SCIENCE OF THIN FILMS

"Rainbow Wafer"
[Courtesy of MJ Willis, personal collection.]

Activity Overview

In this activity you will interpret graphs and charts related to silicon dioxide (SiO_2) thickness on a silicon wafer. Silicon dioxide is a thin film commonly used when fabricating microsystem devices.

Given a rainbow wafer, you will estimate the thickness of several layers of SiO_2, then calculate the <u>etch rate</u> of each layer based on its thickness and time of etch. You will also interpret graphs related to oxide growth and temperature.

This activity will help you to better understand the basics of oxidation, the properties of thin films, and etch rates as they apply to the isotropic wet etch of silicon dioxide (SiO_2).

Objectives

- Interpret Oxide thickness vs. temperature graphs.
- Using a color chart, correctly estimate the thicknesses of several layers of silicon dioxide.
- Using your results, create two graphs showing the relationship between oxide thickness and time.

Oxidation

- Oxidation occurs when pure silicon (Si) is exposed to oxygen forming silicon dioxide (SiO_2).
- SiO_2 is referred to as “oxide”, but also quartz and silica.
- Native oxide is a very thin layer of SiO_2 (approximately 1.5 nm or 15 Å [*angstroms*]) that forms on the surface of a silicon wafer whenever the wafer is exposed to air under ambient conditions.
- Native oxide is a high-quality electrical insulator with high chemical stability making it very beneficial for microelectronics.

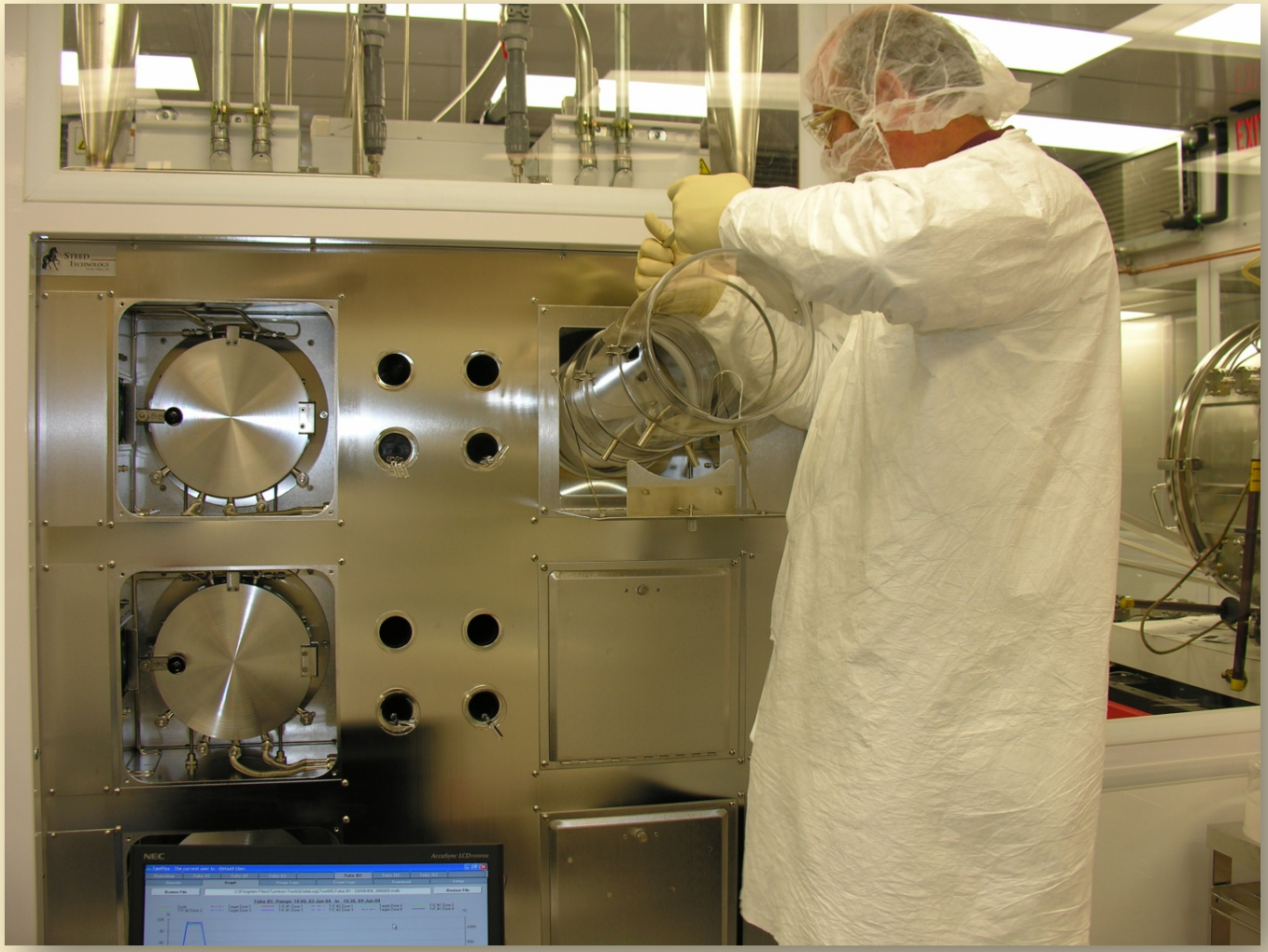
Applications of SiO_2 in MEMS Fabrication

