# Anodic Oxidation-Induced Delamination of the SUMMiT<sup>TM</sup> Poly 0 to Silicon Nitride Interface

Richard Plass<sup>\*a</sup>, Jeremy A. Walraven<sup>b</sup>, Danelle M. Tanner<sup>a</sup>, and Frederick W. Sexton<sup>a</sup> <sup>a</sup>Radiation and Reliability Physics Department, <sup>b</sup>Failure Analysis Department Sandia National Laboratories, Albuquerque, NM 87185

## Abstract

Anodic oxidation can be a catastrophic failure mechanism for MEMS devices that operate in high humidity environments. Shea and coworkers<sup>1</sup> have shown that positively charged polysilicon traces can fail through a progressive silicon oxidation reaction whose rate depends critically on the surface conductivity over the silicon nitride. We have found a related anodic oxidation-based failure mechanism: progressive delamination of Poly 0 electrodes from silicon nitride layers, which then mechanically interfere with device function well before the electrode is fully oxidized. To explain this effect, we propose that the silicon oxide which initially forms at the electrode edge has insufficient strength to hold the local Poly 0 / silicon nitride interface together. This low-density silicon oxide also creates a bilayer system, which curls the edge of the 300 nm thick Poly 0 electrode away from the nitride. As delamination progresses more nitride surface is exposed and more of the interface is then attacked. This process continues cyclically until the electrode edge pushes against other device components, catastrophically and irreversibly interfering with normal operation. Additionally, we observe that the delamination only starts at electrode edges directly under cantilevers, suggesting the oxidation rate also depends on the perpendicular electric field strength.

Keywords: MEMS, anodic, oxidation, delamination, failure, mechanism

#### **1. Introduction**

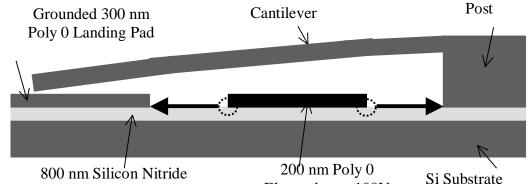
To date, most commercially available Micro Electro Mechanical System (MEMS) devices reside in hermeticallysealed packages where the environment around the device is controlled. However several potential MEMS applications, such as sensors, require at least some of the MEMS device to be exposed to ambient air. Also it is important to understand potential environment-induced failure mechanisms for MEMS in cheaper, nonhermeticallysealed packages and failed hermetically-sealed packages. Because of the relatively high voltages involved in electrostatic actuation (roughly 100 volts), anodic oxidation is an obvious potential failure mechanism of electrostatically-actuated devices operated at humidities above 50% RH.

In an earlier, comprehensive, study of anodic oxidation, Shea and coworkers<sup>1</sup> found that exposure of positively charged polysilicon electrical traces deposited on a silicon nitride insulator to varying degrees of humidity will eventually cause the traces to completely oxidize. In anodic oxidation the electric field enhances the ability of OH ions from the surface water layer to drift through the native oxide to where they can react with silicon at the Si/SiO<sub>2</sub> interface<sup>2</sup>. The oxidation rate is exponentially dependent on relative humidity, and the total amount of oxidized material is proportional to the total charge transferred through surface leakage currents over the nitride. Their data strongly suggest an anodic oxidation mechanism is at work<sup>3</sup>.

## 2. Experimental

We recently conducted a broad study of dormancy-induced stiction of cantilever beams by holding them actuated at +100 volts for extended periods of time under different humidity and temperature conditions. In this study we have found another way anodic oxidation can interfere with device function, namely progressive delamination of the Poly 0 electrode, followed by mechanical interference.

A side-view schematic diagram of the Sandia, Ultra-planar, MEMS, Multi-level Technology (SUMMiT<sup>TM</sup>) Interferometry for Material Properties in MEMS (IMaP<sup>TM</sup>) cantilever test structure is shown in Figure 1. Examples of damage to the Poly 0 pull-down actuation electrodes under both the Poly 12 laminated cantilevers and the Poly 3 cantilevers are shown in the subsequent figures. For the Poly 12 cantilevers, the initial gap between the electrode and the cantilevers is nominally 2 microns, while, for the Poly 3 cantilevers, it is about 6 microns. In Figure 1, the positively charged actuation electrode is shown in black and the surface leakage paths are shown with thick arrows. The OH ions responsible for anodic oxidation travel in the direction opposite the leakage current. In the case of this structure, they react at the point where the polysilicon electrode meets the bare nitride layer, which is also the location of highest field. Hence the primary location for low-density anodic oxide buildup is the edge of the Poly 0 electrode right at the nitride interface, shown by the dashed arcs in Figures 1, 2c) and 3b).



on 630 nm Thermal Oxide Electrode at +100V S1 Substrate Figure 1. Side view schematic of a short Poly 12 cantilever actuated into an arc shape very early in a humidity test. The critical surface leakage currents are shown by black arrows, and the edges of greatest oxide buildup are at the centers of the dashed arcs.

