Piezoelectric Polyimide MEMS Process

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Abstract

We have demonstrated a fabrication process for fabricating MEMS structures using a novel high-temperature piezoelectric polyimide. The process consists of conventional lithography and metalization processes and uses a sacrificial layer of photoresist. Electrodes are fabricated on the upper and lower surfaces of the suspended structures and a self-test electrode is fabricated underlying each suspended component for testing. Particular attention is paid to the baking and curing of the polymer films used for structural, sacrificial and lithography layers in order to maintain the structural integrity of the devices throughout the fabrication process. A key aspect to accomplishing this is the use of a chemically imidized polyimide solution to eliminate the high curing temperature normally required to form polyimide. The entire process sequence, through the removal of the sacrificial layer, is kept below 125 °C. This process has application to building a variety of microelectromechanical sensors and actuators.

1. Introduction

Piezoelectric materials are important materials in a variety of micro-electro-mechanical systems (MEMS). Typical materials used are quartz, zinc oxide, and lead zirconium titanate (PZT) [1, 2]. However, these materials are crystalline ceramics that are brittle. Alternatively the polymer polyvinyldeneflouride (PVDF) has attained widespread use as a piezoelectric material in low cost flexible structures. Unfortunately, the temperature range over which the piezoelectric properties of PVDF are maintained is limited to less than 80 °C. Recent work has shown higher temperature piezoelectric response in newly developed polyimides [3]. In this work, we present a surface-micromachined process for fabricating the structural components for high temperature, polyimide-based micro-electro-mechanical systems (PolyMEMS) as shown in Figure 1. This simple prototype bridge structure was selected for ease of fabrication in order to demonstrate the overall fabrication process. Upper and lower electrodes are used to sense the potential across the polyimide structural bridge and a self-test electrode underneath the bridge can be used to apply an electrostatic force, for calibration and testing. Other structures, such as cantilevered structural components, would be fabricated by lithographically eliminating the second substrate contact.

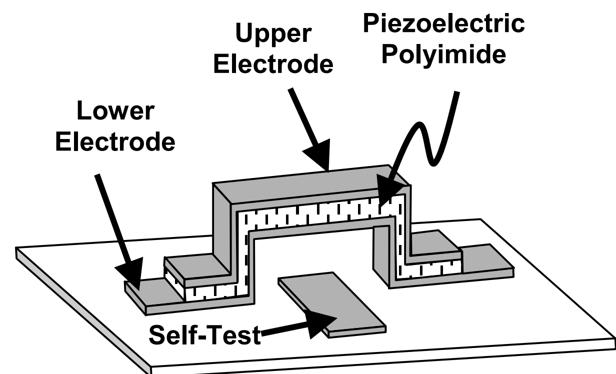

Figure 1. Piezoelectric polymer sensor structure

The key advantage of these new PolyMEMS devices is the high temperature capability of the polyimid structural elements. The high temperature performance of these modified polyimide films allows these films to maintain their piezoelectric properties at temperatures up to 150 °C, twice that of PVDF [3,4]. A comparison of the properties of these piezo-polyimides verses PVDF is given in Table 1 and Fig. 2. The high piezoelectric voltage coefficient (g_{mn}) and strain coefficient (d_{mn}) at higher temperatures coupled with the fact that polyimides resist harsh environments, makes these materials ideal for high temperature PolyMEMS devices. However, it is important to note that even with the relatively high glass transition temperature of these polyimides ($T_g \sim 220$ °C), fabrication processes must be kept at relatively low temperatures by traditional silicon microfabrication processing standards.

Table 1. Piezoelectric voltage coefficients of PVDF vs. newly developed polyimide.

