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Preparation of Large-area Electron-transparent Samples from Silicon Devices

Abstract: A technique using a pulsating chemical jet has been developed for thinning and polishing large areas (750 to 1000 μm in diameter) of silicon devices. The thickness can be reduced to a few micrometers. This technique has been used to prepare bipolar and FET samples for transmission electron microscopy. Physical characterization of more than twenty devices can be achieved by one sample preparation.

Introduction

While the preparation of samples for electron microscopy from singly diffused silicon wafers [1] is relatively simple, it is difficult to obtain adequate electron transparency from wafers containing multiple diffusions because of enhanced chemical attack in the p regions. In planar, bipolar device wafers, the regions of interest are situated in an epitaxial layer a few micrometers thick grown on a suitable silicon substrate. To examine these regions it is necessary to remove the substrate uniformly and completely. In most bipolar devices the thickness of the epitaxial layer is suitable for high-voltage microscopy. For example, 5-μm-thick silicon can easily be examined using a 1-MeV electron microscope. However, with lower voltage microscopy, further thinning of the epitaxial layer is necessary. For other planar devices, e.g., the FET (field-effect transistor), most of the substrate must be thinned uniformly to the thickness (1 to 2 μm) in which diffusions are made. In both cases, uniform polishing is essential.

In the past, jet techniques [1,2] have been reported for the preparation of electron-transparent samples from silicon. Various problems have been encountered, however, such as lack of jet stability, imprecise control of the area thinned, nonuniform thickness of the polished area, and limited size of the electron-transparent area (1 to 5 μm in diameter). An electrochemical jet technique [3] has been reported for polishing large wafers. Its application in the preparation of uniformly thin, large areas on small disks appears to be limited because of electrical contact problems [4]. Electrochemical bath polishing is another possibility. Particular interest lies in the work of Smith et al. [4], who separated individual transistors by electrochemically dissolving the substrate and isolation areas (the p regions are attacked preferentially). The success of Smith's technique depends on some specific device configurations and cannot be easily extended to others.

In silicon devices today, defect density is very low. For example, a silicon wafer having 10^4 dislocations per square centimeter contains only one dislocation line per 10^4 square micrometers. It has been reported [5] that only a specific configuration or particular types of imperfections (such as stair rods in p-n junction breakdown) affect the electrical properties of the device. Existing techniques thus cannot be used successfully for studying correlation between structural imperfections and electrical properties.

The present investigation was undertaken to develop a general technique for preparing large electron-transparent areas from silicon device samples. Transmission electron microscopy can then be performed, section by section, at the highest possible magnification and resolution, so that no structural imperfection is left undetected.

Polishing apparatus

The polishing apparatus we developed is a modified pressurized-jet setup (Fig. 1) similar to that reported by Das and Radcliffe [6]. The equipment consists of a Teflon container (A), 8.89 cm (3.5 in.) in diameter, with a screw-on lid (B) through which a vertical syringe (C) is inserted. The syringe has a nozzle (D) and an axial hole (E) 0.203 cm (0.080 in.) in diameter through

which polishing solution is pumped at the rate of 30 to 40 pulses per minute at a pressure of 117 to 138 Pa (17 to 20 psi). This pulsation of the chemical jet can also be achieved with a minor modification of the syringe. The polishing solution is ejected from four orifices (F), 0.762 mm (0.03 in.) in diameter. Excess electrolyte is recycled to the pump as shown by the arrows in Fig. 1. The sample, 0.318 cm (0.125 in.) in diameter and 0.281 mm (0.015 in.) thick, is placed with the substrate side facing the flow of the polishing solution (Fig. 2) on a seat that is 0.330 cm (0.130 in.) in diameter and 0.254 mm (0.010 in.) in depth. The sample is secured in this position by a Teflon cap (G), which is screwed onto the nozzle. A Teflon gasket is provided between the sample and the cap to prevent leakage of the polishing solution around the sample. Failure to seal the sample results in chemical attack on the device side, i.e., on the side of interest.

Chemical polishing allows the handling of substrate samples with different electrical resistivities and does not require electrical contact, which is difficult to achieve in some device geometries. Materials used in the apparatus are resistant to hydrofluoric acid, which is used in the polishing solution. Safety in operation was realized by completely enclosing the samples in a liquid-tight bath. Use of Teflon also prevents undercutting by the chemical polishing solution.

Three sapphire windows, one at the bottom of the Teflon container, another in the cap and the third at the top of the syringe, are provided so that the sample can be viewed against a source of light placed below the bath. Thinning is stopped before perforation by monitoring transmitted light with a photomultiplier tube or by observing the color of the sample. When the color becomes crimson, the sample thickness has been reduced to a few micrometers and is suitable for transmission electron microscopy at 1 MeV.

Experimental procedure

Silicon samples were ultrasonically machined from device wafers into 0.3175-cm (0.125-in.) diameter disks for use on JEOL-JEM 200A and 1-MeV electron microscopes. Each sample is placed on the tip of the nozzle with the substrate side facing the polishing solution (Fig. 2) and secured and sealed by the screw-on cap. The assembled Teflon syringe is inserted through the hole in the lid until the cap is seated on the bottom of the acid bath. Then a mixture of hydrofluoric and nitric acid is pumped through the system.

