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Photoresist patterned thick-film piezoelectric elements on silicon

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Abstract A fundamental limitation of screen printing is the achievable alignment accuracy and resolution. This paper presents details of a thick-resist process that improves both of these factors. The technique involves exposing/developing a thick resist to form the desired pattern and then filling the features with thick film material using a doctor blading process. Registration accuracy comparable with standard photolithographic processes has been achieved resulting in minimum feature sizes of <50 μm and a film thickness of 100 μm. Piezoelectric elements have been successfully poled on a platinised silicon wafer with a measured d_{33} value of 60 pCN^{-1}.

Keywords PZT · Doctor blading · Thick photoresist

1 Introduction

Screen printed lead zirconate titanate (PZT) thick films have been used in numerous micro electromechanical systems (MEMS) applications such as micropumps [4, 6, 8], accelerometers [1] and resonant sensors [2]. The approach is attractive since it is a simple, low-cost process for depositing thick layers of piezoelectric material in the desired pattern. The process, however, is fundamentally limited by the resolution (minimum feature size typically >100 μm) and alignment accuracy (±50 μm) achievable, compared to micromachined structures that are defined using standard photolithographic techniques. Print misalignments can affect device performance considerably. In the case of resonant sensors, where vibration is excited and detected by screen printed PZT elements, misalignments will reduce the coupling efficiency to the resonator.

These misalignment issues are demonstrated on the triple beam resonator shown in Fig. 1. The PZT is offset to the left and the top electrode is offset from the PZT to the left and down. The net effect of this is inefficient excitation, reduced vibration detection signal and reduced resonator Q-factor. When tested, the output from resonators such as that shown was barely detectable, which highlighted the need for a process capable of improved alignment of features and between layers.

The use of thick photoresist as a micromould has been documented for applications such as electroplating [3, 7] and advanced packaging [5]. This approach provides the alignment accuracy and resolution associated with optical lithography. This paper details a technique of doctor blading screen printable piezoelectric pastes into thick resist moulds, providing improved alignment accuracy and resolution over the conventional printing approach.

A key attraction of screen printing is the thickness of the deposited material, which can be up to 100 μm. In the case of active materials, increased thickness can significantly improve the performance in many actuation applications. Therefore a resist-based patterning process must be capable of producing a minimum feature size of ideally <50 μm with a resist thickness of 100 μm. A second requirement is that the resist must be able to withstand the temperatures used to dry the paste (>100 °C). Once the paste is dried, the

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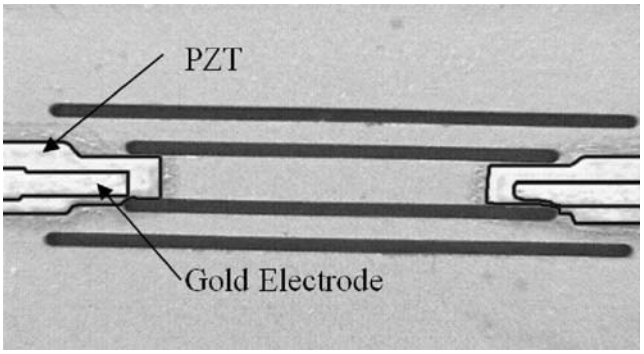

Fig. 1 Poorly aligned screen print of PZT and top electrode on a resonant silicon sensor

resist must be easily removed from the substrate whilst leaving the dried deposited material in place. This paper presents an investigation into the suitability of four resists, two positive (Shipley SPR220-7, Clariant AZ9260) and two negative (Ordyl AM150, BPR-100). SU-8 resist has been evaluated previously and was found to be too difficult to remove from the wafer for this application.

