28] Zimmermann, U., Malinowski, N., Naher, U., Frank, S. and Martin, T.P. (1994). Producing and Detecting Very Large Clusters, Z. Phys. D., 31, 85-93.

[29] Hillencamp, F., Karas, M. and Berkencamp, S. (1995). MALDI-MS in the Infrared: A Critical Evaluation, Proceedings of the 43d American Society of Mass Spectrometry Conference, Atlanta, GA, p. 357.
[30] Kassel, Jr., P.C. (1973). National Aeronautical and Space Administration report TN D-7359, Langley Research Center, Washington, D.C.
[31] Clark, L.G., Kinard, W.H., Carter, D.J., Jr. and Jones, J.L., Jr. (1984). The Long Duration Exposure Facility (LDEF), NASA Report SP-473, National Aeronautics and Space Administration, Washington, D.C.
[32] Dalmann, B.K., Fechtig, H., Grun, E. and Kassel, J. (1978). An Impact-mass-spectrometer for In Situ Chemical Analysis of Cometary Particulates to be Onboard a Flyby-mission, Science Space Instrumentation, 4, 73-83.
[33] Reinhard, R. (1987). The Giotto Mission to Comet Halley, J. Phys. E., 20, 700-712.
[34] Ohmi, T, Morita, M., Teramoto, A., Makihara, K., and Tseung, K.S. (1992). Very Thin Oxide on a Silicon Surface by Ultraclean Oxidation, Appl. Phys. Lett. , 06 (17).
[35] Elrharbi, S., Jourdain, M. and Meinertzhagen, A. (1994). Effect of Tunneling Electrons in Fowler-Nordheim Regime on the Current-voltage Characteristics and Model Degradation of Metal-oxide-Semiconductor Capacitors, J. Appl. Phys., 76 (2).
[36] Wortman, J.J., Griffis, D.P., Bryan, S.R., Kinard, W. and Kassel, P.C. Jr. (1985). Capacitance-type Micrometeroid Detectors, in Trajectory Determinations and Collection of Micrometeroids on the Space Station, F. Horz Ed., Lunar and Planetary Institute Technical Report number 86-05.
[37] Klein, N., (1966). The Mechanism of Self-healing Electrical Breakdown in MOS Structures, IEEE Transactions on Electron Devices, ED-13, No. 11.
[38] Elrharbi, S., Jourdain, M. and Meinertzhagen, A. (1994). Effect of Tunneling Electrons in Fowler-Nordheim Regime on the Current-voltage Characterics and Model of Degradation of Metal-oxide-Semiconductor Capacitors, J. Appl. Phys., 76, 1013-1020.
[39] Lee, K.H. and Campbell, S.A. (1993). The Kinetics of the Oxide Charge Trapping and Breakdown in Ultrathin Silicon Dioxide, J. Appl. Phys., 73, 4434-4438.