When the jet strikes the sample, the fluid breaks into a small circular region of uniform flow beyond which the pressure of the liquid decreases rapidly. It is in this region of uniform flow that polishing takes place uniformly. The main parameters governing the area of uniform

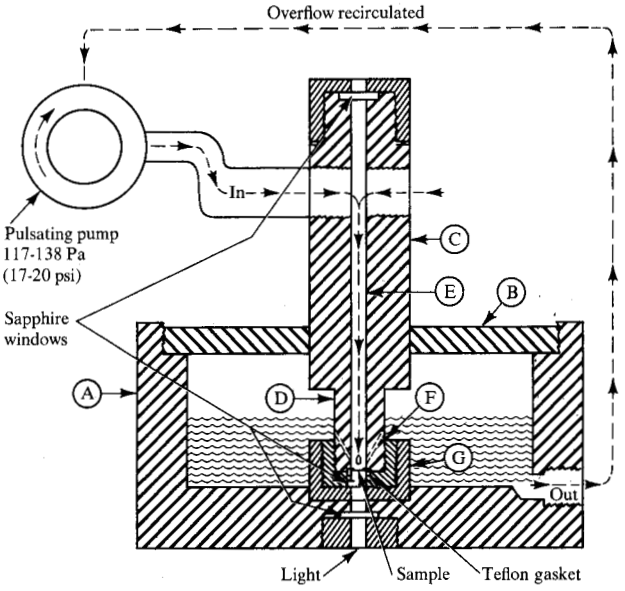


Figure 1 Schematic view of the jet apparatus.

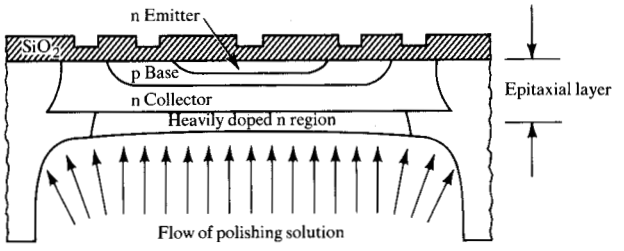


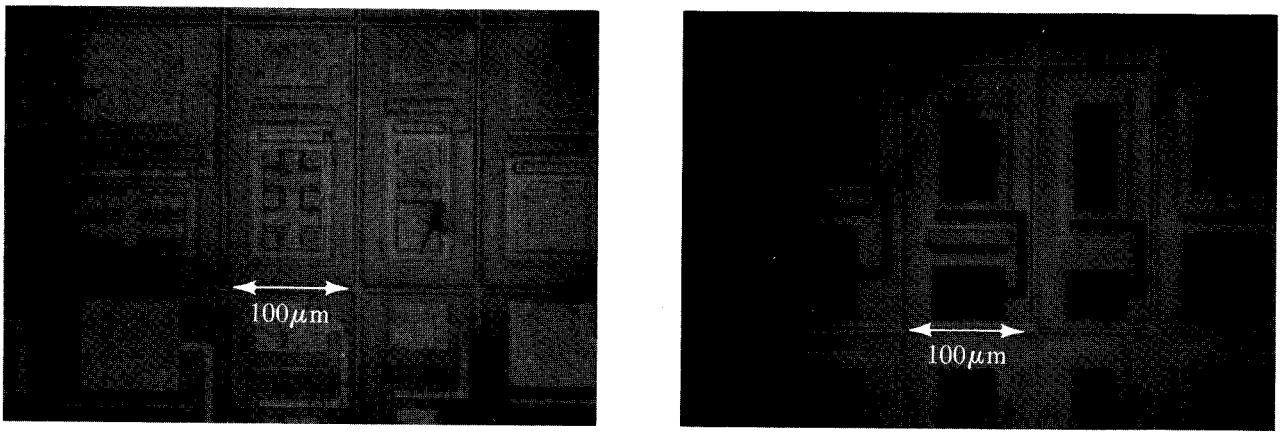
Figure 2 Cross section of typical bipolar sample and chemical jet flow.

polishing are a) the jet pressure and b) the specimen-to-jet distance. However, care should be exercised in choosing the number, diameter, and angle of the orifices through which the excess electrolyte should escape and the distance of the orifices from the sample.

Optimal adjustments of all of these parameters produce uniform flow resulting in uniform polishing. While the exact interdependence of all of these parameters was not determined, the number and diameter of the orifices for escape of excess fluid should be such that a positive pressure is maintained in the fluid between the specimen and the orifices. Otherwise stagnant liquid accumulates in that region and no polishing can be achieved.