- Sacrificial layer or scaffold
- Structural layer or material for devices such as beams or membranes
- Passivation coatings
- Hard mask (protects the silicon)
- Electrical isolation of semiconductor devices
- Diffusion mask (barrier material or mask during implant or diffusion processes)
- Gate dielectric and interlayer dielectric in multilevel metallization structures
- Key component in certain wafer bonding applications

Growing Silicon Dioxide (SiO_2)

- SiO_2 is grown on a pure crystalline silicon wafer in a diffusion furnace using high temperatures (~900 to 1200 C°).
- A diffusion furnace consists of a quartz tube large enough to hold several boats of wafers and able to heat to at least 1200° C.
- The wafers are placed in quartz boats. The boats are then placed on a platen (like a loading dock) which transports the boats into the furnace's quartz tube.
- The next slide shows the manual unloading of 100mm oxidized wafers.

Diffusion Furnace



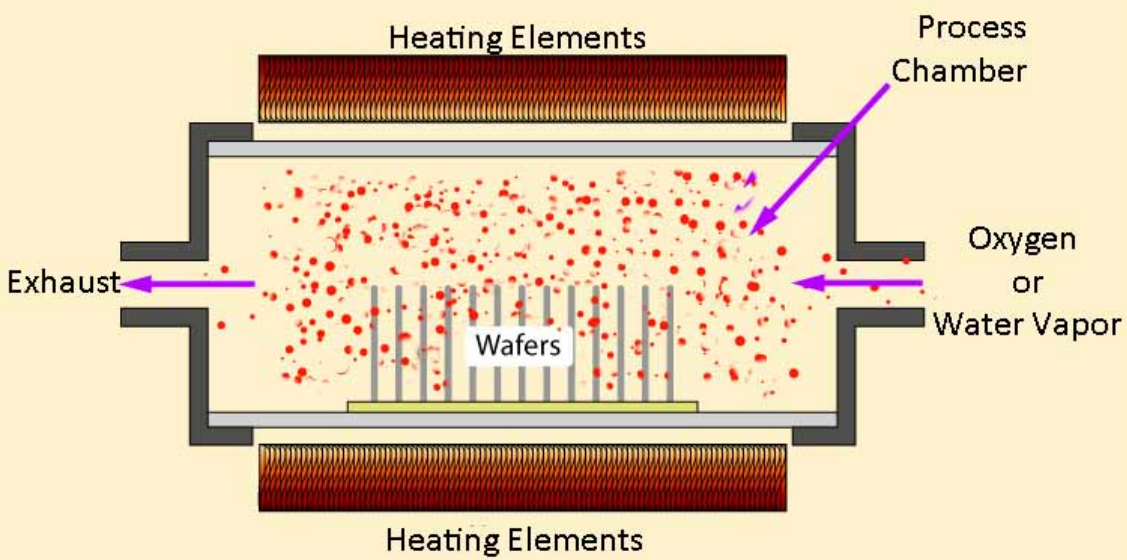
Oxidation furnace being manually unloaded.
[Image courtesy of the University of New Mexico, Manufacturing Training and Technology Center UNM/MTTC]

Growing Silicon Dioxide

- SiO_2 naturally grows on a silicon surface at room temperature. However, this growth is very slow and stops at about 15Å after only two to three days.
- In microsystems fabrication, SiO_2 is either deposited through a chemical vapor deposition process or grown in a high temperature furnace with an oxygen source (gas or vapor).
- This latter process is called thermal oxidation.

Thermal Oxidation Process

- The silicon wafers are placed in a heated furnace tube (typically 900 – 1200° C).
- A source of oxygen (gas or vapor) is pumped into the chamber. This source is either O_2 or H_2O, respectively.
- The oxygen molecules react with the silicon to form a silicon dioxide (SiO_2) layer in the substrate.



Schematic diagram of an oxidation furnace.

Thermal Oxidation Chemical Reactions

Dry oxidation using oxygen gas

$$Si\ (solid) + O_2\ (gas) \rightarrow SiO_2\ (solid)$$

Wet oxidation using water vapor or steam

$$Si\ (solid) + 2H_2O\ (vapor) \rightarrow SiO_2\ (solid) + 2H_2\ (gas)$$

Rust

- The oxygen/silicon reaction is analogous to the oxidation or rusting of metal.
- An oxygen/iron (Fe) reaction forms rust (Fe_2O_3).
- The oxidation rate is dependent on the environment (e.g., the presence or absence of water (H_2O) and the temperature).
- The longer the metal or wafers are exposed to the oxygen source (H_2O or O_2), the thicker the rust (or oxide) to a point.
- The higher the temperature, the faster the reaction rate and the thicker the oxide.

