Review of Polyimides Used in the Manufacturing of Micro Systems

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Acronym List

(β-CN)/APB/ODPA	(bis(aminophenoxy)benzonitrile)/AminoPhenoxy Benzene /OxiDiPhthalic Anhydride
BPDA	BenzoPhenonetetracarboxylic DiAnhydride
CMUT	Capacitive Micromachined Ultrasonic Transducer
HF	HydroFluoric
LIGA	LIthographie, Galvanoformung, Abformung (German acronym for Lithography, Electroplating and Molding)
MEMS	MicroElectroMechanical Systems
NMP	N-Methyl-2-Pyrrolidone
PDMS	Poly-Di-Methyl-Siloxane
PI	PolyImide
PMDA	PyroMellitic DiAnhydride
PMDA-ODA	PyroMellitic DiAnhydride-OxyDiAniline
PMMA	Poly-Methyl-Meth-Acrylate
PVDF	PolyVinyliDene Dluoride
RF	Radio Frequency
SAW	Surface Acoustic Wave
TFMOB	TriFluoroMethOxyBenzidine
UV	Ultra-Violet
VCU	Virginia Commonwealth University

Abstract

Since their invention, polyimides have found numerous uses in MicroElectroMechanical Systems (MEMS) technology. Polyimides can act as photoresist, sacrificial layers, structural layers, and even as a replacement for silicon as the substrate during MEMS fabrication. They enable fabrication of both low and high aspect ratio devices. Polyimides have been used to fabricate expendable molds and reusable flexible molds. Development of a variety of devices that employ polyimides for sensor applications has occured. Micro-robotic actuator applications include hinges, thermal actuators and residual stress actuators. Currently, polyimides are being used to create new sensors and devices for aerospace applications. This paper presents a review of some of the many uses of polyimides in the development of MEMS devices, including a new polyimide based MEMS fabrication process.

I. Introduction

Polyimides have been around since the early 1960's when they were invented by DuPont. Polyimides are thermosetting ring chain polymers that are constructed from imide monomers. See Figure 1 for the chemical structure of a typical PyroMellitic DiAnhydride-OxyDiAniline (PMDA-ODA) polyimide.

Polyimides are an extremely versatile material. They have a large degree of variability in their chemical make-up, and accept a wide variety of chemical additives. This allows for tailoring of the material properties to match the requirements of many different applications. They can be modified to be conductive or insulating, magnetic or non-magnetic, piezoelectric or non-piezoelectric, photosensitive or non-photosensitive as the user sees fit. With the addition of chemically specific additives, polyimides can be made to absorb specific chemical species, while remaining impervious to most other chemicals.
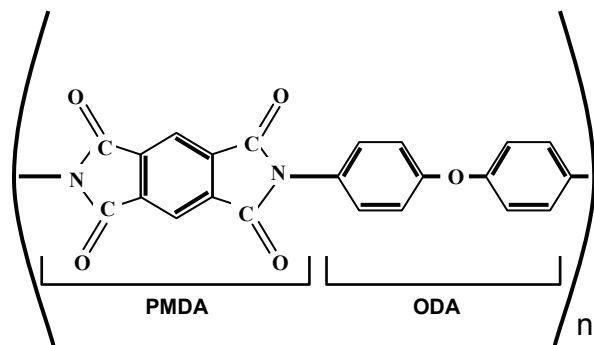
Polyimides exhibit excellent chemical resistance. This is due to the concentration of polymer molecules which pack closely leaving very little room for solvents [1]. Polyimide materials have good dielectric properties. They exhibit a high resistance to radiation from both electrons and neutrons [2]. They are very stable both chemically and thermally; and are also durable and low cost [3]. Polyimides lend themselves to rapid fabrication techniques such as spinning, and they can be removed either isotropically or anisotropically rather quickly. Some of the properties of PMDA/BPDA/TFMOB polyimide useful in the manufacturing of MEMS devices are listed in Table I, from [4]. It is no surprise that they have found many applications in the fabrication of MicroElectroMechanical Systems (MEMS) devices. In fact, polyimide materials have been used in all phases of MEMS fabrication and operation.

Figure 1. Chemical structure of a typical polyimide: PMDA-ODA.

