



## **A New Procedure for the Application and Curing of Polyimide Film on Gold Coated Silicon Wafers**

**by Kimberley A. Olver**

**ARL-TR-5031**

**November 2009**

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## **A New Procedure for the Application and Curing of Polyimide Film on Gold Coated Silicon Wafers**

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**Sensors and Electron Devices Directorate, ARL**

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## 1. Introduction

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Polyimides were originally developed by the DuPont Chemical Company in the 1950s as high temperature engineering polymers, meaning that these materials were designed and manufactured for their structural and functional properties for use in mechanical, civil, and medical engineering applications. Polyimides were found to have the combination of thermal stability ( $>500\text{ }^{\circ}\text{C}$ ), mechanical robustness, high ductility, low coefficient of thermal expansion (CTE), and chemical resistivity to many corrosives and solvents. They were also found to have excellent dielectric properties (1).

Polyimides are in a group of chemicals known as “thermoset plastics,” meaning that they are liquid (resin) prior to undergoing a curing process involving heat. This curing process irreversibly changes the resin into a rigid material by cross linking the polymer molecules (imidization) (2), and at the same time removing the solvent carriers and other volatiles. Polyimide starts as an imide-containing polymer molecule. There are two general structure types of polyimides: linear structure polyimides in which the imide group is part of a polymer chain, and heterocyclic structures, which are polymer molecules in which the imide group is part of a cyclic unit in the chain (3).

For the semiconductor industry, the polyimide resin is applied in much the same way as photoresist. It is spin coated onto the wafer and cured. The curing process removes the solvents and creates a durable polyimide layer.

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## 2. Experimental Procedures and Results

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In the semiconductor industry, polyimide films have several important uses. They are used as protective overcoatings for thin or fragile films of metal or other structures, and have been used as interlayer dielectrics in thin film multichip modules (MCMs). Polyimides used in the semiconductor industry are typically spin coated onto the wafer, allowing for a flat uniform thickness coating of resin material. The resin is then cured into a smooth structural layer, which can be patterned using a lithographic process in much the same way as any other semiconductor layer. Some polyimides contain light sensitive chemicals and can be patterned without the use of photoresist, making fabrication even simpler.

A project in the Electro-Optics/Infrared Materials and Devices branch provided an excellent example of the use of polyimide. For this project, a  $2.8\text{-}\mu\text{m}$ -thick polyimide layer of HD Microsystems PI-2610 polymer liquid was spin coated onto a gold coated silicon wafer, cured, and patterned with  $1500\text{-}\text{\AA}$ -thick gold grating lines. The lines were  $1.5\text{ }\mu\text{m}$  wide and spaced

1.5  $\mu\text{m}$  apart. A metal liftoff technique was used to remove the excess gold (figure 1). The following process steps were developed and found to give repeatable results.

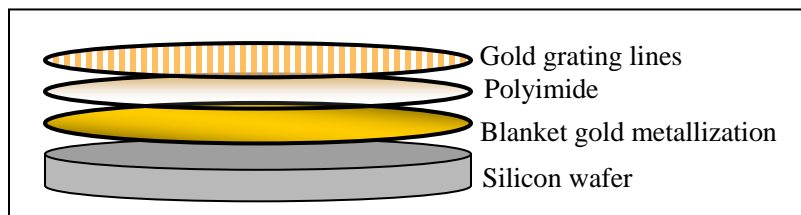


Figure 1. Diagram of the patterned wafer.

## 2.1 Cleaning of Silicon Wafer

A 2-in silicon wafer was cleaned using a standard semiconductor wafer cleaning procedure: an acetone soak and rinse, followed by an isopropyl rinse, and a final rinse in deionized (DI) water. The wafer was blown dry with nitrogen gas.

## 2.2 Metallization of Silicon Wafer

The clean silicon wafer was placed into the bell jar of a vacuum electron-beam evaporator and a blanket e-beam evaporation of 1500 Å of gold was deposited. The wafer was then removed from the evaporator.

## 2.3 Coating the Silicon Wafer with Polyimide

Due to its short shelf life at room temperature, the polyimide resin material was kept refrigerated as recommended by the manufacturer. Bringing the polyimide up to room temperature before opening the container guaranteed that water vapor would not contaminate the resin.