Polymer	*T =25°C*	*T =75°C*	*T =150°C*
	g_{31} (mV-m/N)	**g_{31} (mV-m/N)**	**g_{31} (mV-m/N)**
PVDF	**235**	**376**	**-**
Polyimide	**7.6**	**22**	**152.7**

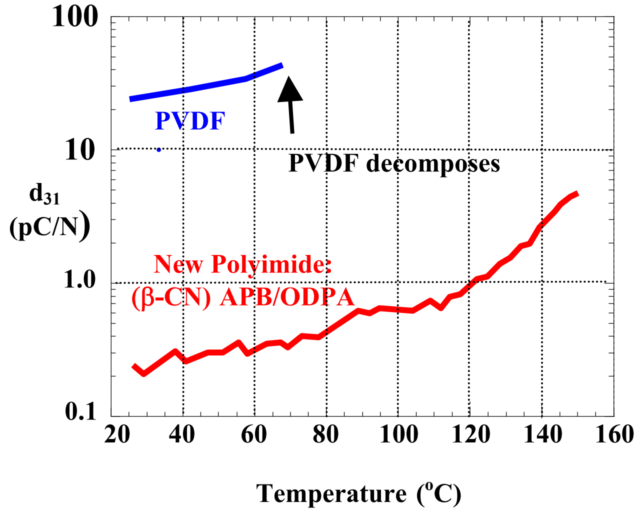

Figure 2. The piezoelectric strain coefficient (actuator response) of polyimides vs. PVDF.

2. Experimental

In order to demonstrate the MEMS utility of the piezoelectric polyimides, we have developed a low-temperature (< 120 °C) surface-micromachined MEMS process that incorporates these films. In this process, the electroactive polyimides are used as the structural elements (such as a cantilever or bridge) and upper and lower electrodes are incorporated onto the upper and lower surfaces for sensing the piezoelectric response of the stressed element. The overall process is illustrated in Fig. 3, while the CAD layout for a typical bridge structure is illustrated in Fig. 4. The upper and lower electrodes in the structure are also used for implementing the poling process, which reorients the polymer dipoles to give the final polyimide structural beam its piezoelectric characteristics.

2.1 Material Development

These new polyimide polymers have unique and attractive advantages particularly suitable for electronic and aerospace applications. With their exceptional thermal, mechanical, and dielectric properties, polyimides are already widely utilized as matrix materials in aircraft and as dielectrics in the microelectronics industry. Consequently, amorphous polyimides containing polar functional groups have been synthesized and investigated at VCU and NASA LaRC for potential use as high temperature piezoelectric sensors [3,4]. To date, we have successfully synthesized, processed and tested a new class of piezoelectric polyimides. Clearly from Table 1 and Fig. 2, the polyimide has a lower g_{31} and d_{31} at room temperature than PVDF. However, as the temperature is increase about 75 °C, g_{31} and d_{31} for these polyimides increase while those of PVDF can no longer be detected. Further material development studies are underway to further optimize the properties of these high temperature polyimide films. The ultimate intent of this ongoing research is to elucidate the mechanisms for piezoelectricity in amorphous polyimides and apply this to designing and optimizing high temperature, flexible, piezoelectric micro devices.

2.2 Fabrication

The prototype PolyMEMS device structures that have been fabricated consist of simple cantilevered bridge structures. As shown in Fig. 1, the suspended structural element consists of electroactive polyimide, with upper and lower electrodes attached, and a self-test electrode fabricated below the suspended structure for electrostatic on-wafer testing.

The overall fabrication sequence for PolyMEMS structures is illustrated in Fig. 3. First, an oxidized silicon wafer is patterned with aluminum, using conventional lithography and wet etching. This forms the self-test electrode that is isolated from the substrate and can be used conveniently to electrostatically test the final structure. Next, a photoresist sacrificial layer is spun on and patterned (Step 2). This can again be accomplished using standard lithography procedures and photoresist fabrication techniques. A spin speed of 2000 rpm with a commercially available (SPR-3012) photoresist, yielded a 1.3 μm thick sacrificial layer, determined using optical interferometric measurement of the resist thickness. Unlike conventional processing however, this photoresist layer remains intact on the device structure as the sacrificial layer that will only be removed at the last stage of the overall process. It is therefore critical to thoroughly bake the sacrificial layer at this point to prevent it from outgassing during later processing steps. Since it will be sealed with an aluminum layer, outgassing will form bubbles, distorting the PolyMEMS structural components. Currently, a 24 hour, 120 °C bake is used to completely dry out the sacrificial photoresist layer.