TABLE I
PROPERTIES OF PMDA/BPDA/TFMOB POLYIMIDE

PROPERTY	Value
Young's Modulus (in plane)	7.5 GPa
Young's Modulus (out of plane)	8.0-15.0 GPa
Shear Modulus	1.0-10.0 GPa
Poisson's Ratio(in plane)	0.35
Poisson's Ratio (out of plane)	0.1-0.45
Dielectric coefficient	2- 4
Coefficient of thermal expansion	$6.0 \times 10^{-6} \, C^{-1}$

The uses of polyimide range from lithography, to sacrificial layers, from mold materials to the active materials for a variety of sensors. Polyimides can be modified to be conductive or magnetic. They have found uses as actuators and hinges for micro robotic applications. Polyimides have been used in the fabrication of biometric sensors that measure topology for the detection of fingerprints [5]. Others have used polyimides in the construction of ultrasound scanners [6]. Polyimides have been used to create planar surfaces on printed circuit boards in the implementation of RF-MEMS switches [7]. MEMS fabrication techniques often create stresses within the material layers. Polyimides have been used to help create structures that can indicate the amount of stress found within thin aluminum layers [8].

Polyimide has been used in the manufacturing of MEMS devices in many ways. A 2 μm thick layer of polyimide has been used as a passivation layer to protect delicate circuits [9]. Dupont PI2555 polyimide has been used as an adhesive for packaging of MEMS devices [10]. Structural elements for a micro hot wire anemometer were constructed out of HD-4000 polyimide [11]. Polyimide structures that can rotate out of plane have been constructed using V-grooves and the contraction of polyimide as it cures:

$$\varepsilon = \frac{\Delta l}{l} = \frac{l_{uncured} - l_{cured}}{l_{uncured}} \tag{1}$$

where ε is the relative shrinkage and l is the lateral length [12]. Hinges have also been made using multiple layers of polyimide with wiring between the flexible polyimide isolation layers [13]. Polyimide hinges have also found uses in walking micro robots [14]. Biologically inspired micro robots have been constructed. These micro robots use polyimide hinges to mimic the motion of insect wings [15]. Along with hinges, thermal actuators can also be fabricated [16].

Sensors are another area where polyimide has found applications. Since some polyimide films can absorb water (Pyralin PI2723) they can be used to create simple capacitive humidity sensors [17]. Humidity sensors can also be created using Surface Acoustic Wave (SAW) techniques and the absorption properties of polyimides [18]. Magnetic micro actuators have been fabricated using polyimides (Dupont PI2555) and strontium ferrite particles [19, 20]. Another SAW device uses polyimide film (Dupont PI273) and N-Methyl Pyrrolidinone (NMP) for measuring temperature [21]. Dupont's PI2808 polyimide with a tensile strength of 210 MPa was used to fabricate shear

stress sensors arrays [22]. Both a floating element and thermal shear stress sensors have been developed using polyimide [23]. Polyimide has also found use in the construction of ultrasonic transducers [24, 25]. Hopefully this partial list of applications of has given the reader an idea of the potential that polyimides possess. The rest of this paper will focus on some uses of polyimides in the manufacturing of MEMS devices.

II. **Photosensitive Polyimide**

Lithography is the most used MEMS fabrication process. The key component of the lithography process is the use of photoresist. Photoresist is important not only because it is used so often but also because it is used to pattern each of the layers of materials that comprise a MEMS device. Polyimides have been applied as negative photoresist in a variety of fabrication techniques. Although photoresist materials play an important role in fabrication they are etched away and do not remain in the finished device.

Fabrication using polyimides as photoresist begins by spinning on a layer of photosensitive polyimide precursor called polyamic acid (Figure 3). The spinning rate is usually 1500-8000 rpm depending on the thickness of layer and the viscosity of the material. The thickness can be calculated using:

$$h = c\beta_0 k^{\beta_1} \omega^{\beta_2} t^{\beta_3} \tag{2}$$

where h is the thickness in meters, β_0 is a calibration constant, c is the polymer concentration by weight, k is the kinematic viscosity in centistokes, ω is the spin rate in rotations per minute, and t is the spin time in seconds. Note that β_0, β_1, β_2, and β_3 are all empirically determined factors [26]. For Durimide 7505 [27] the following values closely match the curves given in the data sheet: $\beta_0 = 3.0 \times 10^3$, $\beta_1 = 0.50$, $\beta_2 = -0.94642$, and $\beta_3 = -0.50$. When the spin time is 30 seconds, the polymer concentration by weight is c = 0.31, and the kinematic viscosity k = 1450 centiStokes, then varying the spin speed will generate the curve in Fig. 2.