Initially, the polyimide resin was brought to room temperature in the original container and carefully poured onto the wafer center in an attempt to avoid adding bubbles to the resin. However, after the curing process, small bubbles were seen trapped in the layer and were found to cause problems with photolithography. The solution was to bring the resin to room temperature in its original container, then degas the polyimide just prior to applying it to the wafer. The polyimide was brought to room temperature overnight in its sealed container. A small amount (approximately 20 ml) of the polyimide was then poured into a 50-ml glass beaker, and the beaker was placed into a Scienceware Mini Vacuum Desiccator (figure 2). A vacuum was pulled, and the polyimide was degassed for several hours just prior to using. This de-gassing aided in the polyimide not having trapped bubbles in the final product.

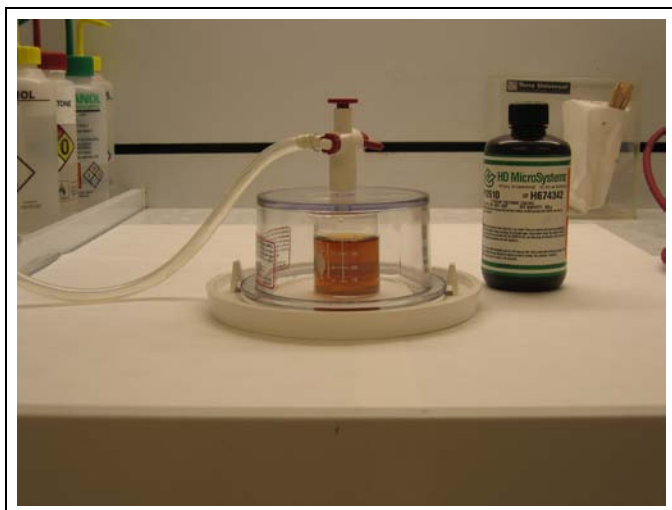


Figure 2. Image of the desiccator setup.

An adhesion promoter was spun onto the wafer prior to the polyimide resin. HD Microsystems adhesion promoter VM-652 was spun onto the gold coated clean wafer at 3000 rpm for 30 s, and the wafer was placed on a 90 °C hot plate for 90 s. For our purpose, a cured polyimide thickness of 2.8  $\mu\text{m}$  was necessary. After some experimentation with application spin speed, the correct parameters were achieved. The polyimide resin was spread at 1000 rpm for 3 s followed by a final spin of 2500 rpm for 25 s. This gave a consistent film thickness of 2.8  $\mu\text{m}$  across the wafer. The wafer was removed from the spinner, and cured.

## 2.4 Curing the Polyimide

Curing the polyimide film involved removing the solvents and other volatiles from the resin slowly and, with the addition of heat, causing the resultant imidization of the polymer resin into a durable polyimide film.

The full curing of the polyimide resin is typically done in steps. After spinning on the liquid polyimide material, the coated wafer was placed on a 90 °C hotplate for 90 s then moved to a hotplate at 180 °C for 90 s for a soft cure. To fully cure the polyimide, a three-zone furnace was used. The wafer was placed in the center of the furnace on a glass carrier. Nitrogen gas was flowed through the furnace. The furnace was turned on and ramped to 180 °C. It was held at 180 °C for 30 min, and then increased to 280 °C. It was held at 280 °C for 30 min, and then increased to 350 °C. It was held at 350 °C for 1 h (4). The furnace was then turned off, and the wafer was allowed to cool to room temperature in the furnace overnight.

## 2.5 Photolithography on Polyimide Wafer

Photolithography on the fully cured polyimide was achieved by first spin coating the wafer with VM-652 Adhesion Promoter at 3000 rpm for 30 s, followed by a hot plate bake at 90 °C for 90 s. This was the manufacturer recommended adhesion promoter for this polyimide. The wafer was removed from the hotplate and cooled. After cooling, a spin coating of AZ #5214 image reversal

photoresist was applied at a speed of 4000 rpm for 40 s. This photoresist was hot plate baked at 110 °C for 2 min, exposed on a JBA vacuum contact mask aligner for 18 s with a bulb intensity of 5 mW/cm<sup>2</sup>, hotplate baked at 124 °C for 40 s, and then flood exposed for 23 s. The photoresist was then developed in AZ 312 MIF photoresist developer for 45 s and rinsed in DI water for 1 min. The wafer was then dried with nitrogen gas. The resulting photolithography was inspected under a microscope for complete clearing (figure 3).