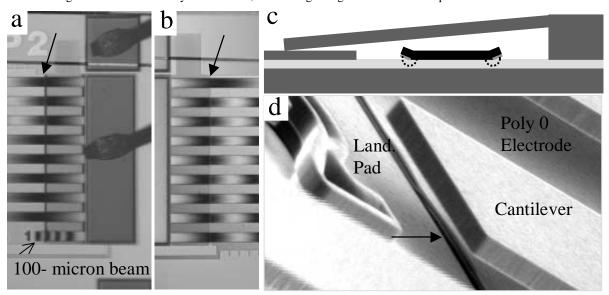


Figure 2. Initial stages of anodic oxidation-induced delamination of the Poly 0 electrode. a) Interferogram of a supercritical  $CO_2$ dried die exposed to 50% RH at 45°C for 20 days while actuated at +100 volts. Anodic oxidation-induced damage is in its early stage. Just barely visible is a contrast increase of the actuating electrode-to-landing pad gap as compared to b) the unactuated electrode. Also the 100-micron long beam is deflected upward very slightly, as seen by the interference fringes on this beam. c) Side view schematic showing the early stage of the delamination and formation of an oxide layer on the underside of the delaminated region (thin gray line under the black electrode). Note that the point of attack of the interface has moved in from the edge of the electrode. d) Scanning Electron Microscopy (SEM) micrograph of the Poly 3, 200-micron long cantilever region of a supercritical  $CO_2$  dried plus oxygen plasma cleaned die held at 50% RH and 25°C for 50 days and +100 volts actuation. The arrow shows where the electrode is starting to delaminate.

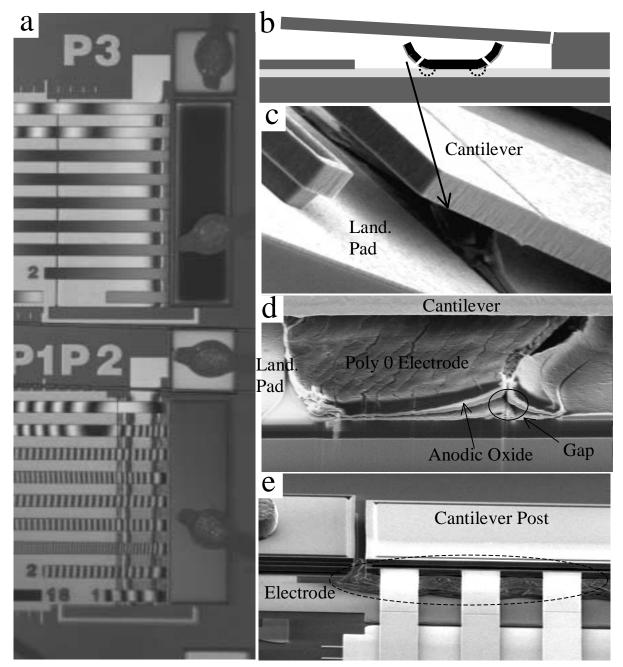


Figure 3. Medium stage anodic oxidation-induced delamination. a) Optical interferogram of a supercritical  $CO_2$ -dried die exposed to 50% RH at 45°C for nine days while actuated at +100 volts. A U-shaped damage pattern is seen around the edges of the Poly 12 actuation electrode. White lines across the poly layers in the side-view schematic of b) show stress points where the polysilicon is likely to crack with increased delamination. c) SEM micrograph of the Poly 12 100-micron long cantilever region of a supercritical  $CO_2$ -dried plus oxygen plasma cleaned die held at 50% RH and 25°C for 50 days showing the mechanical contact of the delaminated poly 0 electrode with the cantilever. d) SEM micrograph of a damaged die taken perpendicular to the long axis of the 100-micron long Poly 12 cantilever showing how the poly 0 electrode has cracked near the delamination point. A focused ion beam cross-section in the foreground of d) shows the delamination gap as well as the (arrowed) light gray anodic oxide under the dark gray poly 0; the medium gray material adjacent to the gap is ion milling debris. e) SEM micrograph of a supercritical  $CO_2$ -dried die held at 50% RH and 25°C for 48 days, where the anodic oxidation delamination was severe on the post side of the electrode (dashed ellipse).