The wafer is then soft baked at 100°C for one minute to remove solvents and to insure good adhesion. Next, for surface and bulk micromachining, the wafer is exposed to UV light through a mask. Cross linking of the polymer chains will only occur where the polyamic acid is illuminated, so polyimide (cross linked polyamic acid) is only created where it is exposed to UV light. In this example the polyimide acts as a negative photoresist. For Durimide 7505, the
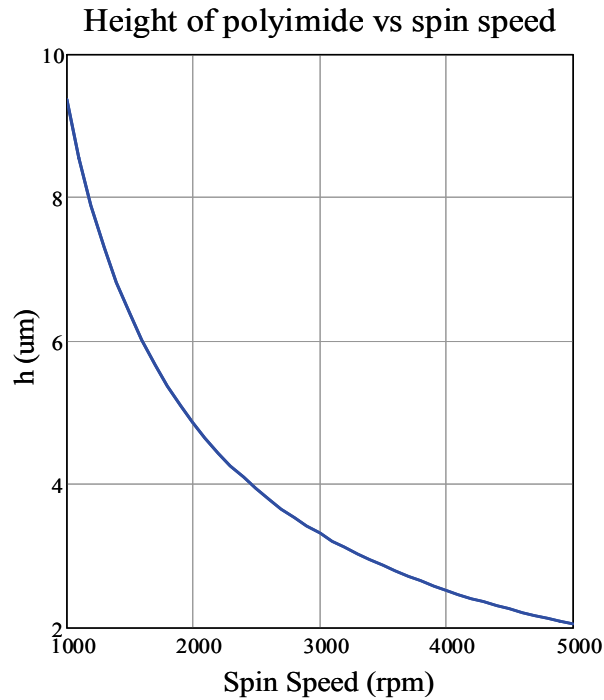
Height of polyimide vs spin speed

Figure 2. Graph of polyimide height as a function of spin speed for Durimide 7505.

wafer is post baked at 50°C for 1 minute. In the developing step, the un-exposed polyamic acid is dissolved away using NMP based strippers, acetone based strippers, or an organic stripper like Brewer Science, Inc.'s SafeStrip™. This will expose areas of the layer beneath and will leave portions of the layer covered by polyimide. Next, the layer beneath can be etched using an appropriate etching technique, like a HF etch which attacks oxides but leaves the polyimide alone. The final step is to strip the polyimide using a H_2SO_4 solution. The steps are shown in Figure 3. This process is usually repeated to pattern each and every layer used in the fabrication of a MEMS device.

In addition to photoresist uses, photosensitive polyimides can be used to directly pattern a polyimide. By directly using the photosensitive nature of some polyimides to pattern layers, one can reduce the number of steps involved in the fabrication of devices. Photosensitive polyimides have also found use in the packaging of integrated circuits [28], and can also be used in the packaging of MEMS devices. Photosensitive polyimides have been used to create micro pumps for controlled drug delivery systems [29]. Although not technically a MEMS application, photosensitive polyimides have been used in the development of micro superconductor insulating layers [30]. These applications demonstrate the many uses of photosensitive polyimides beyond the simple lithographic processes.
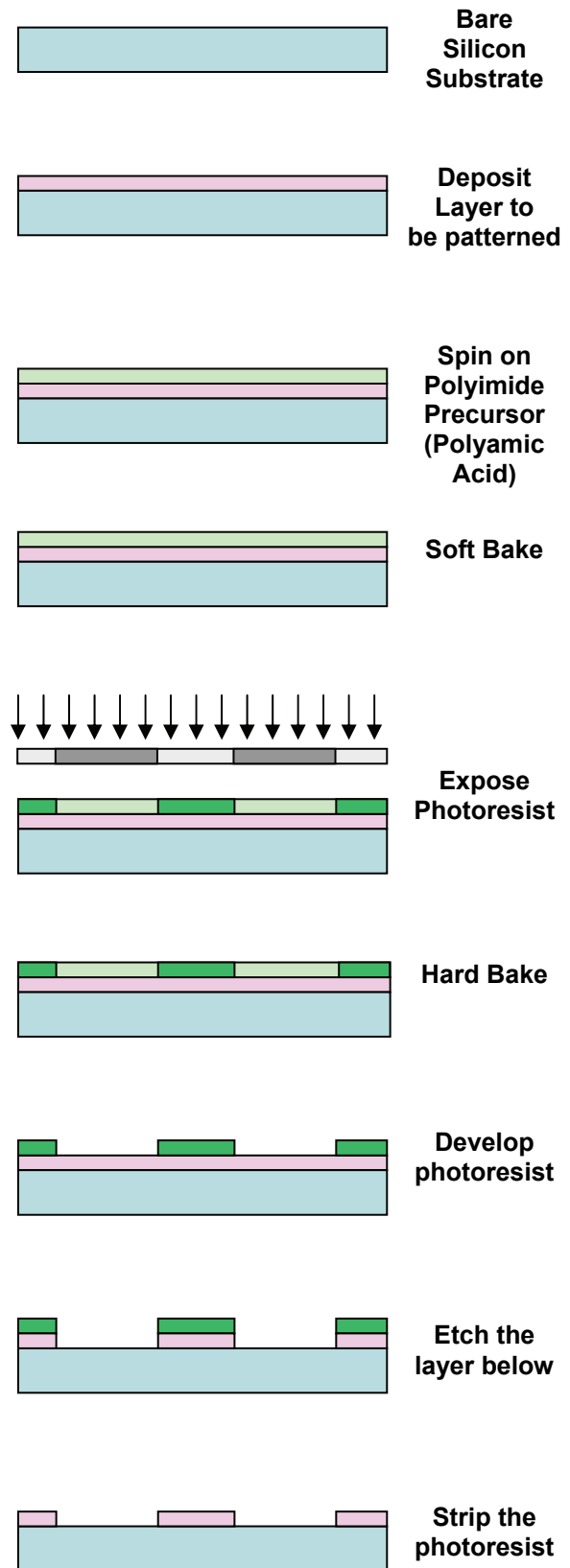