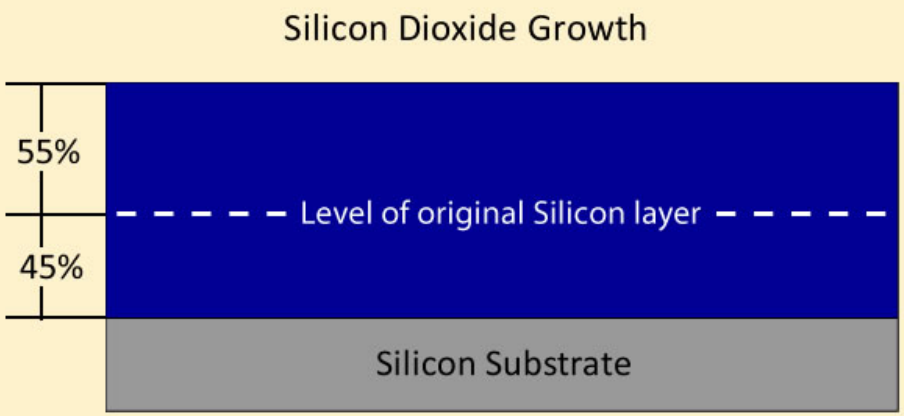
Oxide Growth Kinetics

Initially, the growth of silicon dioxide is a surface reaction only.

After the SiO_2 begins to grow on the silicon surface, new arriving oxygen molecules must diffuse through this SiO_2 layer to get to silicon atoms below the surface. At this point the SiO_2 growth is occurring at the silicon crystal – silicon dioxide interface.

As a general principle, the depth of pure silicon consumed in the oxidation process is 45% of the final oxide thickness.

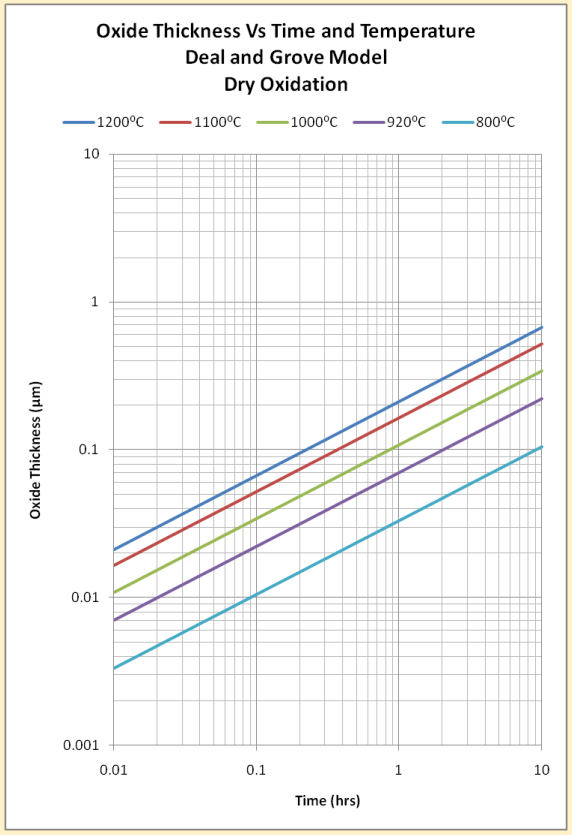
For every 1 micrometer of SiO_2 grown, about 0.46 micrometers of silicon is consumed.

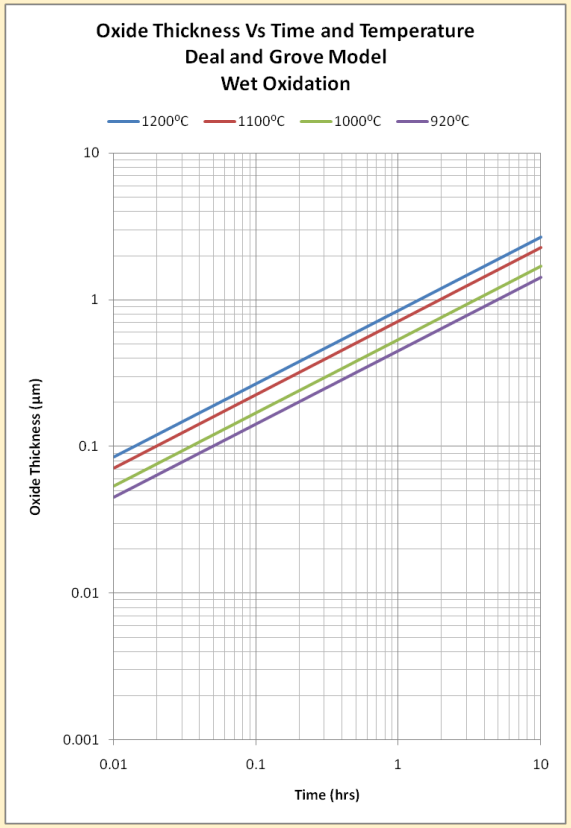


Cross-sectional view showing how silicon dioxide grows into the surface of the wafer surface.

