Objective

- Demonstrate the capability of the MultiPrep™ System to produce 0.5 µm thick silicon samples for SIMS analysis

Process

Sample Description:

- Two (2) small pieces of silicon

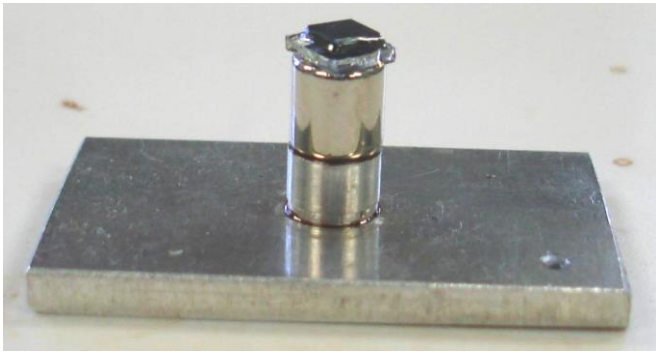
Mounting:

Prior to mounting, the Pyrex of the SIMS/Backside Pyrex Holder was ground flat with 15 µm diamond lapping film at 150 RPM in the clockwise direction on a fully calibrated MultiPrep™ System. This produces a reference surface parallel to the grinding plane so the sample can be mounted as parallel to the grinding plane as possible. The advantage of the MultiPrep™ System is that it maintains geometric alignment of the sample to the platen throughout the grinding and polishing procedure. Prior to mounting, the silicon samples were ultrasonically degreased in GP cleaning solution, rinsed with isopropyl alcohol, and heat dried to remove any oil/debris. This cleaning process improves the adhesion of the epoxy to the sample.

A 6 mm by 6 mm piece of silicon was secured to a glass microscope slide using EpoxyBond 110. The microscope slide facilitates transportation of the sample once thinned. A very small bead of the epoxy should be used between the glass and the sample to maximize flatness. The sample was placed on the epoxy with the dull side up and the area of interest facing the glass in the uncured epoxy. A magnetic clamping system was used to squeeze the epoxy as thin as possible between the sample and glass. This helps the epoxy form an even layer between the sample and the glass, which affects parallel orientation to the Pyrex and with the platen.

The magnetic clamp, with the sample and glass slide, was set onto a hot plate at 120°C for ten minutes to cure the epoxy; this is shown in Figures 1 and 2. A lower temperature was used to prevent warping of the sample, which can have an adverse effect when dealing with sub-micron tolerances. The excess glass was then removed by grinding the edges close to the sample dimensions, using 30 micron diamond lapping film. The glass

was then adhered to the Pyrex on the fixture using wax and the procedure described below.



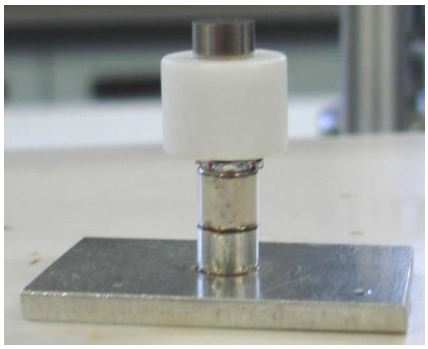


Figure 1, Left: SIMS sample with glass, on top of the magnetic clamp base
Figure 2, Right: Magnetic clamping system applying even pressure over the sample surface

A glass microscope slide was used instead of silicon for mounting the sample because it allows easier observation of the alignment by way of transmitted light. When the silicon is thinned to ten microns, light transmission produces a red color through the sample. A region that appears yellow is thinner than one that appears orange, and both are thinner than one that appears red. Therefore, tilt corrections, if necessary, can be made to the sample before it is too thin to do so without grinding further, or past the sample thickness. When mounted to a piece of silicon to help with conductivity, this technique is not possible. To enable conductivity while using a glass slide, conductive silver paint can be applied to the glass.

The SIMS/Backside Pyrex Holder was heated to 120°C on a hot plate in order to melt the wax used to secure the sample and substrate. A small amount of wax was applied to the Pyrex, where the glass was secured. The fixture was then removed from the hot plate and allowed to cool. While still hot, light pressure was applied to the sample to squeeze the wax as thin as possible, to further ensure parallel registration with the platen. A thick layer of wax could cause the sample to "float" and negatively influence its alignment. It is important that the fixture and wax not be quenched in cold water, as doing so will reduce the adhesive qualities by making it more brittle. The sample/fixture configuration is shown in Figure 3.