Figure 3. Fabrication sequence for patterning layers using photosensitive polyimide.

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III. Polyimide as a sacrificial layer

Polyimides are widely used as sacrificial layers during MEMS fabrication. These layers are used in creating three dimensional structures but are etched away before operation. In this application the polyimide layer is patterned by a photoresist into the volume of material that will be removed in the release step to allow the MEMS structures to move freely. Polyimides are used as sacrificial layers because they can be spun on to wafers quickly and easily and they can be etched either isotropically or anisotropically.

Manufacturing MEMS devices using polyimide sacrificial layers begins much the same as the previous fabrication sequence. The difference in this case is a thicker layer of polyimide that is spun on to act as the sacrificial layer (Figure 4). Next, a non-polyimide photoresist layer is used to pattern the polyimide areas that will comprise the sacrificial layer. For convenience, the soft bake, hard bake and development steps have been omitted from Figure 4, but they remain in the actual process flow. Next, the structural layer is deposited. Metals such as Aluminum and titanium are often used as the structural layer but silicon carbide and silicon nitride also work well [31]. Next, another layer of photoresist is applied and patterned. This sequence of steps, followed by etching of the metal layer and striping of the photoresist, will pattern the structural layer. If the device is simple like the cantilever beam used in the example Figure 4, then the last step is the sacrificial etch. Etching can either be dry [31] or wet. Various liquid etch rates for PI 2556 polyimide can be found in [32]. The polyimide can also be etched using an isotropic dry etch. This is called a dry etch because the etchant is gaseous or a plasma and not a liquid. Stiction issues are avoided when dry etchants are used.

Applications of polyimide as a sacrificial layer are numerous. The process has been used to fabricate microbolometers, where Polyimide is used as the sacrificial layer to create the 2.5 μm gap between the bottom reflector and the polysilicon top layer [33]. The process has also been used to create RF switches. In this case a 3 μm thick layer of polyimide is employed to create a space using DuPont PI2545 polyimide [34]. Capacitive ultrasonic transducers have also been fabricated using polyimide sacrificial layer to create cavities [35]. In this example Olin Probimide 114A polyimide was deposited 2~4 μm thick and the sacrificial etch was performed using HSO4:H2O2.

Bare Silicon Substrate

Deposit Polyimide Sacrificial Layer

Spin on Photoresist

Expose Photoresist

Develop photoresist

Etch the Polyimide layer

Strip the photoresist

Deposit Structural layer

Pattern and etch Structural layer
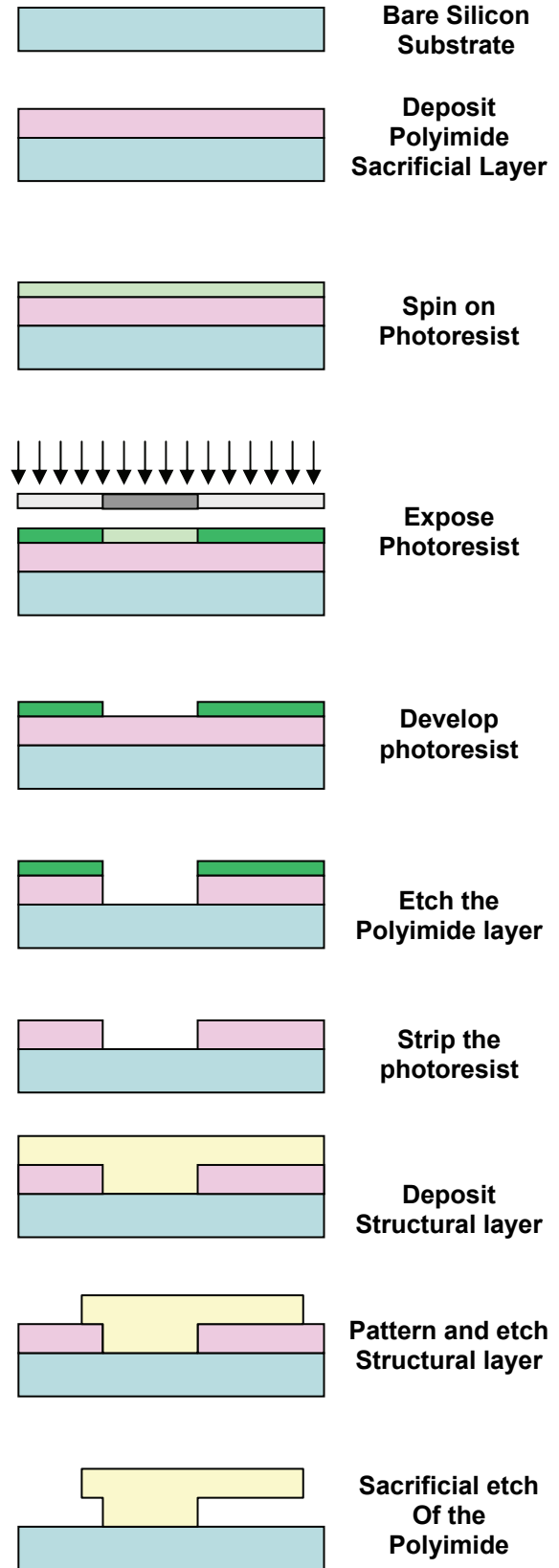
Sacrificial etch Of the Polyimide