#### 3. Results and Discussion

To explain the progressive delamination shown in Figures 2 to 4, we propose that the silicon oxide that forms at the electrode edge has insufficient strength to hold the local poly 0 / silicon nitride interface together. This low-density silicon oxide also creates a bilayer system, which curls the edge of the 300 nm thick poly 0 electrode away from the nitride. As delamination of the electrode progresses, more nitride surface is exposed and more of the interface is then attacked, continuing the delamination. This process continues cyclically until the electrode edge touches the cantilever and forces it upward, catastrophically and irreversibly interfering with its normal operation. However, before the electrode delamination causes mechanical interference, it affects device operation both in changing the original gap spacing and fringing fields (thus altering the electrode voltage to cantilever deflection curve) and in blocking some of the original cantilever range of motion.

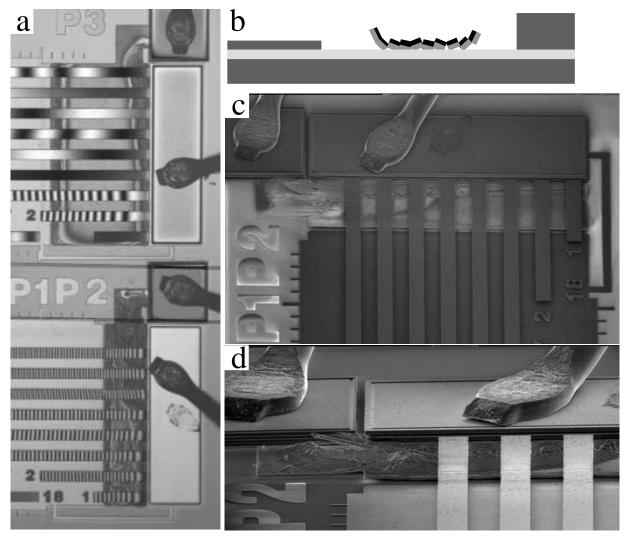


Figure 4. Advanced stage anodic oxidation induced delamination. a) Optical interferogram of a supercritical CO<sub>2</sub>-dried plus oxygen plasma-cleaned die held at 50% RH and 25°C for 101 days showing U-shaped characteristic damage to the edges of the Poly 3 cantilever electrode and essentially complete delamination of the Poly 12 cantilever electrode. The Poly 0 electrode is now mechanically interfering with the 200 and 300-micron long Poly 3 cantilevers, and the Poly 12 900-micron long cantilever has been broken off at its root. A schematic side-view diagram of this situation is shown in b) with details shown in the SEM micrographs of the same die in c) and d) taken at different sample angles.

For prolonged experiments, we find the delamination sometimes produces sufficient force to break a 2.5-micron thick cantilever where it is anchored, as seen in Figure 4. However, the thinner Poly 0 electrode normally breaks near the delamination point. The relative humidity (RH) and temperature conditions under which we see the delamination occur qualitatively agree with those reported by Shea *et al.*<sup>1</sup> and predicted by Comizzoli<sup>3</sup>, namely we see insignificant anodic oxidation at 25% RH and, as discussed below, the oxidation occurs much faster at higher temperatures. Further experiments are in progress.

The failure mechanism only initiates at regions under cantilevers, an example of which is seen in Figure 2 d). This fact suggests that the electric field perpendicular to the current flow plays a role in enhancing the current flow. This dependence could be directly field-related or related to the interaction of the field with the surface water layer. Comizzoli and coworkers<sup>4</sup> have suggested that a locally strong perpendicular field could induce a local increase in nitride surface conductivity by electrostriction. That is, they propose that electrostriction locally increases the number of water layers through which the surface current passes, thus increasing the local surface conductivity and the oxidation rate relative to regions where the perpendicular field is not as strong.

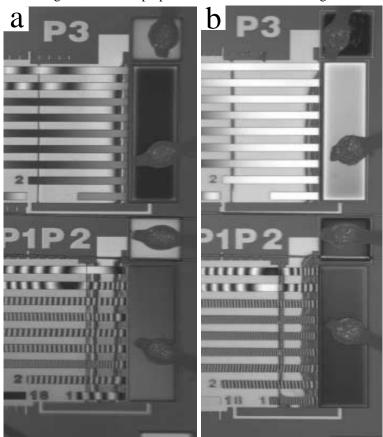


Figure 5. a) Same as Figure 3 a) Optical interferogram of a supercritical CO<sub>2</sub>-dried die held at 50% RH and 45°C for nine days at +100 volts. b) Optical interferogram of a supercritical CO<sub>2</sub>-dried die held at 50% RH and 25°C for 48 days showing comparable delamination levels.