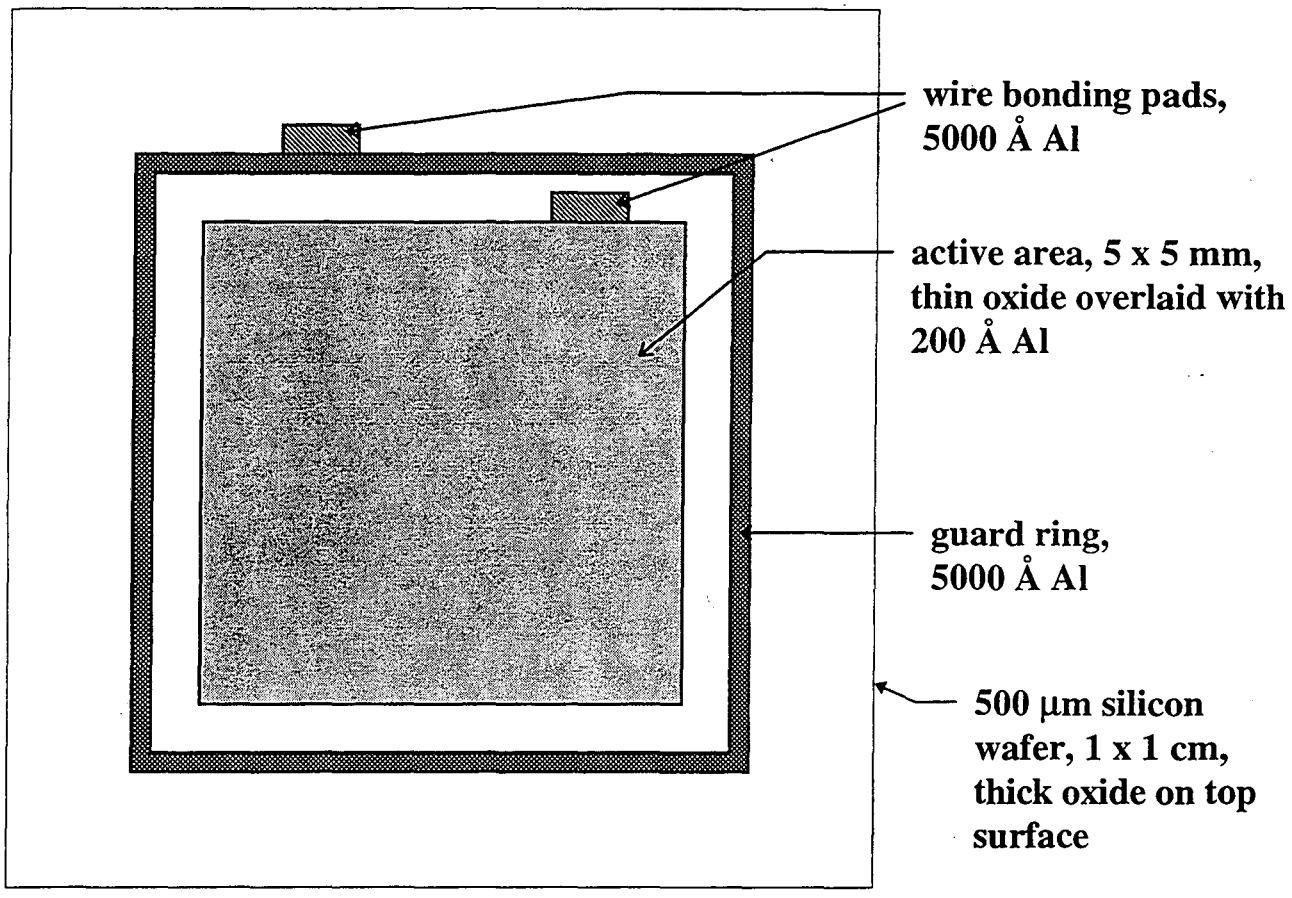


Fig. 1(a) - Schematic of a MOS device showing the central active area which is surrounded with a guard ring. Voltage is applied to each by wire bonding a 700 μm wire between the bonding pads and a more robust connector on the underlying IC socket.

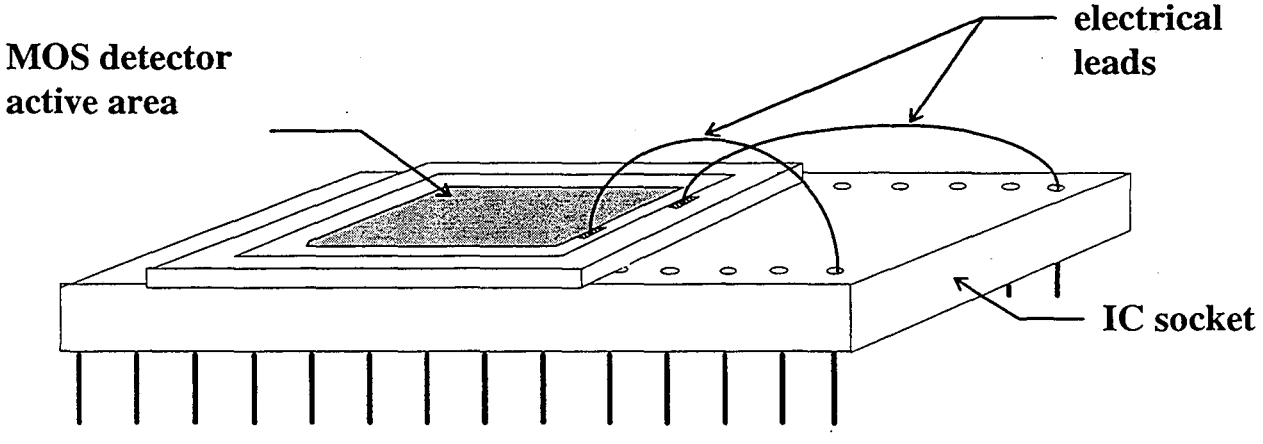


Figure 1(b) - Perspective view of the 5 x 5 mm silicon MOS detector after glued onto an IC socket with conducting paint.