Figure 4. Fabrication sequence for fabrication of a cantilever beam using polyimide as a sacrificial layer.

IV. Micro molding

Polyimide makes an excellent material for micro molds. Not only are they easy to deposit and pattern, but they are also durable and resilient to chemical and thermal degradation. It is for these reasons that polyimides have found application in high aspect ratio micro mold fabrication using LIGA techniques. The benefits of using polymers have also enabled the use of polyimides in technologies besides LIGA such as in expendable micro molds, and re-useable molds for contact printing.

A. LIGA High Aspect ratio devices using Polyimides

LIGA i.e. Lithographie, Galvanoformung, Abformung (German acronym for Lithography, Electroplating and Molding) is a manufacturing technique that begins with lithography. The lithography used in LIGA is similar to techniques discussed earlier but has two major differences. The Poly-Methyl-Meth-Acrylate (PMMA) photoresist that is deposited is very thick, on the order of 10μm to 1000μm [36]. The other big difference is in the use of x-rays for exposing the photoresist. For this paper, LIGA will be considered to be any techniques that use x-rays generated from a synchrotron. The use of Ultra-Violet (UV) light for exposing photoresist will not be considered to be a LIGA type of fabrication technology. This definition will allow a clear distinction between LIGA and UV fabrication techniques.

The LIGA fabrication sequence begins by depositing a thick layer of PMMA photoresist onto the substrate. This is exposed through a mask using x-rays of wavelength 0.1 nm to 1nm. A synchrotron x-ray source is necessary to generate enough energy to expose the extremely thick resist layers completely. Masks used in LIGA processes (i.e. x-ray masks) are different from the simple masks used for UV exposure. However, ultra thin silicon wafers with gold absorbers have been demonstrated to work well [37]. The photoresist is developed, and then metal is electroplated unto the conductive substrate. Next, metal is electroplated into the areas where the photoresist has been removed. The remaining photoresist is stripped, leaving a metal mold. This mold is filled with methacrylic casting resin through a gate plate [38]. Once the mold is filled and cured, the unmolding process removes the metal mold from the resin part. At this stage the resin part can be used, or a

