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Diffusion Barrier Research: Thin Film Growth and Analysis

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As technology advances, the computer industry is compelled to design faster computers. Increasing the speed of their products involves decreasing the size of the transistors within the computer chips in order to increase the number of transistors on each chip, which increases device speed. However, there is a limit to how small transistors can be made, partly because the time required to transmit signals between discrete transistors has become a limiting factor in shrinking the transistor size. This is overcome by changing the insulating dielectric material to one with a low dielectric constant ($\kappa = 2.3-2.6$) and using low resistivity copper for signal transmission. Therefore, the metal interconnects that link transistors together are currently made of copper (Cu), but since they tend to diffuse into the silicon (Si) transistors, it is necessary to create a diffusion barrier between layers of Cu and low- κ dielectric within the device. An ideal barrier should prevent diffusion between the device layers that it is intended to separate, be non-reactive with neighboring device components, be deposited at a relatively low temperature to prevent damage to sensitive circuit components, and be sufficiently uniform to protect all small device features. As device features decrease in size, the materials that compose the diffusion barriers currently may not be able to prevent diffusion from occurring when made much thinner. Therefore, it is necessary to find suitable materials to replace the current diffusion barrier materials by analyzing potential replacements.

My work in the lab this summer included experimentation on potential diffusion barrier materials that contained ruthenium. While learning about the purpose of this experimentation, I

also learned how to grow films using the chemical vapor deposition (CVD) chamber and I cut 10 nm SiO₂/Si squares for use inside the chamber. I also took data using a surface science chamber, which involved the use of the x-ray photoelectron spectroscopy (XPS) and temperature programmed desorption (TPD) processes.

Analysis of materials using the surface science chamber involves the use of an ultra-high vacuum (UHV) chamber that is connected to a cryogenic pump and a diffusion pump to achieve UHV. The system also includes a sample manipulator, which allows the substrate to be moved in all directions and rotated to face different positions for dosing and analysis. The system also uses liquid nitrogen sample cooling and resistive heating, as well as a computer and software, which are used to take data.

I learned how to perform two tasks using the surface science chamber: XPS and TPD. XPS involves sending x-ray photons toward the sample to knock electrons from the atoms near the surface out of their shells due to the photoelectric effect. The electrons will be emitted at certain energy levels based on what element they came from, since different elements have different energy levels at which they hold their electrons. The energy level that is measured by XPS is the binding energy of the orbitals from which the electrons are emitted, and analysis of which of these binding energies is detected can determine what elements are present in the very surface (0.5-1 nm) of the substrate.

TPD involves dosing the sample with precursor molecules and then increasing the temperature of the sample at a certain rate (approximately 7 K/s) to observe at what temperature the adsorbed molecules desorb from the surface. This method also determines whether these molecules desorb from the surface intact or break into fragments.

The process that I used to take data involved first using XPS to scan the surface of the

substrate to make sure that it was relatively free of contaminants. In the next step of the process, the substrate is first moved into the dosing position in order to deposit the precursor, and the substrate is held at a specified temperature (140 K). The precursor is then released into the chamber for a set amount of time. This ensures that a set amount of precursor enters the chamber. After this, XPS is used a second time to identify the different elements that are present in the precursor, the ratio of those elements in relation to each other, and how those elements are bonded to each other, if at all. TPD is then used to see when the molecules that adsorbed onto the substrate desorb. Finally, XPS is used again to determine how much precursor remains on the surface after TPD. The data taken from this entire process describe how the precursor may adsorb onto or react with neighboring materials, at what temperature it may desorb from the surface, and provides an idea of whether the precursor could be used in CVD to deposit a film.

The process of CVD involves the use of a horizontal tube furnace, which has an interface that allows the user to set the temperature of the furnace. Connected to the chamber are gas lines that allow the precursor to be carried into the tube furnace by the flow of argon. Argon flow is controlled by a mass flow controller, and the pressure within the system is regulated by a mechanical pump connected to the gas lines. Heating tape controls the heating of the precursor saturator and the gas lines.

To begin film growth within the CVD chamber, the substrate (a square of SiO_2/Si) is placed within a sample boat and slid into the tube so that it is at a set position. The furnace is set to a certain temperature to allow film growth, and the pressure of the system is maintained at 0.300 Torr. The saturator containing the precursor is heated to a certain temperature to allow it to be transported to the furnace, and the gas lines are set to a temperature 10 °C higher than the precursor chamber. After being heated, precursor is transported to the furnace by argon gas, and then adsorbs

onto the surface of the substrate. Reactions may occur within the chamber, and products may desorb from the surface of the substrate, or remain adsorbed to the surface. After a set growth period, the sample is allowed to cool before removal from the system for analysis.

Analysis of CVD samples can conclude whether the precursor can be used to grow a film at the set temperature and whether the precursor and any reaction products adsorbed onto the surface of the substrate. The sample will also show how uniformly the film was grown. This could lead to conclusions about whether the precursor used to grow the film could be used as a diffusion barrier in computer chips.

Although I was not sure what to expect from thin film research, the time that I spent in the lab was not quite what I had imagined it to be. Lab safety was not as strict as I had expected, but since the way the lab was set up limited contact with the more dangerous chemicals in the lab, closed-toed shoes and safety goggles were generally unnecessary. The equipment was also not what I had expected; most of the machines I worked with were at least a decade old and didn't appear as high-tech as I thought they would. It was also somewhat surprising to discover that the surface science chamber looked quite a lot like an alien satellite covered in aluminum foil, which was there to keep heat inside the chamber. The lab equipment was also somewhat faulty at times due to various reasons; one of the pumps on the surface science chamber, for example, would shut off the power to adjacent labs in addition to our own if it overheated, instead of shutting only itself off. Running experiments also took a lot of time, which I expected, but there was a lot of waiting involved – there were usually no other tasks to complete while experiments were running. Despite the inconveniences, the equipment generally worked properly and although the actual tasks were somewhat uninteresting, I found the concept behind the work intriguing. The lab results were also interesting, but I think they would have been even more so if I had a better knowledge of what they

meant about the compounds in question. Overall, I think that working in a research lab was a valuable experience to me, and my naïveté regarding the field did not hinder me from coming away from the experience with a little more knowledge about an intriguing and very specific subject.