Rate of Oxidation (oxide growth)

The rate of oxide growth is highly dependent upon temperature. Below are two graphs that demonstrate the growth rate of oxide relative to temperature in dry oxidation. process (*left graph*) and a wet oxidation process (*right graph).*





Activity I: Interpreting Graphs

Complete Activity I in your Participant Guide:

"Interpreting Oxide Growth and Temperature Graphs"

Etching Silicon Dioxide

Silicon dioxide is readily etched using hydrofluoric acid (HF) according to the following reaction:

$$SiO_2\ (solid) + 6HF\ (liquid) \text{-->} H_2SiF_6\ (liquid) + 2H_2O$$

Hydrofluoric acid (HF)

- HF is a weak acid meaning that it only partially dissociates in water.
- Because of the low value of hydrogen ion concentration [H^+] in weak acids (HF in our case), the pH is quite vulnerable to change.
- Changes in pH results in changes in etch rate.
- Small dilutions or consumption of the reactant during etching can significantly alter pH.
- These alterations can be limited by the technique of buffering the solution.

Buffered Oxide Etch

- The customary buffer for HF is ammonium fluoride (NH_4F).
- Ammonium fluoride is a salt that dissociates to form fluoride and ammonium ions.
- A typical volume ratio is 20 parts NH_4F to one part HF.
- This mixture is called buffered oxide etch (BOE).
- BOE is a reasonably selective etch for silicon dioxide.
- It will not etch bare silicon, but does attack silicon nitride and photoresist to some extent.

Oxide's Color

Oxide is colorless. However, when you look at an oxide wafer, it has color.

The color of the oxide coated wafer is caused by the interference of light reflecting off the silicon (below the oxide) and the light reflecting off the top of the oxide surface.

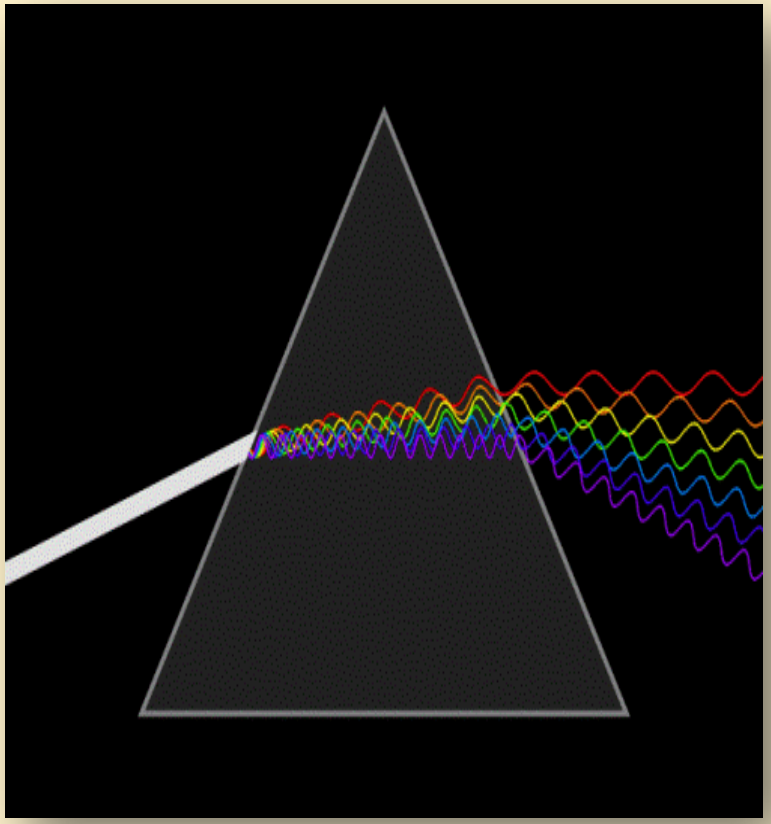
As the oxide thickness changes, so does the interference and the oxide's "seen" color.

Color charts have been developed that state the oxide's thickness based on its "seen" color. *(See the Oxide Thickness Color Chart in your activity.)*

Rainbow wafer showing 11 oxide thicknesses

Thin Film Interference

White light consists of all of the colors of the visible light spectrum. You can see this when you shine white light through a prism.