2 Experimental procedure

Three of the four resists are liquid at room temperature and can be applied by spin coating on a silicon wafer. Manufacturer specifications for spin speed, duration, number of coats and baking time were followed initially and subsequently adjusted to produce the required thickness. Ordyl AM150 is a dry resist and can be applied by laminating the silicon wafer. In each case the substrate was exposed through a test pattern using a Hybrid Technology Group (HTG) contact aligner with a UV light source (350–450 nm spectrum mercury lamp) at 3.1 mW cm^{-1}. Where necessary, any edge bead from the resist spinning process was removed to ensure good contact between the mask and wafer.

The specifications provided by the manufacturer were initially used for exposure times and development processes and were subsequently optimised. The resist profiles were evaluated using an Alpha-Step 200 surface profiler and optically inspected with a microscope and a Hitachi FEG-SEM.

After visual inspection, the wafers were ready for the doctor blading process. This involves smearing the paste across the wafer using a rubber squeegee, which results in the paste filling the resist mould. The resulting film is dried and fired as shown in Fig. 2, The PZT paste is deposited first, followed by the gold paste which is inherently aligned to the PZT layer as the same mould is used.

The PZT thickness can be increased by re-blading another layer of PZT following the first drying stage. This can be done providing that there is sufficient space remaining for the gold to be deposited on to. The amount of PZT shrinkage during drying is determined by the make up of the paste, which is a mixture of glass, milled PZT powder and a pine oil vehicle [9]. More pine oil creates a less viscous paste, which flows into the features more readily but has a higher degree of shrinkage. Once the PZT and gold have been deposited the resist must be removed before the films can be fired.

3 Resist evaluation

Table 1. shows the optimised parameters used to assess the four resists.

SPR220-7 is not designed for thick resist applications but was included in the experiment as it is a readily available resist with known processing parameters. Three layers applied at low spin speeds produces a depth of 100 μm. At this thickness, the resist was approaching (if not exceeding) the limits of its use. Removal of the resist proved problematic at such depths. This was partly due to the pigmentation of the resist being too high and hence limiting the amount of UV light reaching the bottom of the features during exposure. This necessitated multiple exposure/develop cycles hence reducing resolution and registration of the features. All resist must be removed, since any remaining in the bottom of the feature will prevent the deposited paste from bonding to the substrate. The resist also proved sensitive to the temperature used for drying the paste: slight bubbling and reflow were observed. Therefore SPR220-7 was found not to be suitable for the process.

The dry film nature of the Ordyl AM150 resist meant that a thickness of 100 μm could be achieved with two applications of the 50 μm thick film. The dry film resists are very consistent which leads to a high level of repeatability in the process. This resist produced excellent results as shown in Fig. 3. Features demonstrated vertical sidewalls with no resist residue visible on the surface of the wafer. Removal of the resist, however, proved to be much

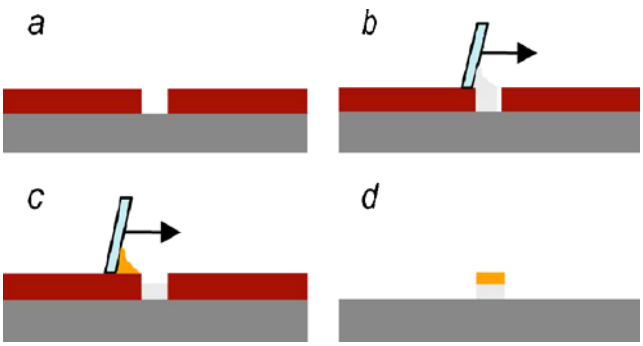

Fig. 2 Doctor blading PZT paste onto wafer. **a** Resist with patterned features. **b** Doctor blade PZT paste across wafer. **c** Dry PZT, shrinkage leaves space to doctor blade top electrode. **d** Dry electrode, remove resist then fire

Table 1 Optimized parameters for photoresists.