metal part can be created by a second electroplating step. In this case the resin would be removed to leave a metal part.
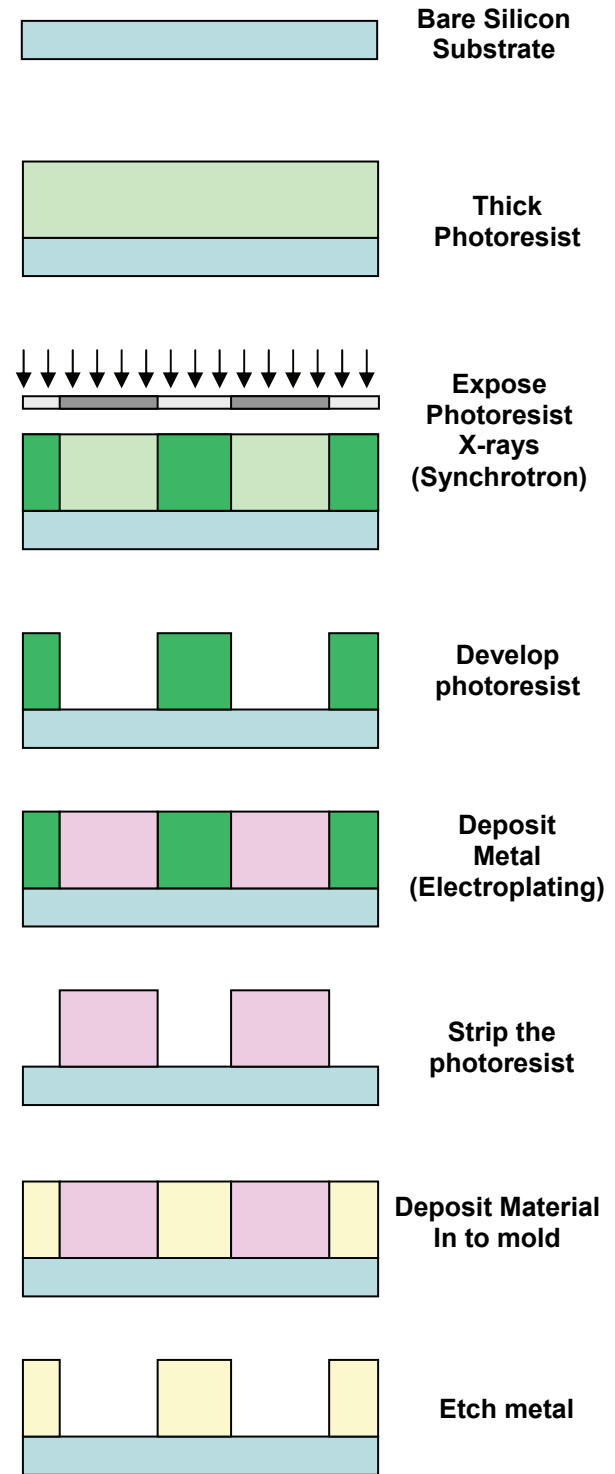
Figure 5. LIGA Fabrication sequence for manufacturing, using micro mold and electroplating technology.

For a standard LIGA process PMMA is the most common photoresist used, but often interest lies in the use of polyimide. When using polyimide as the photoresist, a thickness of 1000 µm can be obtained because photosensitive polyimide is 100 times more sensitive then PMMA [39]. This means that when using polyimide in a LIGA process, the radiation dosage of x-rays can be reduced by 100 times. This is very important because of the high costs associated with synchrotron operation.

B. Expendable molds

The hourly operating costs of LIGA techniques can be 8 times as much as the hourly costs for UV methods [40]. The high cost of LIGA fabrication, which is due to the high cost of synchrotron operation is the driving factor for the development of LIGA like techniques which use UV radiation instead of x-rays. Because polyimides have high sensitivity they have been investigated as a substitute for PMMA. High sensitivity is necessary when thick layers of photoresist are used. The fabrication sequence for expendable molds using polyimides is the same as is shown in Figure 5 for LIGA processing. The differences are that UV light is used instead of synchrotron generated x-rays and polyimide is used instead of the standard PMMA photoresist. The change of exposure sources causes a reduction in the thickness that can be achieved. This LIGA like process can yield a thickness of 3 to 150 µm for a single coat of polyimide [41]. Like the LIGA process the molds are filled using injection molding techniques and a gate plate [42].

C. Reusable flexible molds

Some applications do not require the destruction of the polyimide mold. To make reusable molds a process similar to the expendable mold process is used. A thick layer of polyimide is deposited and patterned using UV radiation. At this point the mold is complete. For contact printing the mold is filled with an inking material to be patterned in the printing process.

For some biomedical systems, polyimide molds are used as master molds to create molds out of Poly-Di-Methyl-Siloxane (PDMS) which is attached to a glass plate, cured, and then filled with the protein that is to be patterned [43]. Other biomedical applications involve the use of polyimide molds for transferring proteins and biological molecules such as neuroblastoma cells. The process begins with a double layer deposition of Dupont Pyralin 2611 polyimide 20

µm thick, next a layer of titanium is deposited and patterned to form a mask which is used in patterning the polyimide during an O_2 plasma Reactive Ion Etch (RIE). This creates a grid from which an elastomer mold can be fabricated. The finished mold is filled with a protein and printed onto glass slides [44].

V. PolyMEMs Process

Aerospace applications require new light weight, low power sensors. Piezoelectric sensors have demonstrated a potential for many applications, but some require a material that is compatible with MEMS processing and has a wider temperature range than is currently available. The new process must be inexpensive, require few fabrication steps, and must not require overly complex tooling or hazardous chemicals. It is preferred that the new material have the same or better chemical resistance than that of PolyVinyliDene Dluoride (PVDF). It is also preferred that the new material not require stretching during the poling process as required for PVDF, because stretching is incompatible with most MEMS processes. After investigating existing fabrication techniques, the development of a new MEMS fabrication process for aerospace applications was found to be necessary. Polyimides are clearly very versatile because they can have tailored material properties. The material properties are controlled by the addition or substitution of the constituent chemicals. For example, piezoelectric properties can be introduced into polyimide materials. This led us to create the PolyMEMS process.