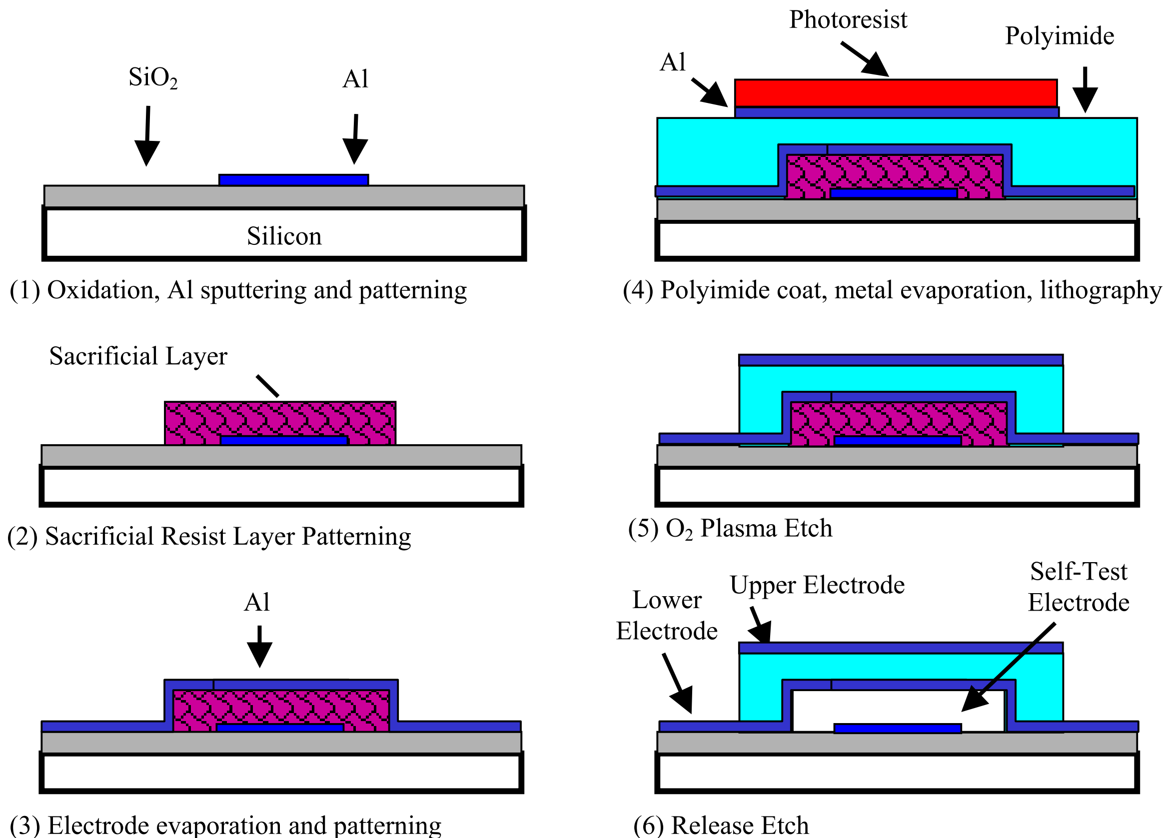

Figure 3. Prototype PolyMEMS fabrication process with electrostatic self-test capability.

In Step (3), the lower bridge electrode is formed by sputtering a thin coat of aluminum over the entire substrate, and patterning it by lithography and etching. The deposition process is performed immediately after the 24-hour sacrificial resist bake, to prevent the underlying resist from absorbing moisture from the atmosphere before being sealed in with the metal layer. The sputtering process is accomplished in a D.C. magnetron sputtering system (MRC 603), which provides an 100 nm thick film in a single pass at 5 kV and 8 microns pressure. Using a single pass provides a thin layer of aluminum as well as minimizes substrate heating. We have not observed any bubbling or outgassing of the sacrificial layer during the aluminum sputtering process.

The subsequent lithography process for the lower bridge electrode is slightly modified by lowering the post-exposure bake temperature (90 °C for 2 minutes) to prevent any possible outgassing of the underlying sacrificial layer. The aluminum layer is then etched in a standard wet chemical etch using phosphoric acid, acetic acid and nitric acid in a (16:1:1) mixture at 40 °C. Our typical etch rates are 12 nm per minute, so that within one minute our aluminum film is completely etched through. We do not see any deleterious effects on the etch resistance or adhesion of the photoresist mask in the aluminum etching process from baking at the lower temperature.

