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ENGR 241: Nanofabrication Laboratory Survey Paper

Pre-project Information

Before beginning the project, it would have been useful to have scheduled training time in the SNF on standard pieces of equipment before the quarter began so that the core training sessions could be completed in the first week or two, rather than by week four. The core training sessions for basic SNF training would be the lab safety tour, the "All-Litho" training, the SVG coater and developer tracks, and the wetbenches: *lithosolv, wbclean, wbflexsolv,* and *wbflexcorr*. Ideally, course participants could be automatically signed up for SNF membership and scheduled for core tools automatically by class administrators.

Additional information that could have guided the project was the availability of Scanning Acoustical Microscopes (SAM) to attempt to view bonded samples at a high resolution without breaking them apart. X-ray Computed Tomography (XCT) was attempted, but the resolution of the machine was barely enough to see that patterns existed in the bonded sample. SAM may have provided a method to view and quantify how much of a sample was bonded based on the relative densities of polysilicon, nickel, and nickel silicide without having to guess from dicing the samples. Any techniques that could have provided more quantitative bonding analysis methods would have been valuable to this project, and knowledge of the double cantilever beam tester earlier on would have lended much more insight into the directions to take the experiments.

Project Selection and Methods Rationale

We chose nickel silicide as the bonding materials system because Eric's research project direction is on MEMS-fabricated thermionic converters. Thermionic converters are ultra-high temperature electron heat engines that operate in vacuum environment. The vacuum seal for a thermionic converter requires the following unique properties: (1) it should be fabricated in conventional MEMS processing equipment but should be able to withstand elevated temperatures (+600°C); (2) it should be conductive, allowing the seal to also serve as a vertical electrical interconnect; and (3) it must withstand prolonged exposure to Cs vapor, which is extremely corrosive. Wafer bonding by nickel silicidation to form a vacuum seal potentially satisfies all of these requirements.

For bonding in SNF, there are two choices, the evbond and the flip-chip. We chose the evbond for the following reasons: (1) the evbond can perform bonding under vacuum, which is necessary in order to fabricate a vacuum package by wafer bonding; and (2) the evbond can apply higher temperatures and forces than the flip-chip, which we thought might be necessary for forming the silicide. One of the consequences of using the evbond is that we had to use full wafers rather than just pieces, as it is far easier to handle wafers in the evbond than it is pieces. Precision alignment for pieces is also not possible with the evbond.

Liftoff used in place of etching because it was more straightforward for us to plan and execute. While nickel etching has been performed in the SNF in the past, the nickel etchant is not a standard chemical that is kept in stock.

We used evaporated nickel films rather than sputtered films for easier liftoff, as evaporation results in reduced sidewall deposition. We chose the AJA evaporator for doing these evaporations, because its loadlock allowed for reduced pumpdown times, and because it is the only evaporator in ExFab.

Process Summary

1. Clean new prime wafers in wbclean-res-piranha.
2. Fabricate the polysilicon wafers: LPCVD 1-2μm polysilicon, store wafers until needed.
3. Fabricate nickel wafers: pattern for liftoff with Heidelberg, evaporate Ti adhesion layer and Ni bonding layer in AJA, liftoff.
4. Prebond clean to strip off surface oxides: 5:1 water:29% ammonium hydroxide for 5 minutes for the Ni wafer, 50:1 HF for 1 minute for the polysilicon wafer. Do this immediately before bonding.
5. Bond in evbond using the steel plate (NOT quartz). Optimal process conditions are 450°C with as much force as possible (3340N maximum force). 40 minutes at temperature with force applied worked well

Essential Aspects and Lessons Learned

The prebond clean/oxide etch steps is crucial for bonding. This step must be performed immediately before bonding, to ensure that the surface oxides do not re-form before the wafers are bonded. Wafers that did not undergo this process did not bond at all… An additional suggestion was to oxide etch the Si wafers before deposition of the polysilicon layer by CVD - some surprising results observed during DCB testing may be a result of leaving this step out.

The best bonding occurred at 450°C with the maximum 3340 N force applied. For nickel silicide bonding in general, we suggest bonding at 450°C with the highest amount of force compatible with the rest of the process. Direct tool force is also a more critical variable than initially anticipated, due to the need for flattening the silicon wafer substrates after deposition.

Doing the liftoff step in acetone in the ultrasonic bath does not cleanly remove the layer - instead it tends to shred the metal layer into tiny particles and redeposit them on the bonding surface. These particles are nearly impossible to remove after-the-fact. This is very bad for bonding, as ultra-flat clean surfaces are needed to successfully bond. Instead, we suggest performing liftoff by soaking the wafer in acetone without agitation for a few hours.

Double Cantilever Beam testing is available in the Dauskardt lab for quantitatively evaluating bonding quality. Special samples need to be prepared in order to test with double cantilever beam, the designs for which are shown in our main paper. It would have been useful to prepare samples from other standard metal bonding methods such as gold-tin eutectic bonding to compare with the nickel silicide system.

Something weird happens to cobalt in acetone - sample turned brown, a guess is that this is cobalt carbonate. The main document we used for appropriate etchants listed only HF as a cobalt etchant and nothing was really listed for cobalt oxides or carbonates, but a late web search mentioned nitric acid as a possible oxide etchant.