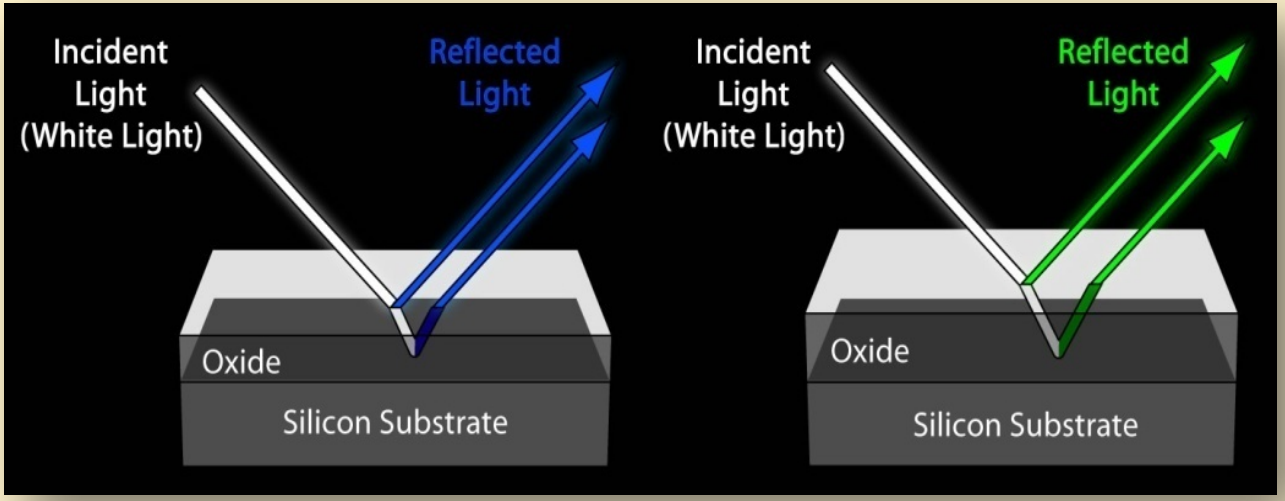


The dispersion of white light as it travels through a triangular prism.

[Illustration is Public Domain]

Thin Film Interference

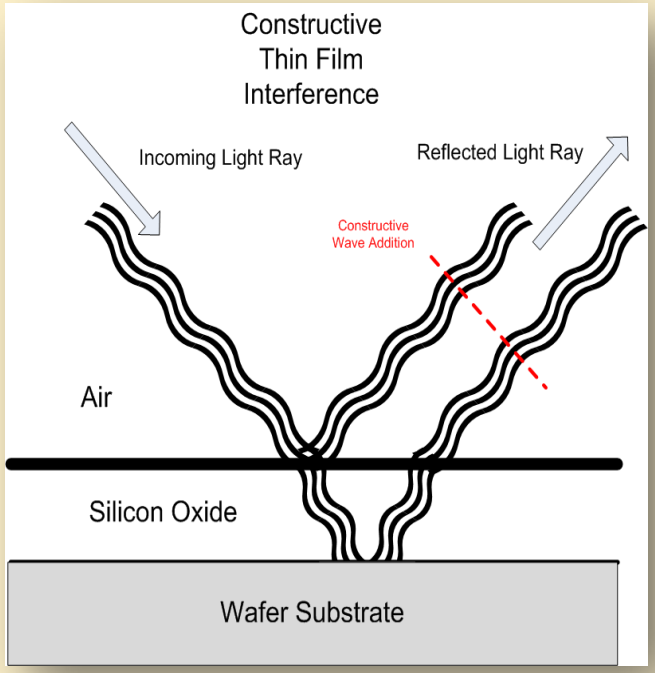
- Below are two wafers of two different oxides thicknesses reflecting different colors.
- The incident ray (white light) reflects off the lower substrate/oxide interface surfaces and the top air/oxide surfaces. These two reflected rays of light recombine.
- Depending on the oxide thickness, only certain colors will constructively recombine, while the other colors which make up the white light will not.

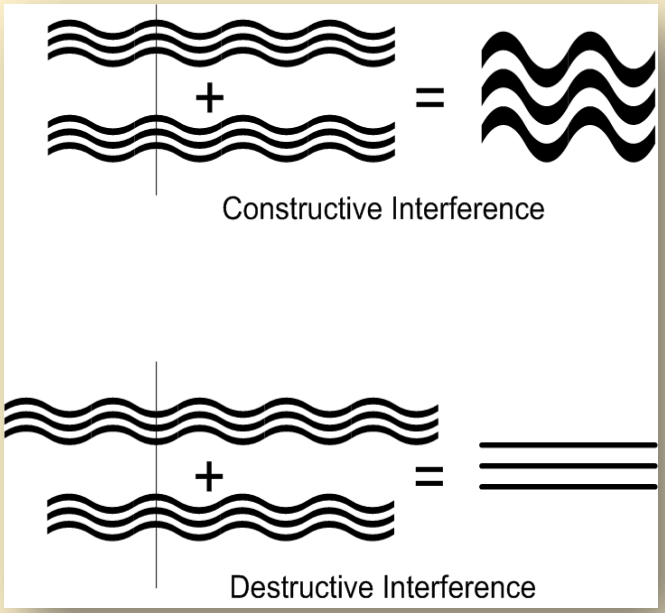


Constructive vs. Destructive Interference

When the light reflected off the substrate is in phase with the light reflected off the surface of the oxide, the resultant wave is the sum of the amplitudes. This is *constructive interference.*

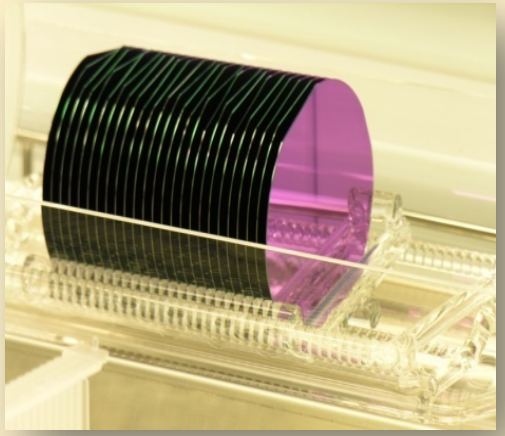
If the two reflected waves are out of phase, then their amplitudes cancel each other out. This is *destructive interference.*