Once the aluminum layer is patterned, the photoresist above the aluminum is removed. Since the underlying sacrificial resist is exposed at its edges, solvent solutions cannot be used for photoresist removal at this point. We have found that an isotropic plasma etching/ashing process in oxygen is a suitable for photoresist removal at this point. However, care is taken to turn off the substrate heating capability commonly used in photoresist ashing, to prevent undue substrate heating. Typical removal rates in our barrel asher for unheated photoresist are 21 nm/min at 120 Watts at approximately 30 mtorr pressure.

Next in Step (4), the piezoelectric polyimide film is spin-cast onto the wafer. The polyimide pre-cursor solution tends to present adhesion problems with surfaces that contain moisture, so a short, 15 second, 90 °C prebake is used to remove moisture from the wafer surface. Polyimide solutions of 15% and 20% by weight have been investigated for this spin casting process. At a nominal spin speed of 4000 rpm, this results in a polyimide film thickness of 1.3 µm and 4.0 µm respectively. The 20% by weight solution is dramatically more viscous and difficult to apply effectively to the

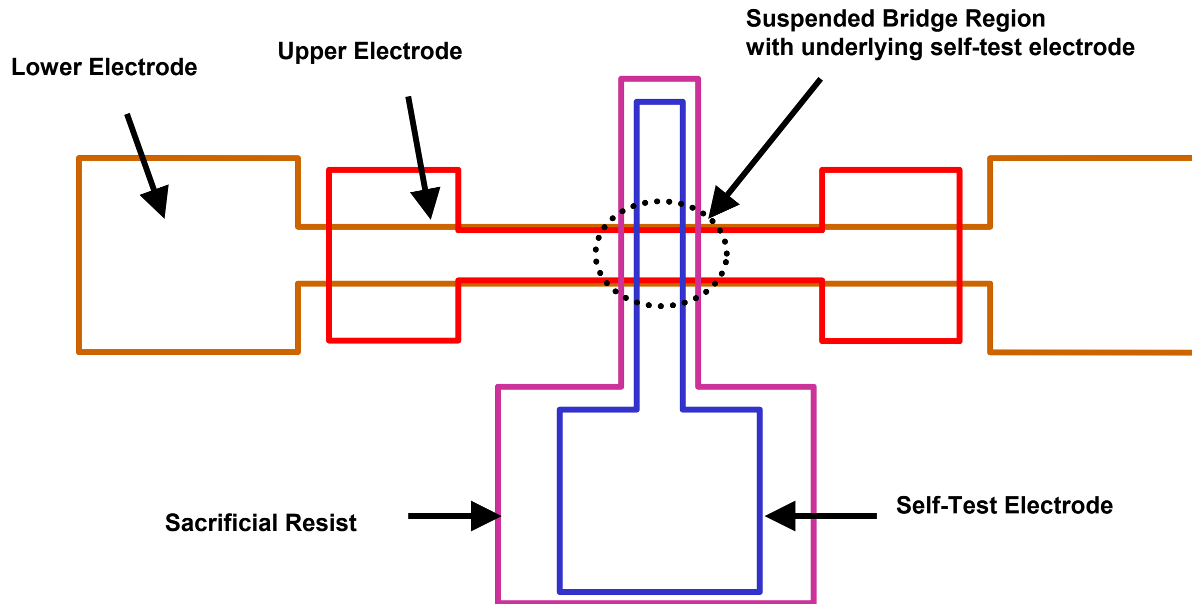

Figure 4. 4-level CAD layout of polymer MEMS bridge structure

substrate. Also film uniformity is poor for the thicker polyimide film, varying 10 – 15% over the wafer surface. The 1.3 µm films, applied with the 15% by weight polyimide solution, had film uniformity of 5% or better as measured by optical interference. Solvent absorption of the sacrificial photoresist underneath the polyimide did not appear to be detrimental to the device structure.

In order to keep the polyimide film preparation temperatures low (to keep the underlying sacrificial photoresist intact), we used a chemically pre-imidized polyimide solution for this process. In this case, the only requirement to form the completed polyimide polymer film is to ensure that the solvents are completely driven out. We are currently using a 24-hour bake at 90 °C to cure the polyimide film. Differential scanning calorimetry (DSC) measurements are used to verify that the films are completely cured.

