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Summary: Stressed Ni films electroplating for controlled spalling technology

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Abstract:

There are several methods of fabricating thin films to fulfill the demand in thinner electronics. We are interested in a newer method called Controlled Spalling Technology (CST). CST is consist of two layers: substrate and stressor. Stressor requires a large intrinsic stress to initiate the process. We are using Nickel (Ni) for the material of the stressor layer because it has high fracture toughness. Electroplating is used to deposit this stressor layer on desired substrate. Then we characterize the stressor to measure its intrinsic stress and to know whether it is possible to apply CST.

Introduction:

As the semiconductor industry progress toward thinner device due to cost and efficiency, a number of methods have been developed to fabricate thin films, such as Eltran, ELO, and Smart-Cut. Nevertheless, these methods have drawbacks like the need of using sophisticated and expensive equipment, unwanted material damage due to ion bombardment, high temperature, or aggressive chemicals, limited options for removing partial or finished devices, and limited set of applicable materials.[1] Seeing the importance of the field yet with unreliable technology, we realize we need a better technology, and our proposed solution is Controlled Spalling Technology (CST). CST overcomes the weaknesses from other technologies because it can be done without expensive equipment; it doesn't cause substrate modification; it can be applied at nearly any point during device fabrication; and it is applicable essentially to any brittle substrate.

We are specifically interested in applying this technology on Gallium Nitride (GaN) because of its importance and usefulness in our life. GaN has two main applications: blue light emitting diode (LED) and high-electron mobility transistors (HEMT).

CST is based on a fracture mode named spalling, it is defined by a planar fracture that runs parallel to the material surface that can be initiated by a sufficiently high tensile stress in an applied surface film in a process. We need two layers to do spalling (Figure 1): substrate and stressor. Stressor is

the top layer we deposit to apply tensile stress, we will use Nickel (Ni) because it is low-cost, easy
to deposit and has high fracture toughness. [1] Relying on the deposited stress on the stressor, we can peel a thin layer off the substrate, which is our final goal for this project.

Outline of original research project:

This project was carried out during Summer 2018. To do spalling, we need to apply a Ni stressor layer at the wafer and we will use electroplating to do it. However, before electroplating, we need to deposit Ni seed layer using an evaporator in Micro and Nanotechnology Laboratory (MNTL) cleanroom. I will specifically use CHA evaporator and deposit Titanium (Ti) / Nickel (Ni) as adhesion and seed layers. I already gained MNTL cleanroom access from my Summer 2017 work and I will have training to use this machine alone. After the electroplating, I will carefully handle the wafer and apply tape on it to spall it. Finally, I want to know the quality of

Figure 1 CST diagram [1]

our film, therefore I will use characterization equipment such as scanning electron microscope (SEM) in Frederick Seitz Materials Research Laboratory (MRL).

Results and discussions:

At the start of the summer, we did not have a consistent seed layer production and electroplating. So we used Silicon (Si) wafers to perfect our technique and procedure before actually using GaN. Our first accomplishment was to have a consistent seeded wafers production.

We stopped using e-beam at MNTL and use the one on MRL instead. In addition to that, we also made a taping template to attach our two inch wafer to the four inch wafer holder so we could load and unload the wafers safely while preserving their cleanliness.

Our next accomplishment was to have a consistent electroplating. Most of our electroplating failed because of adhesion problem. Our electroplating setup was not waterproof so there was Ni plated under the metal contact. So we made a completely new electroplating setup based on two o-rings.

Figure 2 An example of failed electroplating. There were delamination on the edges and the discoloration on the Ni layer

Figure 3 The new electroplating setup. On the right is the part holding the sample. The inner o-ring touches the sample and with the outer o-ring they made a waterproof seal. On the left is the complete electroplating setup. The clamps secure the o-ring seal.

Once we made sure that the setup was waterproof, we got successful electroplating with some failure outside the plating area. We hypothesized that the failure was not an adhesion problem between the seeded Ni and electroplated Ni

layer but between the Si wafer and seeded Ti. There's no feasible way to quantify the adhesion between each layer we deposited. We decided to try to clean the wafer before e-beam with RCA cleaning and piranha to see which works. RCA is a set of cleaning steps, it cleans both organic and metallic contaminant. BOE cleaning is supplemental to RCA and served to clean the oxide layer. We did not use BOE because factory produced oxide is good. Piranha is a chemical mixture of sulfuric acid and hydrogen peroxide used to clean organic residues on wafers. Piranha worked better and faster.

Lastly, to improve the final electroplating result, we investigated the best drying method after the electroplating. Without proper drying, the leftover electroplating solution residue damaged the surface quality. We tried many methods such as baking, rinsing or dipping with chemicals such as acetone and isopropyl alcohol. We learned that deionized (DI) water rinse works the best.

With all these improvements we made two, four, six, eight minutes electroplating sample.

We used film stress measurement (FSM) to measure the radius of curvature and profilometer to measure the thickness.

Figure 4 Electroplating sample without any pre-cleaning. The electroplating area had good adhesion, but there was delamination on the outer part

Figure 5 DI water rinsed sample, the Ni surface looked better than any sample before

Electroplating Time (mins)	FSM Radius Measured (m)	Ni Thickness (µm)	Calculated stress (MPa)
2	3.21	2.34	554
4	2.46	4.48	377
6	1.89	6.48	340
8	2.21	9.27	209

Table 1: Using Stoney equation we can calculate the stress from measured radius and thickness.

We also tried to confirm the FSM curvature measurement using optical microscope and we got 1.74 m for two minutes sample and 1.26 m for six minutes sample. The calculated stress was not high enough to apply CST.

I looked the successful samples under SEM:

Figure 6 SEM on four minutes sample (left) and eight minute(right).

The grain size is increasing with thickness (electroplating time), while the number density of the grain boundary decreases with thickness.

Th expansion at the grain boundary can be
responsible for the stress formation.
As a comparison I also looked discolored
sample under SEM. This was a two minutes
sample without DI rinse. The grain size looked
smaller than figure 6 as expected. There are holes everywhere because the surface was not cleaned
properly. During the SEM, I couldn't find a location without single hole.

Figure 7 SEM on a two minute sample without DI rinse

While perfecting our technique, we observed that, pH of the solution decrease over plating.
O-rings left some residue on the wafer which caused electroplating failure, we learned that
different materials left different amount of residue. Viton and fluorosilicone left less residue than
bunan-n. This problem was solved by taping the o-ring or putting sharpie on it before touching the
wafer.

Conclusion and future directions:

We have successfully demonstrated stressed Ni electroplating on two inch Si wafers.
Piranha pre-cleaning and DI water rinsed worked best to support our electroplating process. The
stress decrease as the thickness or electroplating time increase. We will keep trying to increase the
stress to achieve CST desired stress.

Reference:

[1] S. W. Bedell, K. Fogel, P. Lauro, D. Shahrjerdi, J. A. Ott, and D. Sadana, "Layer transfer
by controlled spalling," J. Phys. D. Appl. Phys., 2013.