	SPR220-7	AZ9260	BPR-100	Ordyl AM150
Application	30 s at 500 rpm, 30 s at 650 rpm, Softbake 100 °C for 15 min, ramp from 60 °C for first layer.	10 s at 500 rpm, 20 s at 900 rpm, Softbake at 95 °C for 4 min—layer 1, 100 °C for 10 min—layer 2, 100 °C for 15 min—layer 3.	10 s at 500 rpm, 30 s at 800 rpm, Softbake at 100 °C for 7 min.	Laminated at 115 °C and roller speed 0.15 m min^{-1}
Application cycles	3	3	1	2
Attained depth	100 μm	100 μm	100 μm	100 μm
Exposure	500 s develop, 100 s	650 s	330 s	40 s
Develop	4 min then, 2 min	6 min at 25 °C	Untried	3 min

less straightforward. As the doctor bladed PZT paste was dried, the elevated temperatures caused the resist to harden and become insoluble to solvents and developing solutions. It could not be removed without causing damage to the doctor bladed paste. It was therefore decided that this was not suitable for use in the process.

BPR-100 is a high viscosity resist and was found to be unsuitable for processing using the equipment available. The resist is designed to be applied using a pump to control flow rate and prevent air from entering the system. Achieving an application with no bubbling using a pipette or pouring the resist was very problematic, the resist was not level across the wafer making reliable contact exposure difficult. As a result, the high definition features required by the process specification could not be achieved.

Finally, the AZ9260 resist achieved the target depth of 100 μm by applying three layers, each with a 900 rpm peak spin speed. Each layer of resist was allowed to settle for 8 min before 10 min soft baking at 90 °C for the first layer then 100 °C for layers two and three. The delay between spinning and softbaking prevented the resist from blistering and pulling back from the wafers edge upon heating.This led to a well formed 100 μm resist layer with very few defects.

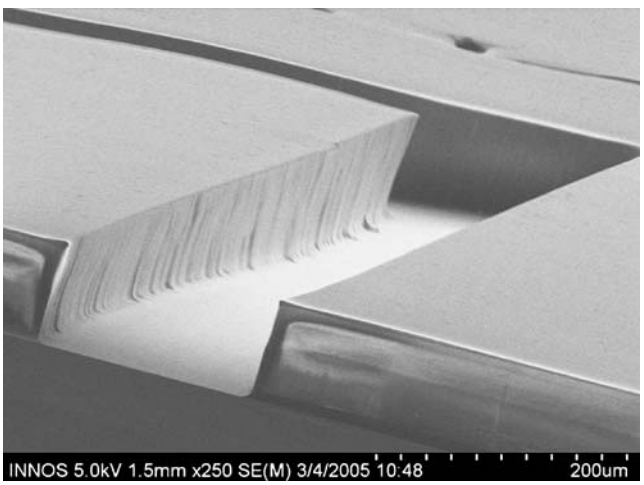

Fig. 3 SEM photograph of Ordyl AM150 trench

The lighter colouring of AZ9260 gave good transmission of the UV energy during exposure [3] and a single exposure and development was found able to process the film. The optimised parameters proved to be a 650 s (2,015 $mJcm^{-2}$) exposure and a 6 min development in a 25 °C bath of one part AZ400K developer mixed with four parts deionised water. The development stage was very sensitive to temperature and agitation. Excessive agitation caused over development and non-vertical sidewalls.

Once optimised, AZ9260 proved to be the most suitable resist for the doctor blading process as it was capable of producing the desired resolution and required depth. It was also able to withstand the drying temperatures.

4 Resist removal

Initial tests evaluated the use of solvents, such as acetone, to remove the resist. It was found that once the resist became sufficiently thick (in excess of 60 μm) the time taken for the solvent to remove the resist also resulted in solvent damage to the PZT features. Shorter immersion times, with drying intervals between, were evaluated but the solvent could not be driven from the PZT at a sufficient rate to prevent damage.