Immediately following the curing of the polyimide, the wafer is metalized with 100 nm of aluminum using the sputtering process as before. No deleterious effects have been observed to the underlying polymer layers during this final metalization. The upper electrode is patterned using standard lithography and etching as in the previous steps. Because the sacrificial photoresist is still intact on the wafer, the post-exposure bake is again kept to 90 ºC as before. The standard aluminum wet etch completes before, completing Step (4).

In Step (5), the upper electrode provides an etching mask for the polyimide in an O_2 plasma. This not only defines the bridge element itself, self-aligned to the upper electrode mask, but also removes the photoresist from the previous lithography step. The etching rate of the polyimide is approximately 30 nm/min, similar to the photoresist, so that by the time the thicker polyimide layer is removed, the upper (0.7 µm thick) photoresist layer has been completely removed as well. The isotropic nature of our current oxygen plasma etching process does result in significant undercutting of the polyimide structural layer, approximately equal to the thickness of the film (1.3 µm). This limits the minimum width of these PolyMEMS structures in our current process to greater than 5 µm. A directional reactive ion etching process would alleviate this limitation and reduce the minimum sizes achievable.

The last fabrication step of the PolyMEMS process is the release process for the suspended structural components. The sacrificial layer in this case is a photoresist that has been baked at a maximum temperature of 120 °C in the processing up until this point. At these low temperatures, photoresist can be removed using acetone. It is critical that the acetone does not dissolve the polyimide layer and test samples of polyimide submerged in acetone showed no loss of thickness. A second important aspect of the release process is to ensure that the suspended components are not brought into contact with the substrate by capillary action during drying, becoming permanently adhered by Van der Waals forces. This is prevented in the release process by a sequential solvent sequence using successively higher vapor pressure solvents. The acetone solution is replaced with methanol, which is then replaced with pentane and finally, hexane. The wafer can then be dried in air at room temperature (Step (6)). The

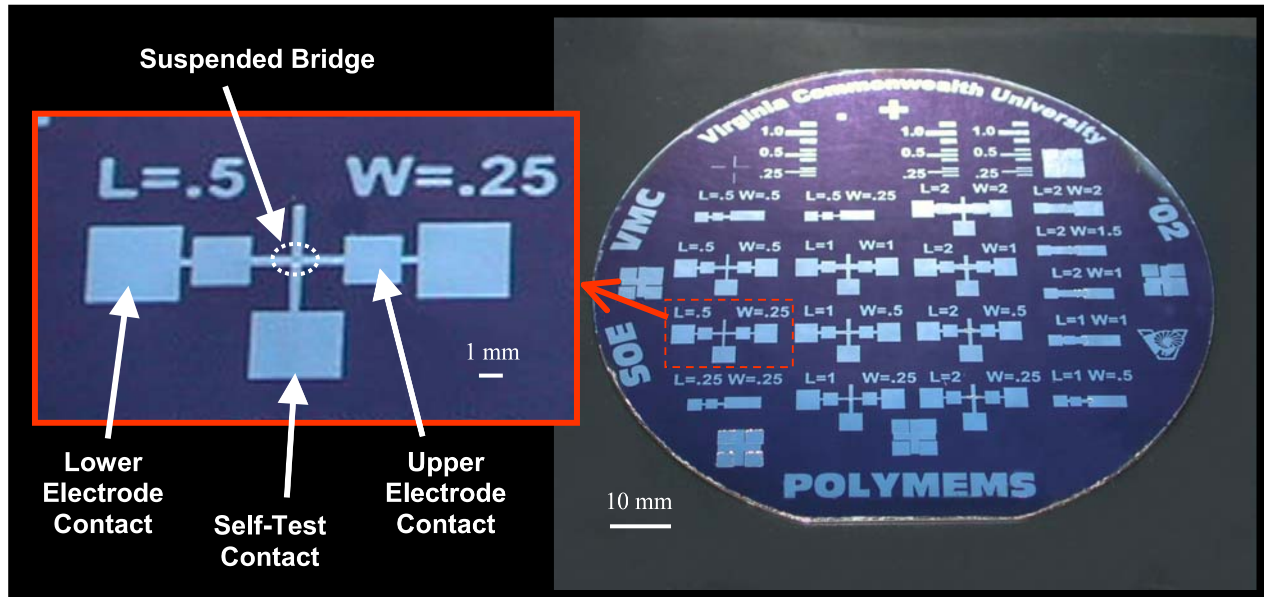

Figure 5. Optical photograph of a PolyMEMS wafer prior to release.

increasing vapor pressure of the solvent sequence reduces the capillary forces on the structural components during the air-drying step and prevents adhesion and stiction of the structural components to the surface [5].