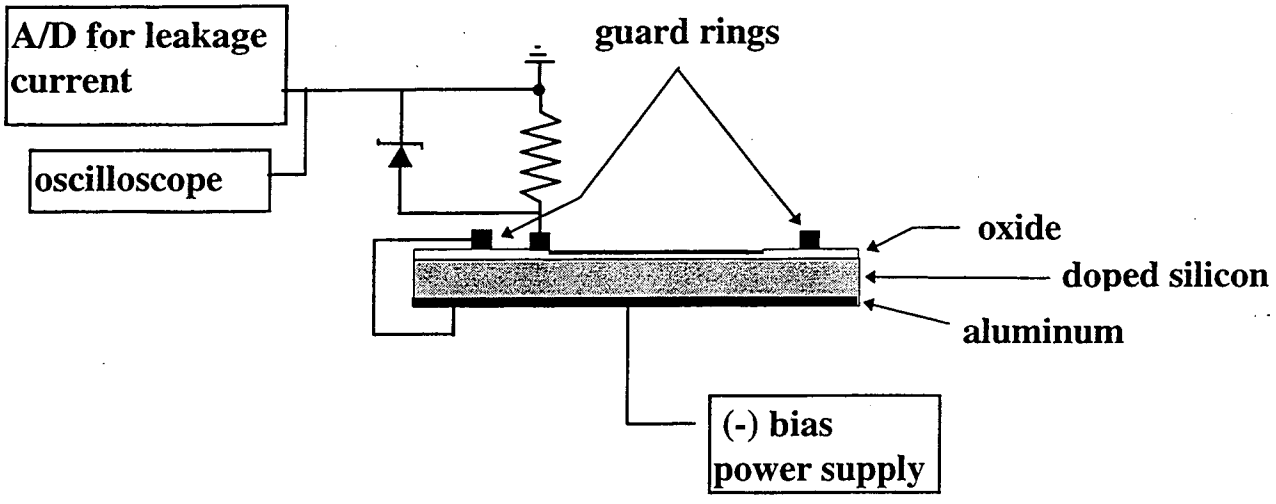


Fig. 2 - Electrical contact and biasing polarity for testing MOS devices. The power supply was ramped in voltage via computer control.

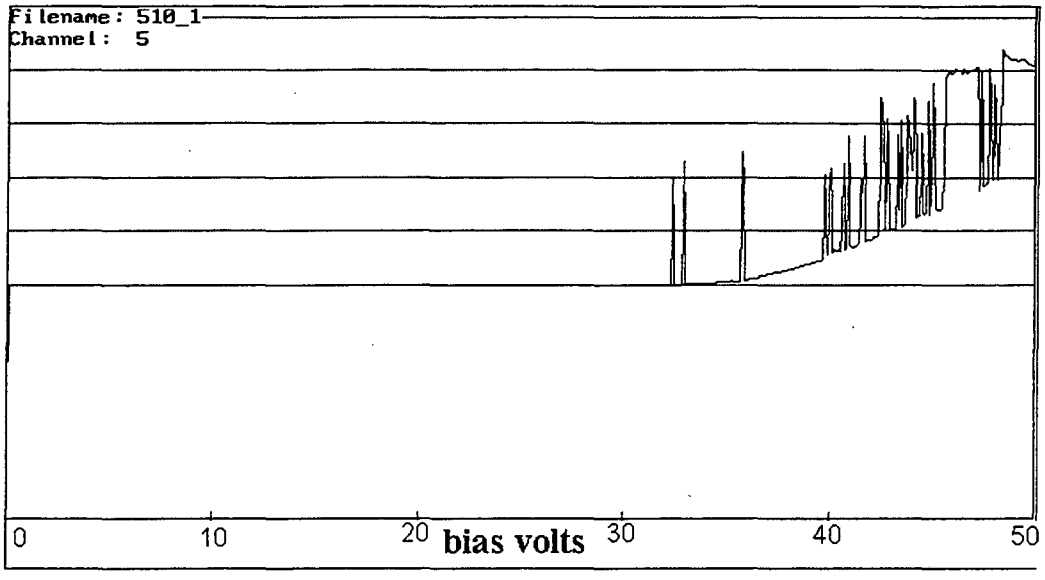


Fig. 3 - A plot of leakage current as a function of detector bias voltage for a 500 Å thick oxide device. The voltage was ramped at stepwise at 0.2 V/ 3 sec. Pulses appear above 30 volts and correspond to the "burning out" of defects in the oxide. The leakage current starts out at 0 and rises nearly to full scale (100 μA).