The use of KOH as an alternative was evaluated and a 4% KOH solution (10 g KOH crystals in 250 ml DI water) at 35 °C was found to remove the resist in 2 min although it caused slight damage to the features. As depicted in Fig. 4, by splitting the process into two 1 min immersions, with a deionised water rinse and infra red dry between stages, the

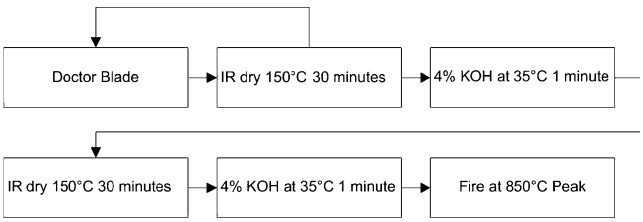

Fig. 4 Doctor blading and resist removal process

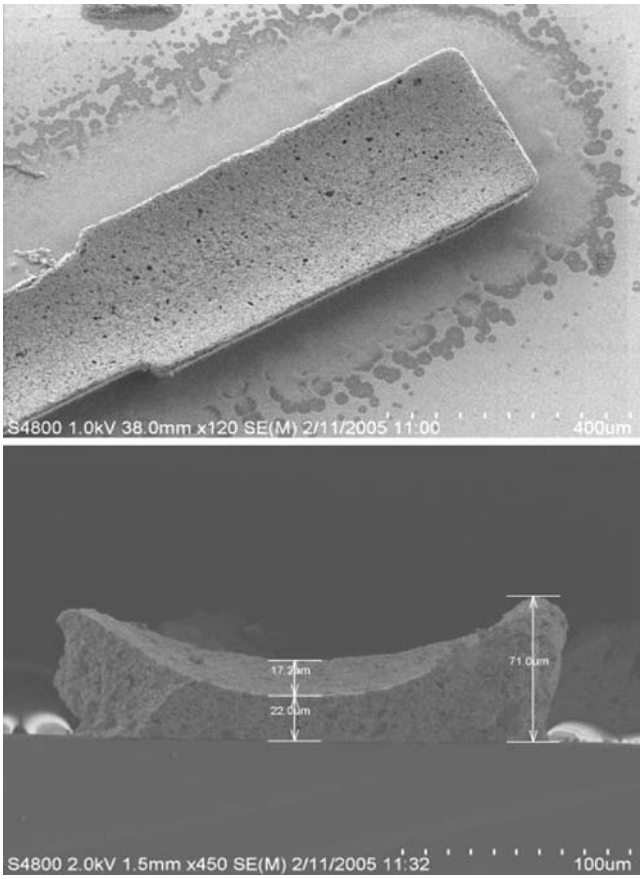

Fig. 5 SEM photograph showing shouldered beam of printed PZT

resist was removed without damaging the features. The wafer was then fired to sinter the paste.

5 Process summary

The complete process can be summarised as follows:

- Dehydrate wafers for 1 h at 150 °C.
- Prime wafers for resist.
- Spin first layer of resist at 900 rpm peak for 30 s, ramp 900 rpm/s.
- Allow to stand for 8 min.
- Soft bake at 90 °C for 10 min.
- Allow to cool for 8 min.
- Repeat the resist application steps for the second and third layers using 100 °C soft bake temperature.
- Expose with a 2,015 $mJcm^{-2}$ exposure.
- Develop in 1:4 AZ400K for 6 min.
- Blade PZT paste across the surface of the resist with a pressure of 5 N.
- Infra red dry at 100 °C for 30 min.
- Blade gold paste across the surface of the resist with a pressure of 5 N.
- Lightly buff excess gold from surface.
- Infra red dry at 100 °C for 30 min.
- Immerse in 4% KOH solution at 35 °C for 1 min.
- Repeat infra red dry and KOH immersion with a fresh solution.
- Infra red dry at 100 °C for 30 min.
- Fire at 850 °C PK, ramp 850 °C/h.