The Virginia Commonwealth University (VCU) has developed a polyimide manufacturing process called PolyMEMS [45] which uses a piezoelectric polyimide ((β-CN)/APB/ODPA) developed at the NASA Langley Research Center. This polyimide has greater potential for MEMS processing because it exhibits greater chemical resistance than that of flouropolymers such as PVDF [46]. Another reason for developing a new process is the limitation of PVDF, which does not function above 80ºC [47]. Electroactive (piezoelectric) polyimides have the potential of functioning at temperatures up to 150ºC.

The processing steps for construction of a cantilever beam using the PolyMEMS process begin with an oxidized wafer (Figure 6). An aluminum layer is deposited and patterned to make the bottom or sense electrode. Next, a standard photoresist is deposited and patterned to act as the sacrificial layer (~1.3 µm). A second layer of aluminum is deposited and patterned to form the underside electrode for the polyimide

material. The piezoelectric polyimide is deposited over the entire wafer [48] using the spinning process (~2.15 μm). The wafers are then cured in an oven with a nitrogen purge at 90ºC for 2 ~ 4 hours. Next, the third layer of metal (Al) is deposited to form the top polyimide electrode. This metal layer is patterned using standard techniques. The layer is now used to form the mask for the patterning of the polyimide layer. The polyimide is etched using an anisotropic O_2 plasma etch. This etch is performed at 120W for ~80min for a 2.15 μm thick layer. This etch patterns the polyimide to the same shape as the third layer of metal. The plasma etch is followed by a sacrificial etch where the standard photoresist is etched away. It is important that this etch does not allow the moving parts to be permanently bonded to the underlying layers. This could be caused by capillary action during the drying step of a wet etch. In this case, acetone is the first the etchant, but it is replaced with methanol, which is then replaced with pentane and finally, hexane. Each successive liquid has a lower surface tension [49]. In this example the process would leave the cantilever free to move. In most designs stiction bumps are normally added to reduce the contact area between moving parts and any underlying layers or the substrate. Before the device is operational the polyimide must be cured and polled. The polling process involves baking the wafers at 227ºC for two hours while placing a voltage of 100V on the two electrodes surrounding the polyimide (metal layers 2 and 3), to orientate the dipoles within the piezoelectric polyimide. During operation, movement causes a voltage to be generated between metal layers 2 and 3. This voltage is proportional to the cantilever stress; therefore the device behaves as an electroactive polyimide sensor.

The PolyMEMS process has many applications in both the aerospace and biomedical fields. Polyimides have proven to be non-toxic and resistant to organic solvents [52], therefore, they have potential for biomedical applications. The process can withstand higher temperatures than that of standard piezoelectric polymer materials [50]. The process is also compatible with standard integrated circuit processing techniques; it does not require stretching during the poling process, so it has the potential to allow for integrated sensors and electronics. This will enable sensors with reduced power, mass and volume to be developed. In fact, the emergence of MEMS make picosatellites (1 mg to 1g mass) feasible [51]. For these reasons the PolyMEMS process can be applied to aerospace sensors.
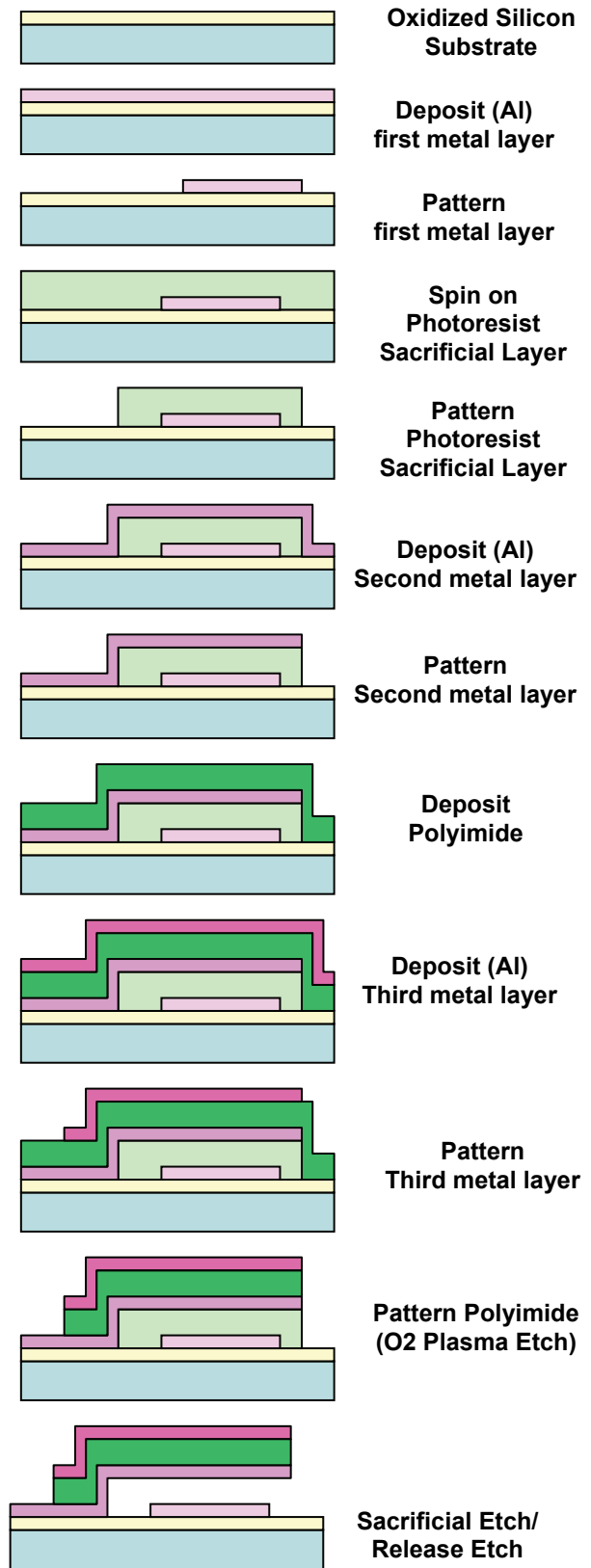