2.3 Poling

In order to give the polyimid film its piezoelectric properties, the structural polymer bridge must undergo a poling process to orient the dipoles that have been incorporated into these novel polyimides. This post-fabrication poling process is accomplished by applying a strong electric field (50-75 MV/m) between the upper and lower bridge electrodes, at an elevated temperature ($T>T_g$). The temperature is then reduced, with the field still in place, freezing the dipole orientations in place. In order to accomplish this step, we are currently using a 100V (applied between the upper and lower electrodes) for our 1.3 µm and 2.15 µm thick polyimide bridge structures (77 MV/m and 47 MV/m respectively) at temperatures of 170 °C and 200 °C.

3. Results and Discussion

An optical photograph of a PolyMEMS test wafer just prior to the release step is shown in Fig. 5. Cantilever and bridge structures vary from 250 µm - 2000 µm in length and width. The entire process was completed and demonstrated that with proper temperature control, the structural polyimide and photoresist layers can be applied and patterned without compromising a standard sacrificial photoresist layer underneath. Additionally, adhesion of the aluminum to the polyimide (verified with a tape test) was excellent. However, adhesion strength of the polyimide to the substrate oxide was low, resulting in lifting of upper electrode patterns not anchored by lower electrode metalization. This will be alleviated in future process runs by adding an electrically isolated island of the self-test aluminum layer as an anchor under regions where the polyimide normally would contact the oxide surface.

These results also demonstrated the solvent release method for polyimide bridge structures. Structures were released up to 2000 µm in length with isolated electrode layers and a visible gap below the structures as verified by a scanning electron micrograph (Fig. 6). Lifting the bridge structures from the surface and inspecting revealed that there was no visible resist material underneath. Some difficulty was encountered with the cantilevered structures, which need to be redesigned with stiction bumps and less aggressive aspect ratios.

The preliminary results of the polyimid poling process indicate that additional curing may be taking place in this final step, as indicated by DSC measurements which revealed a lower than expected glass transition temperature (T_g ~ 165 °C). Because the sacrificial resist has already been removed at this point, the remaining materials are the aluminum electrodes and the polyimde structural film. Therefore, it is possible to cure the film at a higher temperature to drive out the small amount of remaining solvent at the final stage. Further work is underway to optimize the final poling and curing process in these devices.

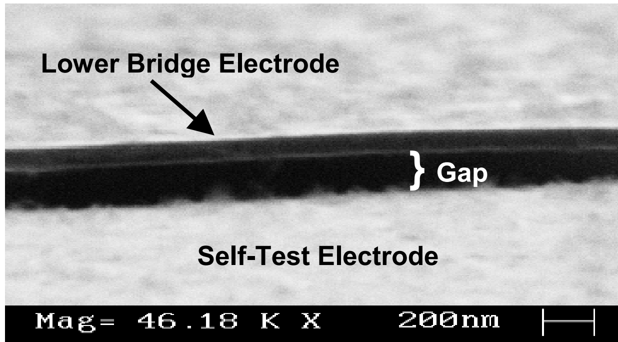

Figure 6. Suspended electrode with visible gap.

4. CONCLUSION

We have fabricated suspended structural components in high temperature piezoelectric polyimides for polymer based MEMS or PolyMEMS. These structures consist of cantilever and bridge structures with upper and lower electrodes and an integral self-test electrode. These devices have the potential to offer higher temperature sensor performance than conventional polymer micromechanical devices fabricated from PVDF. Additional work is required to optimize the curing and poling processes to enhance the piezoelectric performance of these devices. These studies will use DSC, thermally stimulated current (TSC) measurements and dynamic dielectric spectroscopy (DDS) to measure the dipole reorientation and piezoelectricity. Finally the performance will be verified using electrostatic probing with the on-wafer self-test electrode.

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