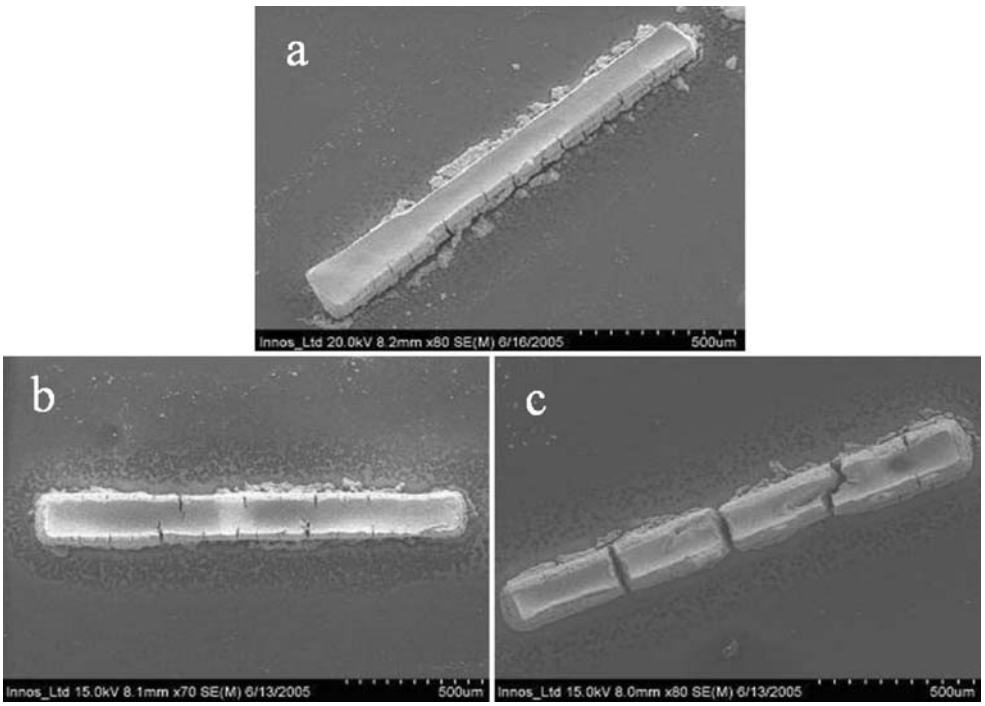

Fig. 6 SEM photographs of varying paste viscosities: **a** High viscosity. **b** Medium viscosity. **c** Low viscosity

6 Results

An initial investigation involved doctor blading a single layer of paste onto the wafers to establish the feasibility of the process. Figure 5, shows a shouldered beam of PZT with good paste cohesion and a resolution of less than 40 μm.

From the cross-section it can be seen that the PZT is only 22 μm thick in the centre and 71 μm at the sides. This leaves a useful space into which the gold can be deposited. The piezoelectric properties, however, will be reduced by the lack of active material.

The addition of a second PZT blading step increased the depth of the deposited piezoelectric film as expected, but resulted in cracking of the film. This was investigated and shown to be a function of the viscosity of the paste, which depends upon the amount of vehicle in its composition. The paste consists of PZT and glass particles mixed with an organic vehicle. During drying, the vehicle evaporates leaving the PZT and glass particles. Three pastes were evaluated: high viscosity (45 PaS), medium viscosity (25 PaS) and low viscosity (17 PaS). The results of the tests are shown in Fig. 6, It can be seen that the paste with the highest viscosity, least vehicle, performed best, giving the least cracking.

PZT elements with a gold top electrode were successfully deposited onto a platinised silicon wafer. Using the platinum as the bottom electrode and the gold layer as the top electrode the PZT was poled at 130 °C and 220 V for 30 min. A d_{33} value of 60 pCN^{-1} was measured for an element 2,300 μm long and 180 μm wide using a PM35 piezometer.

7 Conclusions

A process has been developed that is capable of depositing thick-film material with resolution and alignment accuracy comparable to standard photolithographic processes. The technique will be applied to deposit actuation and sensing elements onto a resonating silicon triple beam pressure sensor.

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