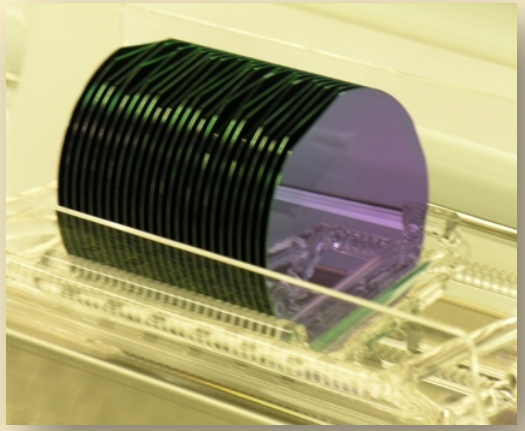


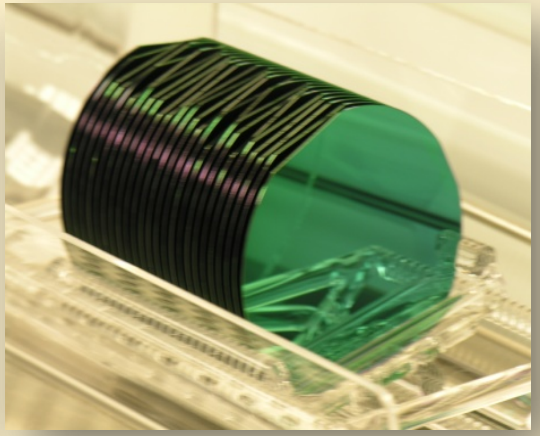


Color is Deceiving

- Tilt the wafer, the color changes.
- In one wafer, of a specific thickness, you will see different colors as you view the wafer at different angles (tilt).
- The color you see depends on the angle at which you view the wafer's surface.
- Following is a series of photographs taken of the same oxidized wafers, but at three different angles (all of these wafers have had approximately 5700 Å of oxide growth).





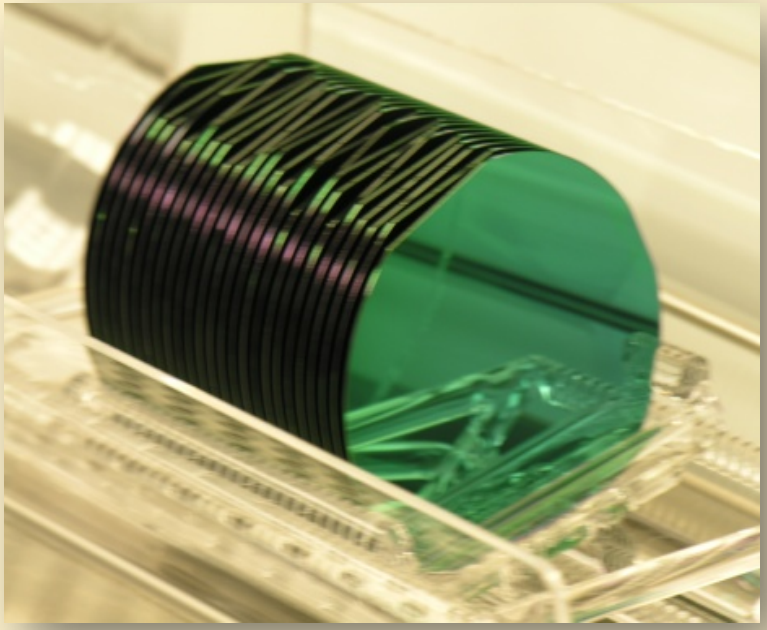


Color and Angle

- The color you see comes down to the thickness of the film that the light travels through before reaching your eyes; this is called the optical path length.
- If you look straight down (perpendicular to the surface), the light reflected off the bottom (SiO_2 and Si) will have traveled through two times the thickness of the film.
- If you look at the same film at an angle, the light will have traveled through more than twice the thickness of the film; the light has therefore traveled through a longer optical path length.
- Effectively a thicker film is being observed; hence, the color looks different.

Thickness vs. Angle of Sight

Based on the color chart at the end of the Science of Thin Films Activity, estimate the thickness of a “green” wafer?

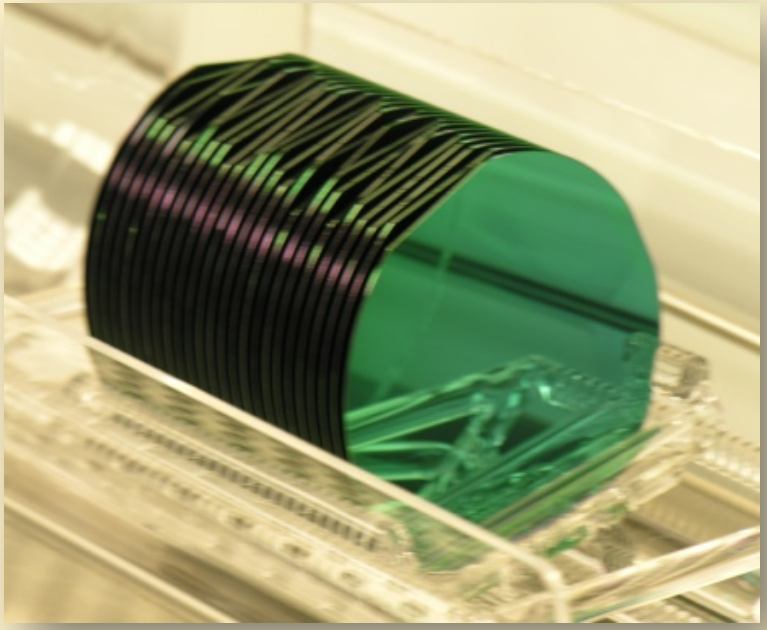


Thickness vs. Angle of Sight

Based on the color chart at the end of the Science of Thin Films Activity, estimate the thickness of a “green” wafer?