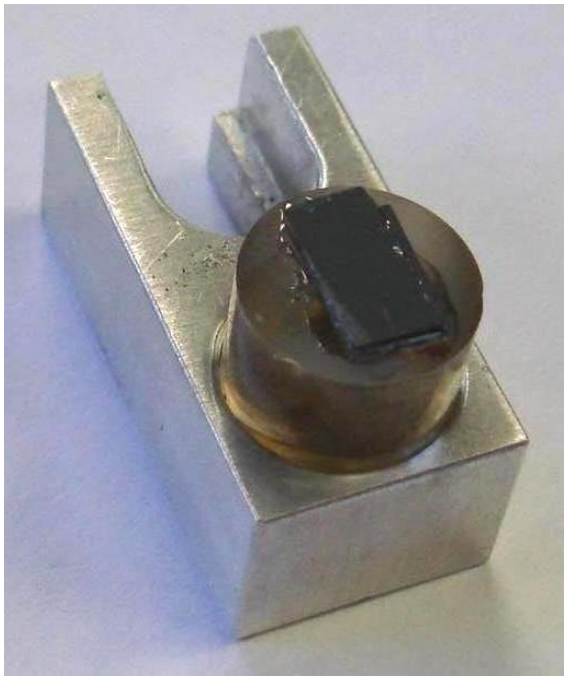


Figure 3. Silicon sample affixed to the SIMS/Backside Pyrex Holder

Polishing:

The sample was prepared on the MultiPrep™ System as outlined in Table 1. It is important when thinning the sample to follow a "3-X" rule. Grinding deforms the crystal structure of the silicon as the abrasive travels through the material. In order to remove deformation, an amount of material needs to be removed that is three times greater than the previous abrasive size with the next abrasive step. For example, after the 6 µm diamond lapping film polishing step, a minimum of 18 µm must be removed with the next polishing abrasive. BlueLube was chosen as a polishing lubricant since it better facilitates the cutting action of the lapping film below the 15 µm abrasive size.

Table 1. Silicon SIMS polishing procedure for the MultiPrep™ System

	Step	1	2	3	4	5	6	7
Consumables	Abrasive Size	30 µm	15 µm	6 µm	3 µm	1 µm	0.5 µm	0.05 µm
	Abrasive Type	Diamond	Diamond	Diamond	Diamond	Diamond	Diamond	Colloidal Silica
	Carrier	Film	Film	Film	Film	Film	Film	Suspension
	Polishing Cloth	-	-	-	-	-	-	Red Final C
	Lubricant	H_20	H_20	BlueLube	BlueLube	BlueLube	BlueLube	H_20 *
Settings	Platen Speed	80 RPM CW	80 RPM CW	80 RPM CW	50 RPM CW	50 RPM CW	50 RPM CW	175 RPM CW
	Sample Rotation	Limit (Speed 1, 90 degree sweep)	Limit (Speed 1, 90 degree sweep)	Limit (Speed 1, 90 degree sweep)	Limit (Speed 1, 90 degree sweep)	Limit (Speed 1, 90 degree sweep)	Limit (Speed 1, 90 degree sweep)	Full (Speed 6)
	Sample Oscillation	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep	Speed 1, 2.5" sweep
	Sample Load	600 g	600 g	300 g	300 g	200 g	200 g	500 g
	Time/ Material Removal	To 166 µm from the area of interest	To 76 µm from the area of interest	To 31 µm from the area of interest	To 13 µm from the area of interest (Red in transmitted light)	To 4 µm from the area of interest (Orange in transmitted light)	To 1 µm from the area of interest (Yellow in transmitted light)	1-4 minutes (Yellow and interference fringes in transmitted light)
	CW: Platen rotates in the clockwise direction							
	*** Water is only used to soak and rinse the cloth**							

When the sample is approximately 1 or 2 microns thick, usually when using the 0.5 µm diamond lapping film, transmitted light interference fringes start to appear. The spacing of these fringes can be used to estimate the flatness and thickness profile of the sample, which can be adjusted using the micrometer heads on the MultiPrep™.

Final angle adjustments, if needed, should be implemented as soon as the alignment of the sample can be identified. This is best done during the 1 or 0.5 micron diamond film step. Final polishing of the sample was performed on Red Final C with 0.05 µm colloidal silica suspension. Red Final C is a dense, low-napped silk cloth that provides an excellent final polish. Longer polishing times remove material but also yield a smoother and flatter surface finish. Polishing time also depends on the surface area of the sample.

The AD-5™ Automatic Fluid Dispenser provided automatic dosing of the polishing suspension and lubricant, allowing for unattended and consistent sample preparation.