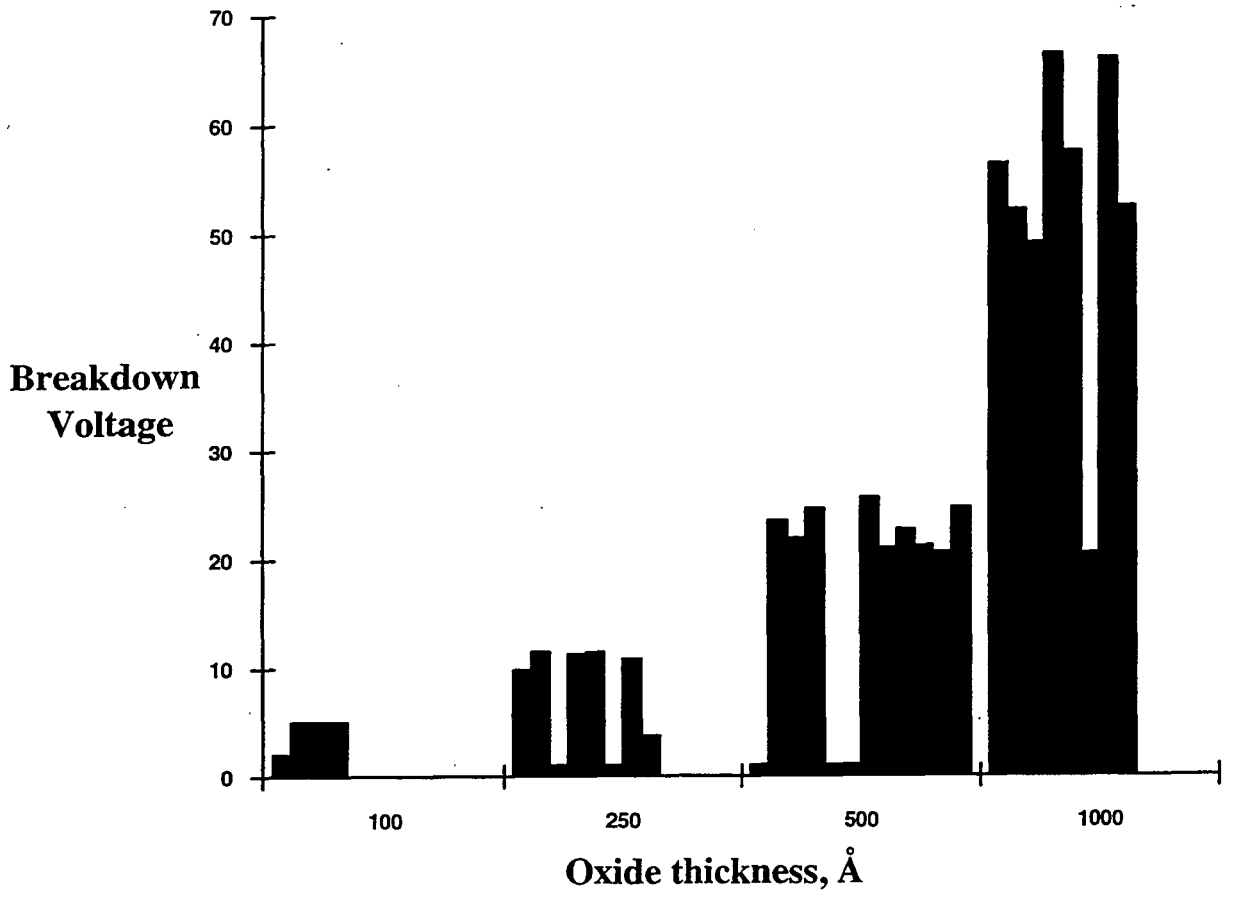


Fig. 4 - A distribution of breakdown voltage for MOS devices with four different thicknesses of oxide. Each bar represents one device.