Results and discussion

The extent of the transparent area in silicon devices obtained by this technique is illustrated in Fig. 3. Figures 3(a) and 3(b) are transmission optical micrographs viewed from the device and substrate sides, respective-



(a) (b)

Figure 3 Transmission optical micrographs of sample area thinned by chemical jet as viewed by (a) device side; (b) substrate side. Micrographs show aluminum patterns not indicated in Fig. 2. The uniformly thinned area suitable for transmission electron microscopy is about 500 μm × 400 μm.

ly. The uniformly thinned area (500 μm × 400 μm) containing isolation, base, collector, and emitter regions is shown. The diameter of the transparent region exceeded 750 μm. In the reflection optical micrograph, Fig. 4, the uniform removal of the substrate is illustrated. Samples of this thickness are transparent to 1-MeV electrons, Fig. 5. Note the extent and uniformity of the transparent area, in which dislocation loops are seen in the emitter as well as in the base regions. For examination at lower voltages, e.g., 200 keV, samples are further thinned by an ion-milling technique which can maintain uniformity.

Figure 4 Reflection optical micrograph of substrate side of sample shown in Fig. 3.

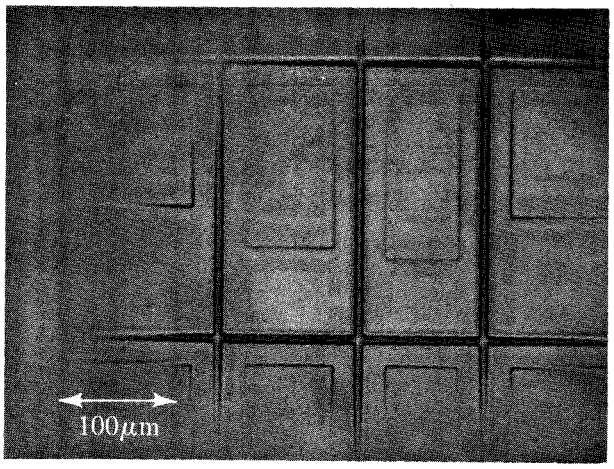
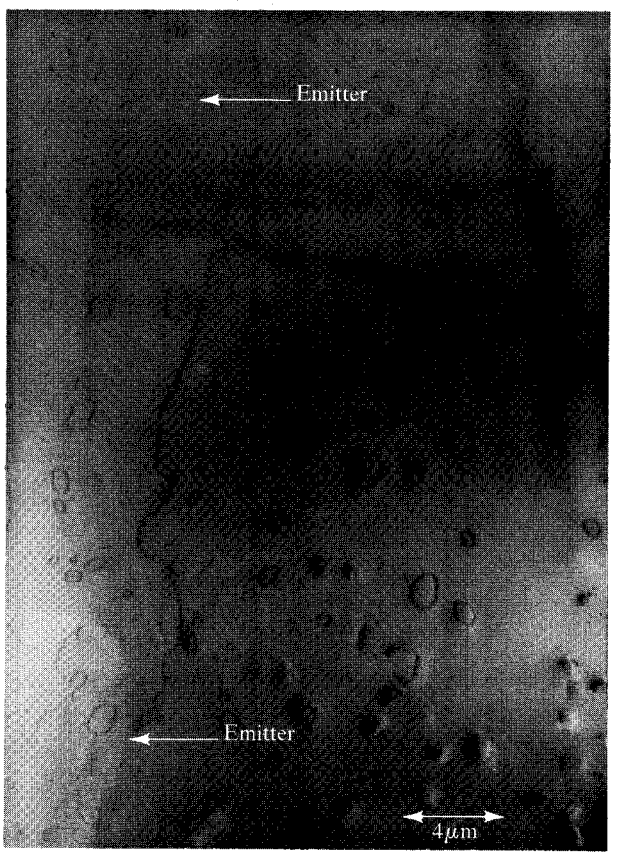


Figure 5 One-MeV transmission electron micrograph showing prismatic dislocation loops and helical dislocations in the emitter and base regions. Aluminum patterns were removed with HF.



The proportions of the chemical polishing mixture and the rate of pulsation for optimal polishing are sensitive to the resistivity and the type of silicon substrate. For Czochralski-grown p substrates (10 ohm-cm), a 9:1 mixture of nitric and hydrofluoric acids, a 30-to-40 cycles-per-minute pulsing rate, and a pressure of 117 to 138 Pa produced the best results. Jet pressure is limited by the crack resistance of the thinned sample. For 0.381-mm-thick substrates, five minutes are required for the sample to become crimson in color in the thinned area. The jet is then stopped and the sample is recovered, washed in 200-proof ethyl alcohol, and air-dried after excess solvent has been removed with absorbent paper. In conventional jet techniques, the specimen is physically separated from the jet and, consequently, uniform flow regions in the sample are very limited. A decrease in specimen-to-jet distance or an increase in jet pressure can expand the uniform flow region, but the dissolution kinetics of silicon are such that, under these conditions, etching rather than polishing would take place. When the sample is enclosed and the chemical polishing solution hits the sample directly, optimal jet pressure for uniform thinning can be obtained.

Acknowledgment

The authors thank Manfred Wurster for his technical assistance.

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Received June 13, 1973

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