Between grinding and polishing steps, and before imaging, the sample was cleaned with Micro Organic Soap to remove micro contaminants and polishing solutions from the sample surfaces. This helps to reduce particle contamination from one cloth to the next, which results in scratches and a poor surface quality. The sample was then rinsed in isopropyl alcohol and gently dried using Aero-Duster compressed air.

Following completion of the polishing and image analysis, the sample fixture was heated to 120°C on a hot plate to melt the wax. A lower temperature was used to avoid warping of the thin sample. Very carefully, the sample was removed from the fixture; acetone was used to dissolve any remaining wax from the sample, followed by a rinse with isopropyl alcohol. The sample was then placed in a Gel-Pak for transportation.

Imaging and Analysis:

Figures 4 through 7 are micrographs of the thinned sample, which were captured with various magnifications using the ZEISS Axio Imager.A1m™ upright microscope, Axiocam MRc 5™ digital camera, and AxioVision 4™ imaging software. The various settings were used to illustrate the capabilities of the microscope and reveal as much of the samples' features as possible. Please note that all magnification values are original optical magnification.

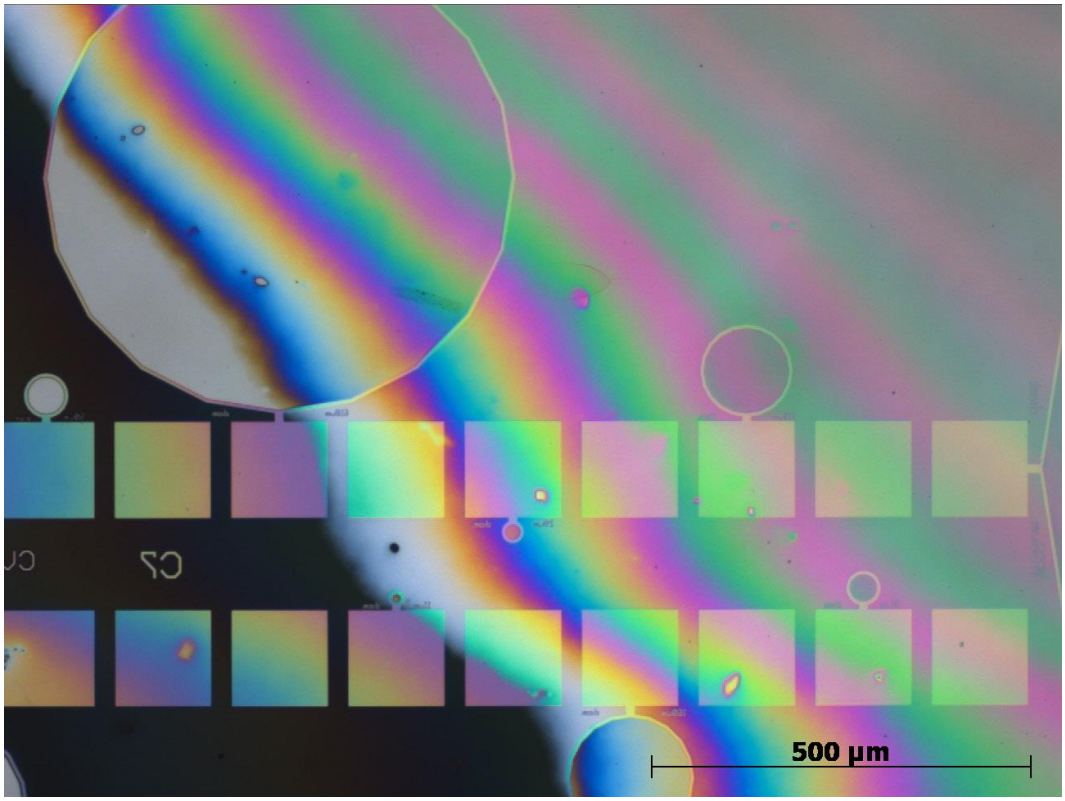


Figure 4. Light interference fringes, Brightfield, Taken at 100x

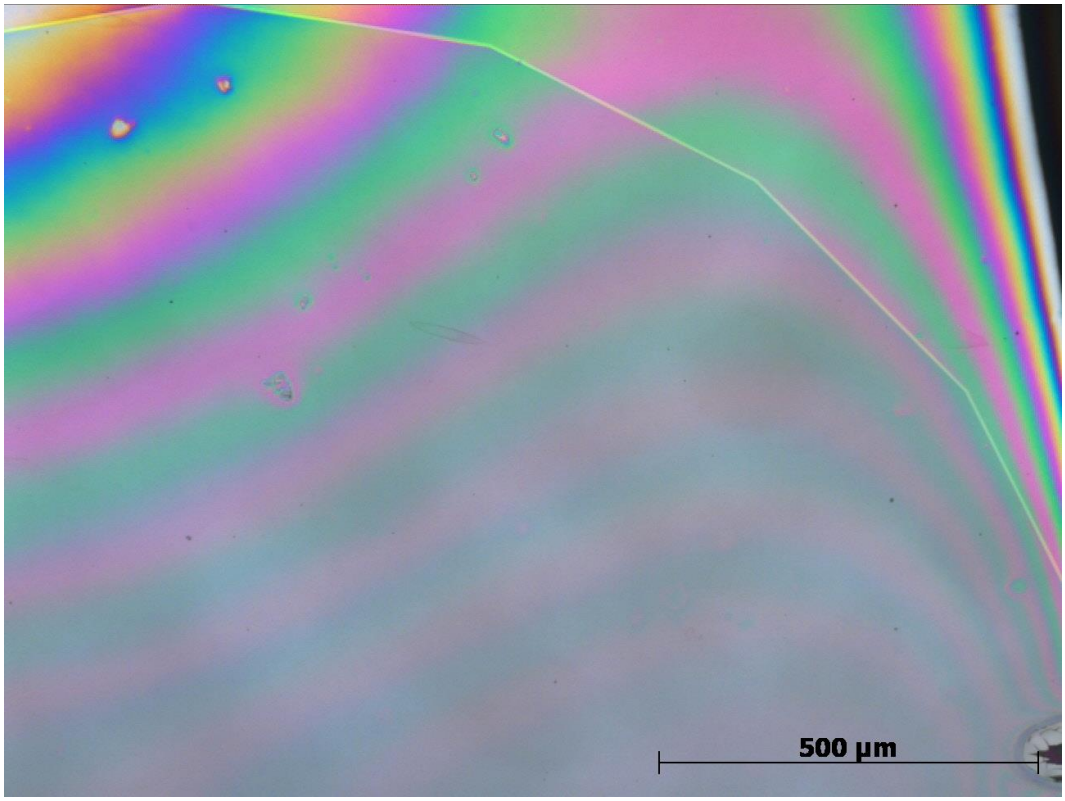


Figure 5. Light interference fringes, Brightfield, Taken at 100x

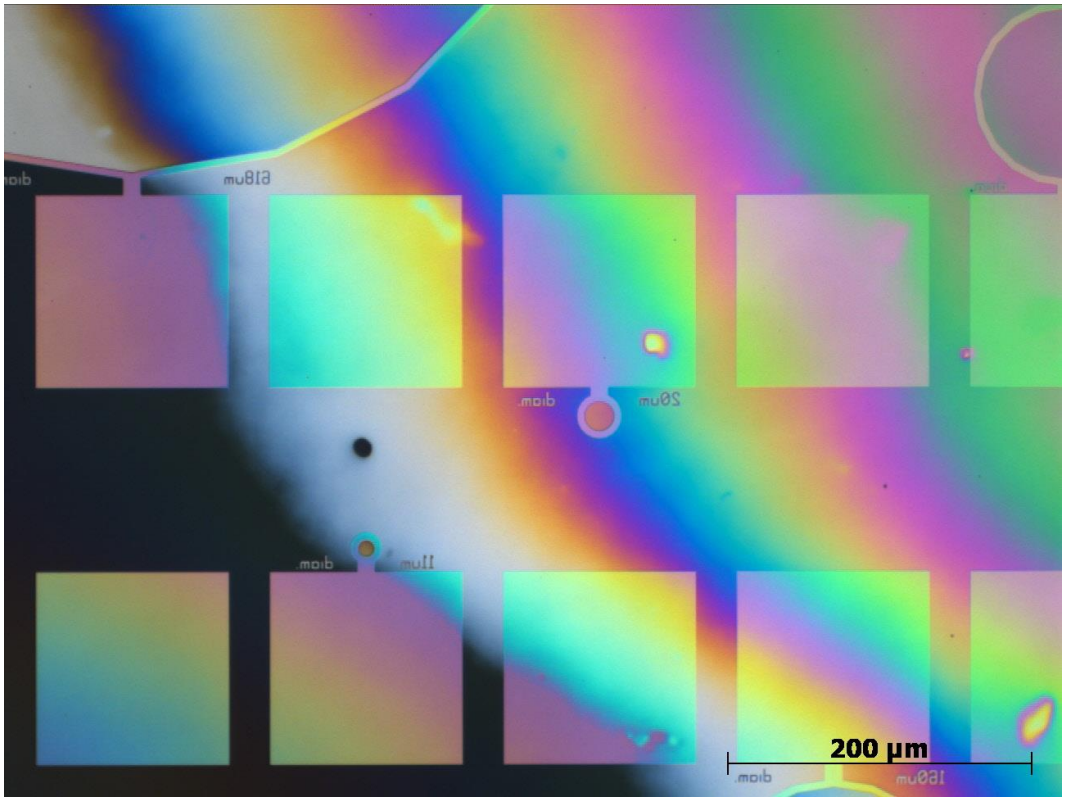


Figure 6. Light interference fringes, Brightfield, Taken at 200x

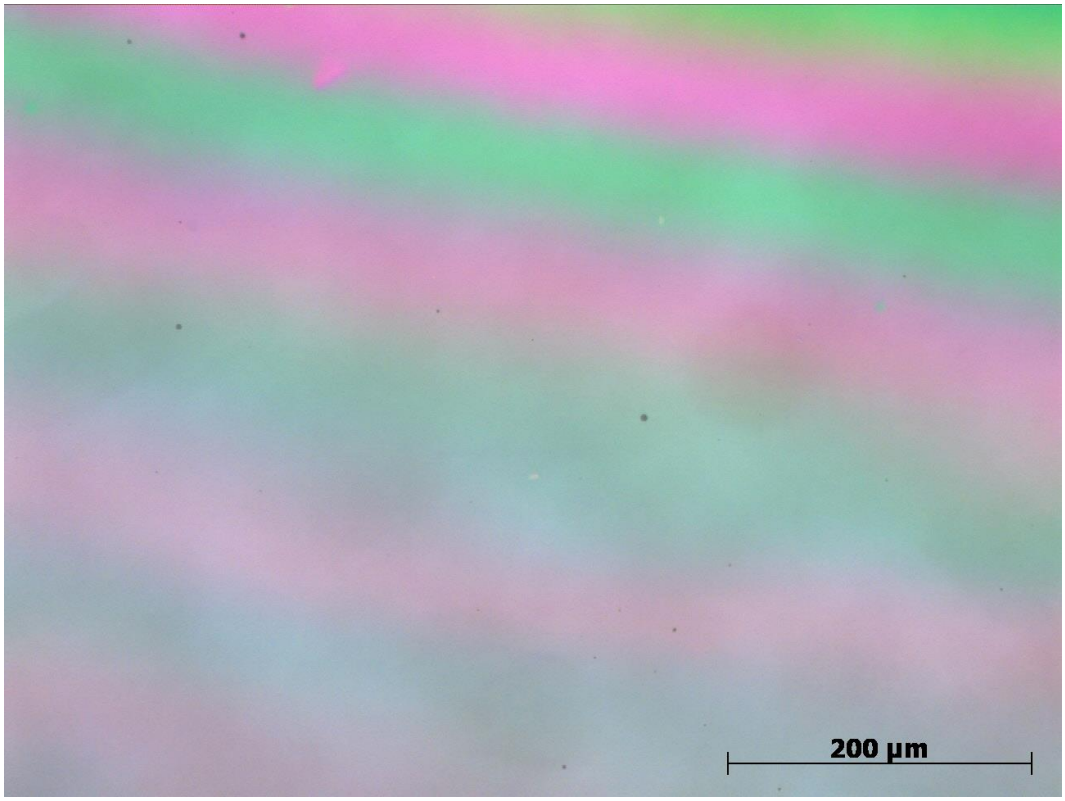


Figure 7. Light interference fringes, Brightfield, Taken at 200x

Equipment and Consumables

Equipment Used:

- MultiPrep™ System, Gear Drive
- SIMS/Backside Pyrex Holder
- AD-5™ Automatic Fluid Dispenser

ZEISS Stemi DV4™ Stereomicroscope
ZEISS Axio Imager.A1m™ Upright Microscope
ZEISS AxioVision 4™ Imaging Software
ZEISS Axiocam MRc 5™ Digital Camera

Note: Item numbers for microscopes and software depend upon desired configuration.

MultiPrep™ System &
AD-5™ Fluid Dispenser

Consumables Used:

- GP Cleaning Solution
- EpoxyBond 110 Kit
- Hot Mounting Wax
- 30 µm Diamond Lapping Film
- 15 µm Diamond Lapping Film
- 6 µm Diamond Lapping Film
- 3 µm Diamond Lapping Film
- 1 µm Diamond Lapping Film
- 0.5 µm Diamond Lapping Film
- 0.05 µm Blue Colloidal Silica
- BlueLube Polishing Lubricant
- Red Final C Polishing Cloth
- Micro Organic Soap
- Aero-Duster Compressed Air

Axio Imager.A1m™
Upright Microscope