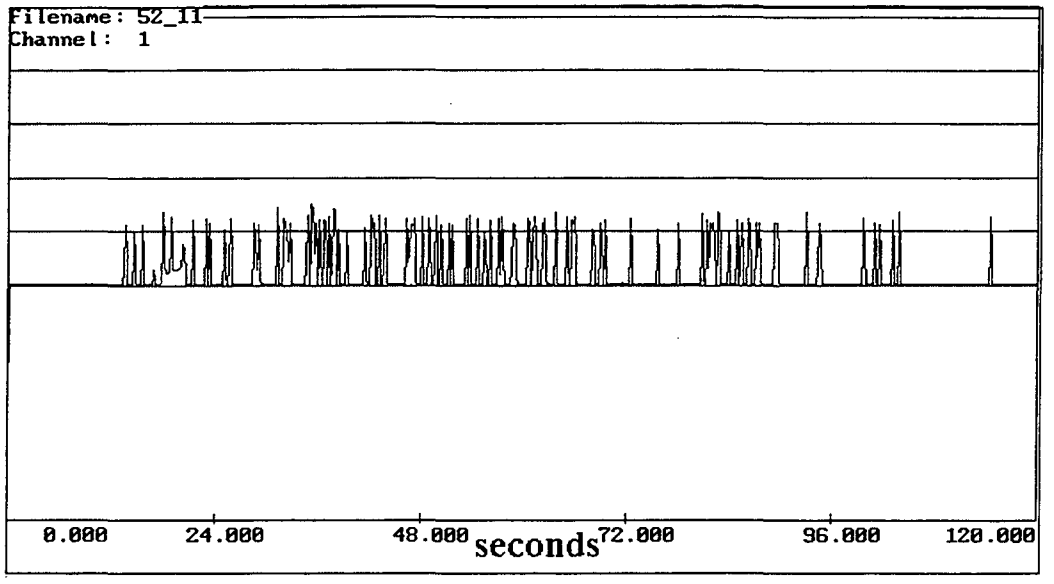


Fig. 5 - Pulses created by the impact of 2 μm diameter glass particles striking a 500 Å thick oxide detector. The impacts were created by accelerating particles through a nozzle in an aerosol impactor and impacts occurred near to sonic velocity. Pulse rise time is in the order of several msec. Each horizontal line on the vertical scale represents 20 μA.

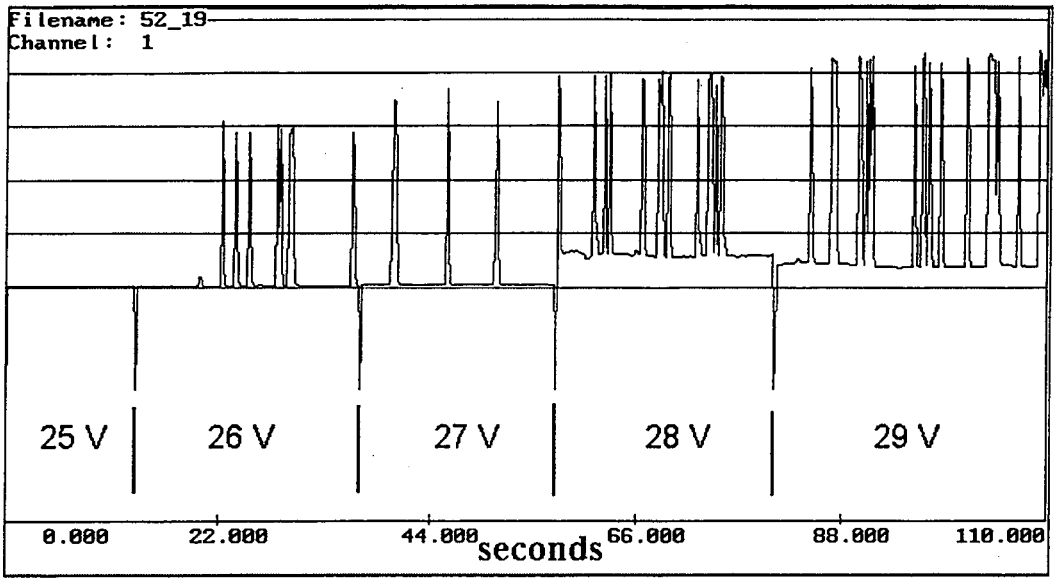
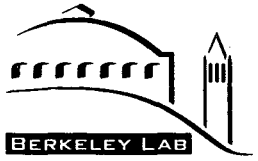


Fig. 6 - A record showing the pulse height for particle-impact induced breakdown in a 500 Å thick oxide detector as a function of bias voltage. Pulse height increases in correspondence with stored charge as expected for electrical capacitors. Each horizontal line on the vertical scale represents 20 μA.



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