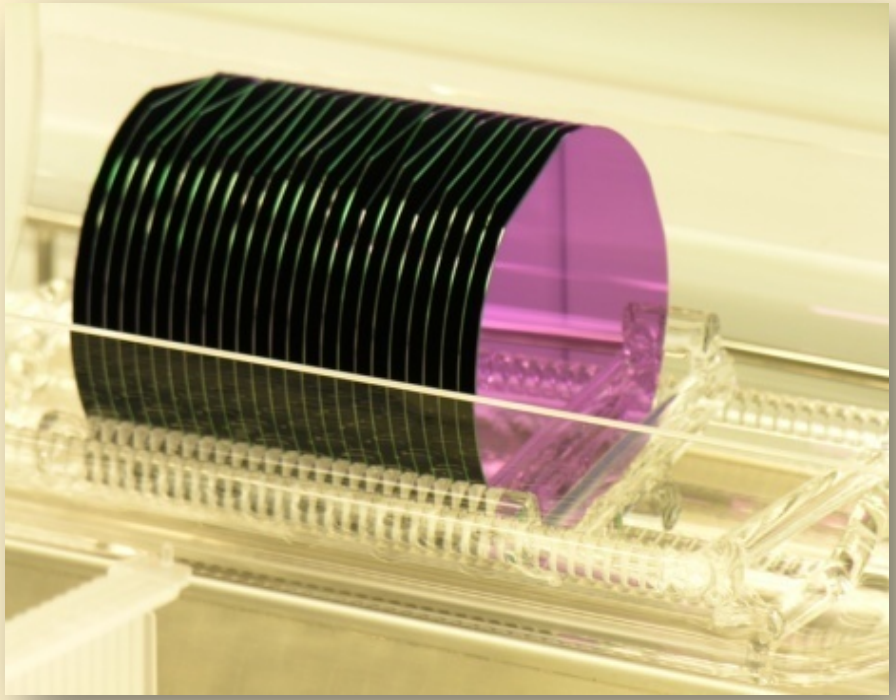
Answer:

5200 angstroms



Thickness vs. Angle of Sight

Based on the color chart at the end of the Science of Thin Films Activity, estimate the thickness of a “carnation pink” wafer?

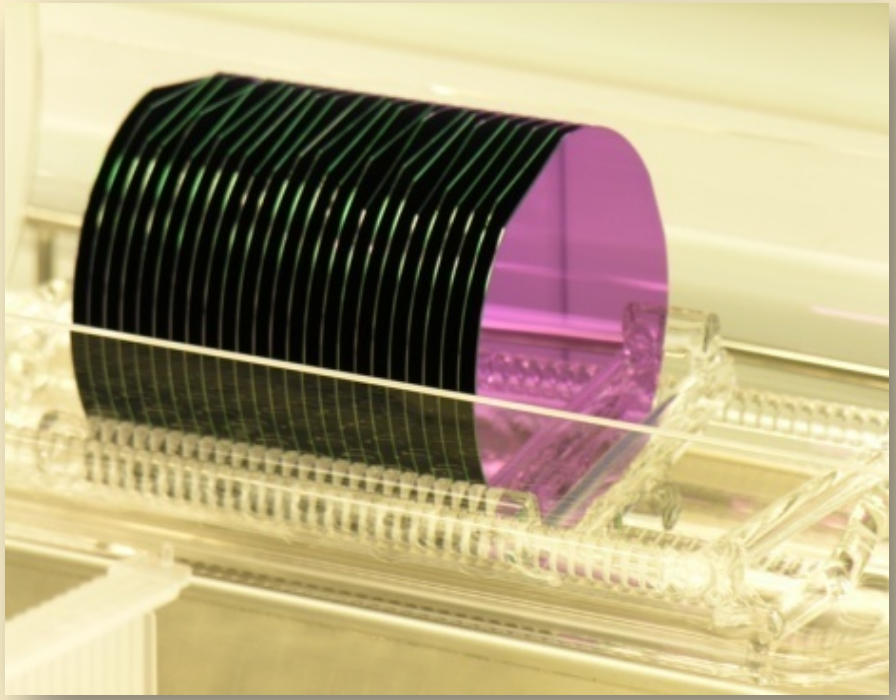


Thickness vs. Angle of Sight

Based on the color chart at the end of the Science of Thin Films Activity, estimate the thickness of a “carnation pink” wafer?