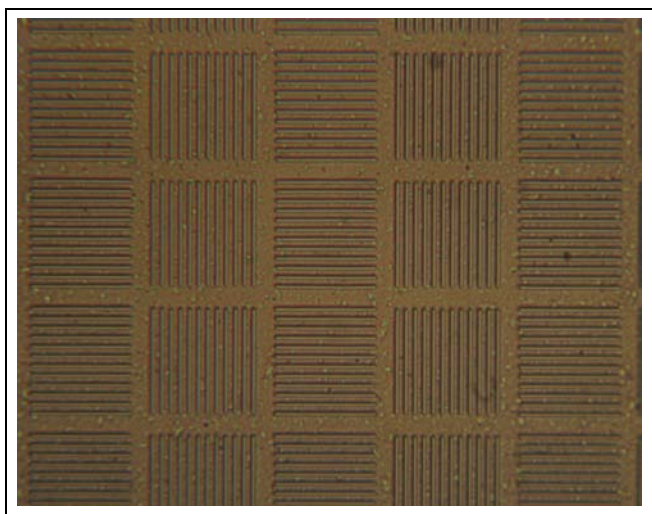


Figure 3. Image of patterned photoresist on surface of polyimide.

## **2.6 Plasma Ashing of Patterned Polyimide Wafer**

Prior to loading the patterned polyimide wafer into the evaporator, a photoresist cleaning in a plasma asher was performed. The wafer was placed in a Plasma Therm Barrel Asher with oxygen (O<sub>2</sub>) (5 sccm) at 150 W for 120 s followed by argon (Ar) (5 sccm) at 150 W for 60 s. The O<sub>2</sub> plasma cleans up any residual photoresist that did not rinse away with the developing/rinsing step and the Ar plasma slightly roughens the exposed polyimide material and helps with the metal adhesion in the next step.

## **2.7 Metallization of the Cured Polyimide Wafer**

The wafer was loaded into a vacuum electron-beam evaporator and the system was pumped down to a pressure of  $1 \times 10^{-6}$  torr. Then, 150 Å of chromium followed by 1000 Å of gold was deposited on the wafer. The wafer was unloaded from the evaporator, and a metal liftoff of the photoresist and excess metal was done. This process consisted of soaking the wafer in acetone for several minutes, rinsing with acetone, isopropyl alcohol, and DI water, then drying with nitrogen gas. An image of the final product is shown in figure 4.

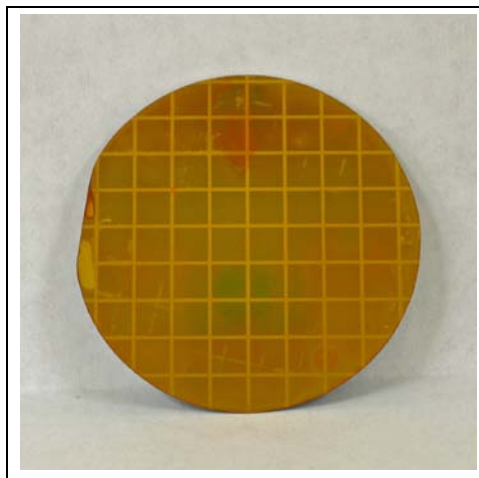


Figure 4. Image of finished wafer.

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### 3. Conclusions

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A new repeatable procedure was developed for the purpose of fabricating a uniform thickness polyimide coating on a 2-in silicon wafer and patterning the resulting polyimide with gold grating lines. Polyimides are in a group of polymers known as thermoset plastics, which means that they start as liquid resin material prior to undergoing a curing process involving heat. They are chemically resistant to many corrosives and solvents, have low CTEs, and have excellent dielectric properties. In the semiconductor industry, polyimides are used for a variety of purposes. They are relatively easy to apply, and once cured are stable, durable layers.

The new procedure consisted of metalizing a 2-in silicon wafer, spin coating a vacuum degassed polymer resin onto the wafer, curing the resin resulting in a polyimide film, performing photolithography on the polyimide surface, and lifting off the excess metal.

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