The Art of Tungsten Etching in Semiconductor Chips

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In the course of both physical and failure analysis of semiconductor chips (*i.e.*, verifying what you actually deposited as a layer, vs. what caused the circuit to fail), it is essential to have appropriate deprocessing tools at your disposal in order to evaluate complex semiconductor structures. Deprocessing techniques are developed for each product manufactured and involve multi-step procedures that reveal the layer-by-layer secrets of the chip. These techniques require constant tweaking in duration and procedure as the manufacturing process imposes changes and as the architecture of the semiconductor changes. While there are many tools that assist in these analytical pursuits, such as RIE (reactive ion etching - a dry etching technique), ion milling, and microcleaving, the wet chemical etching of tungsten is sometimes more reproducible than RIE techniques.

Tungsten is a popular metal deposited both for interconnections and circuitry in MOS (Metal On Silicon) devices. Lining materials are used to bridge metal-to-silicon, or to prevent interactions between the metal and doped silicon substrate. Refractory interconnect liners may include MoSi or WSi, while barrier materials include TiW and TiN. In some instances, foreign material (FM) introduced during the manufacturing process corrupts the barrier or liner materials. The FM can form an unwanted connection (a short) or an unwanted insulation (an open). After a chip is electrically tested, failing signatures often point the way to the defect source that caused the circuit to fail. As an example, if the electrical test signature indicated a defect below the tungsten layer, it would be desirable to remove only the tungsten while maintaining the liner integrity to evaluate the source of the defect. In other instances, a highlight etch is desirable to slightly recess the tungsten and define the lining layer. In both cases, some surface (either top-down or cross-sectional) of the tungsten must be exposed prior to tungsten etching.

Etchant Descriptions

Two fairly quick and easy wet etches that will remove the tungsten but leave most liners intact are: 1) sodium hypochlorite (NaOCl) and 2) a 1:1 ratio of ammonium hydroxide (NH_4OH) and hydrogen peroxide (H_2O_2). The etchants are as follows, and based on ending up with a soluble product, the chemical reactions are provided for each of these etchants.

1) Use household bleach (5.25% sodium hypochlorite). A reagent grade is also fine, but usually not worth the expense. Heat about 50 to 100 mL in a Pyrex beaker to 60° C. This etch can be used for 6 hours without significant degradation, but should be disposed and replenished after that point.

The resulting likely reaction yields tungsten dioxydichloride (WO_2Cl_2) as a solubility product:

$$3NaClO + W + H_20 \rightarrow WO_2Cl_2 + NaCl + 2NaOH$$

2) Mix a 1:1 solution of reagent grade NH_4OH to H_20_2 (use 35% H_2O_2) at room temperature in a Nalgene, glass or teflon beaker. Use within 2 hours of mixing for the best reliability, as the H_20_2 will decompose. The resulting likely reaction yields ammonium paratungstate ($(NH_4)_6W_70_{24}$•6H20) as a solubility product:

$$7W + 21H_20_2 + 6NH_4OH \rightarrow (NK_4)_6W_70_{24}•6H_20 + 18H_20$$

Stainless steel or chemical resistant tweezers are fine. Position the chip on edge and leaning against the beaker to assist with removal of the chip from the beaker. Ensure the chip is large enough to grab with tweezers without damaging the defective area. The tungsten will fizz while under attack. Experiment with 2 to 10 minutes depending on tungsten thickness for removal, or only 5 to 15 seconds for a highlight etch. After etching, dip and wiggle chip in a clean beaker of deionized (DI) water, then rinse in a stream of DI water for several seconds. Still holding the chip, blow-dry with dry N_2. Ultrasonic cleaning is fine if your structure can stand it, but is usually not required.

Optimization Tips:

To pinpoint the "perfect" etch time, use incremental times with these etches and check with a SEM to determine the depth of the etch. Try to use approximately the same chip size and deprocessed state to maximize the reproducibility of the etch. In delicate circumstances, the amount of tungsten available to be consumed around the defect area can affect the etch time.

To speed the development of this procedure, use low voltage SEM (*i.e.*, 2 KeV, 500 µA - preferably FESEM) for uncoated top-down and simple cleaved section evaluation. Always use a fresh sample while optimizing the time for these procedures. Even at low voltages, a sample from the SEM can be coated with beam deposited carbon, back streamed oils, and contaminants within the chamber, which will significantly change both the etching time and quality if this type of sample is re-etched. While ashing with an oxygen plasma can assist in removing SEM generated depositions, the results are not optimal. Even so, ashing and re-etching a sample is sometimes required if there is only one sample. Once etching is complete, perform the SEM evaluation as quickly as possible, as a freshly etched surface is both susceptible to continued etching and to environmental attack. While optical microscopy will show color changes as unlayering occurs, and confocal microscopy is useful to evaluate removal depth, SEM provides the most universal information during deprocessing.

Wet vs. Dry Etching

There are several reasons why deprocessing or even highlighting is considered an "art" rather than a "science". Metal deposition technique, profile, density, architecture, and microstructure will all play significant roles in the way the metal will etch. Etch times and formulas that work today are not guaranteed to work on the same product made tomorrow. In the case of tungsten interconnects, a seam or change in metal density or microstructure in the center of the interconnect

creates a path of least resistance (and hence of faster etch rate) than the perimeter metal. The core of the interconnect etches out first, leaving a progressively thinner metal shell lining the interconnect perimeter, and undesirable etching of underlying tungsten lines may ensue prior to clearing the interconnect.

Why choose a wet etch technique over a Reactive Ion Etch (RIE)? In theory, a RIE technique should be less sensitive to tungsten deposition flaws than wet etching. RIE can be optimized for etching with differing aspect ratios (either isotropic or anisotropic etch). The truth behind the theory is that completely uniform tungsten removal by RIE is a delicate operation at best. Gas flows, ratios, pressures, and times are all variables that require successive testing to begin the optimization process. Also, changes in the metal pattern (dense vs. less dense areas) have a more profound effect on a RIE technique than on chemical etching in most instances. Surrounding structures can shadow a RIE process, and the platen of a RIE chamber often has a "etch pattern", *i.e.*, the RIE etch rate can be directional and even angular depending on placement within the chamber.

The loading of a RIE chamber also affects the removal rate, as a full eight inch wafer will etch at a much slower rate than will a few one centimeter chips loaded into a chamber. This is the "loading factor", which refers to the surface area of material to be etched. As the gas flow, time, and power remain constant, the amount of consumed surface material is limited by the amount of reactive gasses present. A full 8" wafer will not etch the same way as only a few chips cut from that wafer will *(i.e.*, you can't empty the Nile with a pair of cupped hands, but you can empty a sink).

Unlike a wet etch, discreet chips will always have an etch gradient from the edge towards the center of the chip even in the most carefully monitored RIE systems. Also unlike wet etching, the RIE chamber is plagued by the buildup of reacted components and absorbed gasses on the interior chamber walls, especially if the tool is not dedicated only for tungsten etching. These buildups require cleaning and chamber "conditioning" prior to uniform etching. While these are all considerations that effect the RIE tool, careful evaluation of the etch characteristics within the chamber and analysis of RIE etch results can yield a technique that is, in some cases, preferable over wet etching.

While both wet and RIE etching techniques can provide successful results and dramatically enhanced analysis capabilities, neither technique provides optimal "off the shelf" results.

Choosing the "correct" method to deprocess or highlight tungsten is not always straightforward, hence the art: an iterative process of testing techniques, time, and results to achieve the optimized process. ■

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