Oxidized Silicon Substrate

Deposit (Al) first metal layer

Pattern first metal layer

Spin on Photoresist Sacrificial Layer

Pattern Photoresist Sacrificial Layer

Deposit (Al) Second metal layer

Pattern Second metal layer

Deposit Polyimide

Deposit (Al) Third metal layer

Pattern Third metal layer

Pattern Polyimide (O2 Plasma Etch)

Sacrificial Etch/ Release Etch

Figure 6. PolyMEMS fabrication sequence for manufacturing a cantilever beam using polyimide technology.

VI. Conclusions and Outlook

This work presents the many uses of polyimides in the manufacturing of MEMS devices. Historically photosensitive polyimides have been used in MEMS processing techniques. This is mainly due do the fact that polyimides are easy to deposit, easy to etch both isotropically and anisotropically, and have many other benefits such as a low dielectric constant and thermal and chemical stability. These benefits also allow non-photosensitive polyimides to be used in many MEMS applications such as for structural and sacrificial layers. The desire for high aspect ratio devices drove the use of thick polyimide films that are UV curable as a method of creating molds much like those created using a standard LIGA process, but at a much reduced cost. A related technology uses polyimides to create flexible molds for imprinting. Humidity, temperature, magnetic, and ultrasonic sensors can be constructed using polyimides.

The versatility of polyimides has led us to develop a new fabrication process technique utilizing an electroactive polyimide. This new type of MEMS fabrication process (PolyMEMS) was presented. The use of this new process will allow for new sensor applications in harsh, high-temperature aerospace environments. This new process will enable a new class of polyimide sensors to be developed. One such device is the new type of ultrasonic sensor that can be constructed using electroactive polyimides. This device has the potential to become a low cost, Capacitive Micromachined Ultrasonic Transducer (CMUT) sensor for aerospace applications [53].

The outlook for polyimides in the future is very positive. As the progress on new materials and new polyimides continues, so will the development of new micro devices that employ polyimides. Researchers have yet to exploit all of the potential that exists in polyimides. Therefore, the future holds the promise of new and better devices, sensors, and actuators due in part to polyimides.

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14. ABSTRACT

Since their invention, polyimides have found numerous uses in MicroElectroMechanical Systems (MEMS) technology. Polyimides can act as photoresist, sacrificial layers, structural layers, and even as a replacement for silicon as the substrate during MEMS fabrication. They enable fabrication of both low and high aspect ratio devices. Polyimides have been used to fabricate expendable molds and reusable flexible molds. Development of a variety of devices that employ polyimides for sensor applications has occured. Micro-robotic actuator applications include hinges, thermal actuators and residual stress actuators. Currently, polyimides are being used to create new sensors and devices for aerospace applications. This paper presents a review of some of the many uses of polyimides in the development of MEMS devices, including a new polyimide based MEMS fabrication process.

15. SUBJECT TERMS

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