The delamination failure mechanism consistently attacks the electrode region under the poly 12 100-micron long beam first. An example of this damage is shown in Figure 2 a), where there are interference fringes only on the Poly 12 100-micron cantilever. The interference fringe patterns of the cantilevers in Figure 3 a), combined with a defocus measurement, indicate that most of the cantilevers have been deflected upward substantially, a situation illustrated in Fig. 3 b). Also worth noting in Figure 3 a) is that the general lack of fringes on the Poly 3 cantilevers indicate the

accompanying electrode has not yet delaminated far enough to deflect most of them. The nonuniform contrast of either edge of the Poly 12 actuation electrode in Figure 3 a) illustrates the characteristic damage that we would expect, given the surface leakage paths.

We find that, generally, parts cleaned with an oxygen plasma after supercritical CO<sub>2</sub> drying are more susceptible to this anodic oxidation effect than parts wich were just supercritically dried. Among die that received the two final surface treatments, the extent of anodic oxidation varies significantly for the same test conditions, a variation we attribute to different levels of local surface contamination on the nitride surfaces from die to die<sup>5</sup>.

Figure 4 shows examples of advanced stages of anodic oxidation-induced delamination, where a 900-micron long Poly 12 cantilever has fractured at its root. Though this particular electrode is badly shattered and in contact with the rest of the cantilevers, it can still hold +100 volts without shorting, since the electrode debris is heavily oxidized. Considering the effects of temperature on this failure mechanism, anodic oxidation-induced delamination levels after nine days test duration at 45°C are comparable to parts held for 50 days at 25°C under the same relative humidity conditions, as is shown in Figure 5. The damage at 45°C appears to be more uniform and is perhaps less affected by local electric field variations.

Ways to mitigate this failure mechanism center on minimization of electric fields through both device design (e.g., increasing electrode to grounded feature gap distances) and minimization of operating voltages, as have been mentioned previously<sup>1</sup>. Care must also be taken in choosing operating voltage polarity.

## 4. Summary

We have found that for test conditions near 50% RH, anodic oxidation generally occurs with a rate that quickly increases with temperature, depends on final surface treatment, and likely varies with local surface contamination. Since our test structures are more complicated that the line traces studied by Shea et al., we have also found that the anodic oxidation rate seems to depend on the field strength perpendicular to the surface current. Most importantly, anodic oxidation causes Poly 0 electrodes to steadily delaminate from the silicon nitride surface. Depending on device design, this delamination may progressively alter the electrical properties of the device and limit its range of motion. At some point, the delaminated Poly 0 electrode will mechanically interfere with device operation, usually well before the electrode is fully oxidized.

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#### References

<sup>&</sup>lt;sup>1</sup> H. R. Shea, A. Gasparyan, C. D. White, R. B. Comizzoli, D. Abush-Magder, and S. Arney, "Anodic Oxidation and Reliability of MEMS Poly-Silicon Electrodes at High Relative Humidity and High Voltages", Proc. of SPIE, 4180, pp. 117-122, 2000.

N. Cabrera and N.F. Mott, "Theory of Oxidation of Metals", Rep. of Prog. in Phys. 12, pp. 163-184, 1948.

<sup>&</sup>lt;sup>3</sup> R.B. Comizzoli, "Surface Conductance on Insulators in the Presence of Water Vapors", *Proc. of the Fourth* 

Electronic Materials and Processing Congress, Quebec, Canada, August 19-22, pp. 311-316, 1991.

<sup>&</sup>lt;sup>4</sup> R. B. Comizzoli, J. W. Osenbach, G.R. Crane, G. A. Peins, D.J. Sinconolfi, and C.-C. Chang, "Failure Mechanism of Avalanche Photodiodes in the Presence of Water Vapor", J. of Lightwave Tech., 19, pp. 252-265, 2001.

<sup>&</sup>lt;sup>5</sup> P. J. Resnick and P. J. Clews, "Whole Wafer Critical Point Drying of MEMS Devices", *Proc. of SPIE*, **4558**, pp. 189-197. 2001.

<sup>&</sup>lt;sup>\*</sup> raplass@sandia.gov; phone 1-505-844-6695; fax 1-505-844-2991; Sandia National Laboratories, M.S. 1081, P.O. Box 5800, Albuquerque, NM 87185, USA