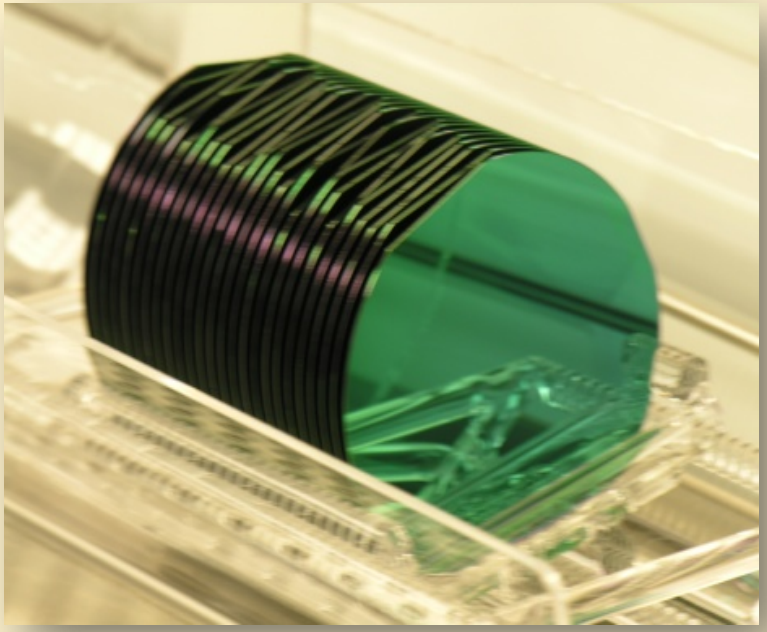
Answer:

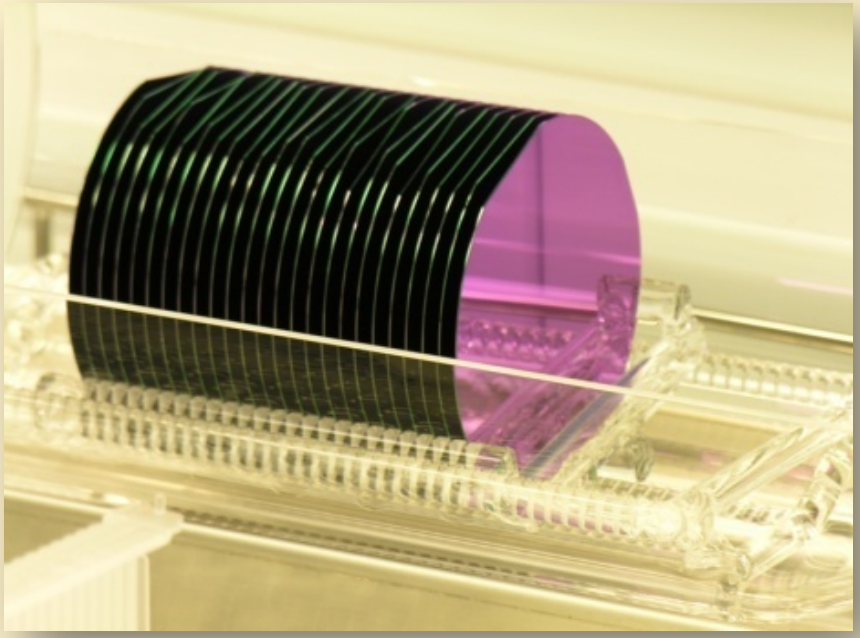
6000 angstroms



Thickness vs. line of sight

Both of these wafers have had approximately 5700 Angstroms of oxide growth. The angle at which they are shown yields a “false” reading of the oxide thickness. Remember – create a line of sight perpendicular to the wafer’s surface when interpreting a color.





Color Charts

- In the Science of Thin Films Activity, use the provided color chart to estimate oxide thickness consistently.
- It is very important that your line of sight is perpendicular to the wafer's surface. In other words, look straight down on the wafer, not at an angle.
- Keep this in mind when completing this activity. Your outcome will be affected if you do not view the wafer from a direct, top-down perspective in a consistent manner.

Activity II – Science of Thin Films

Complete Activity II in your Participant Guide:

"Science of Thin Films"

- Use a Rainbow Wafer and an Oxide Thickness vs. Color Chart to determine the oxide thickness of each color on the wafer.
- Develop several graphs from which you can extract the average etch rate. The etch rate is the amount of oxide etched in a given amount of time. It will be determined by calculating the slope of the straight line through your data points.

Summary

Even though oxide is transparent, the interference of white light reflected off the silicon crystal/oxide interface with that reflected off the oxide's top surface, creates a variation in color depending on the thickness of the oxide.

Hydrofluoric Acid (HF) can be used to etch SiO_2. The longer the etch time, the more oxide is removed. If you know the etch rate and the initial oxide thickness, you can calculate the amount of time needed to remove a specific thickness of oxide or how long you need to etch an oxide coated wafer to get a specific thickness.

Acknowledgements

Southwest Center for Microsystems Education (SCME)

800 Bradbury Drive SE, Suite 235

Albuquerque, NM 87106-4346

Phone: 505-272-7150

Website: www.scme-nm.org

The work presented was funded in part by the National Science Foundation Advanced Technology Education program, Department of Undergraduate Education grants: 0902411.