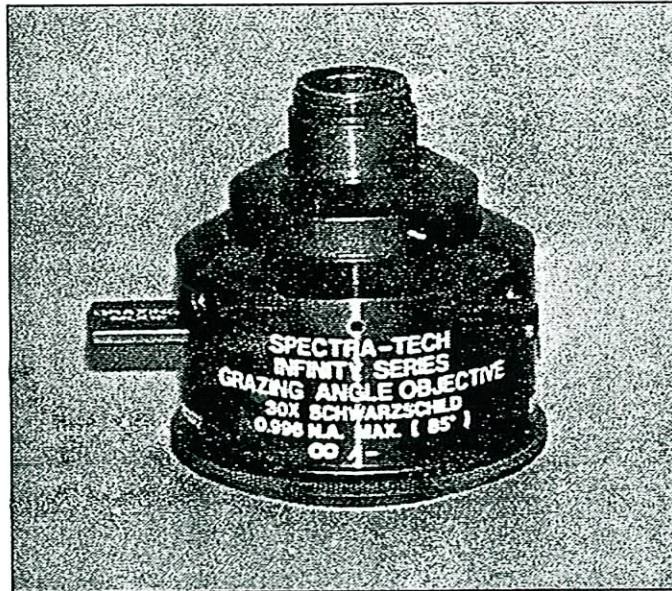


INFINITY SERIES
GRAZING ANGLE OBJECTIVE
P/N 0045-407
USER'S MANUAL

Version 1.0



P/N 700-0132

SPECTRATECH

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GENERAL INFORMATION

Introduction to Grazing Angle Microscopy

The Grazing Angle Objective (GAO) has been developed to allow high sensitivity analysis of monomolecular films in microscopic areas (from 250 to 25 μ diameter areas). Typically, samples consist of organic or inorganic ultra-thin films on metallic surfaces. The measurement of the energy reflected from the surface of these samples can yield important spectral data which provides information about the molecular chemistry and orientation of surface films. Some applications of this technique include:

- contaminants on semiconductors
- surface impurities on magnetic disks
- coating imperfections on metallic surfaces

Theory

For a description of the theory of Grazing Angle Microscopy please refer to Spectra-Tech's FT-IR Technical Note #3: *External Reflectance Spectroscopy of Surfaces*. Also, please refer to Appendix A : cutaway diagram of the Grazing Angle Objective.

The Manual

This manual is designed as a tutorial guiding you through a typical grazing angle microscopical analysis. It is recommended, however, that you familiarize yourself with the operation of the microscope before using the GAO.

The Grazing Angle Objective holds U.S. patent #4,810,077.

GENERAL INFORMATION

Installation

Installation of the Grazing Angle Objective (GAO) is provided by a qualified service engineer. Additionally, if the GAO is being retrofitted to an existing IR-Plan Research Microscope, a purge upgrade is provided. If you have any questions regarding the installation or purge, please contact a service engineer at 1-800-243-9186.

CAUTION: Removal of the GAO from the nosepiece can result in misalignment. Before removing the GAO please contact a Spectra-Tech service engineer.

Technical Specifications

Magnification:	30X
Numerical Aperture:	0.996 at 85° 0.51 at 33°
Range of incidence:	33 - 45° near normal 65 - 85° grazing angle
Working Distance:	1 mm
Objective Type:	Schwarzschild design

Technical Support

Technical materials describing the use and theory of Grazing Angle Microscopy (GAM) are available from Spectra-Tech. Additionally, a team of scientists are available at Spectra-Tech to answer any questions you have concerning Grazing Angle Microscopy and the use of the Grazing Angle Objective.

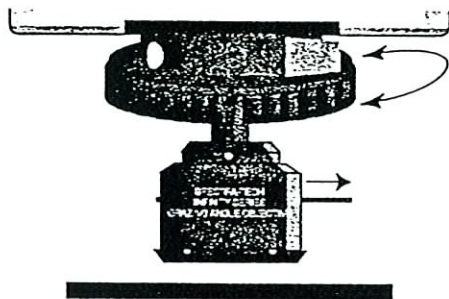
Getting Started

Initial Preparation

Check Purge

The operating purge flow rate should be checked periodically to ensure that it always remains at 1 ft³/min (28.3 liters/min.).

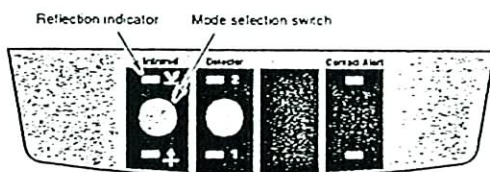
CAUTION: It is imperative that the stage is lowered before attempting to move the GAO into position.



Adjust GAO for Viewing

Firmly grip the knurled black nosepiece ring and swing the GAO into position.

Slide the Grazing/Viewing Selector (on the GAO) into the *viewing mode* position.



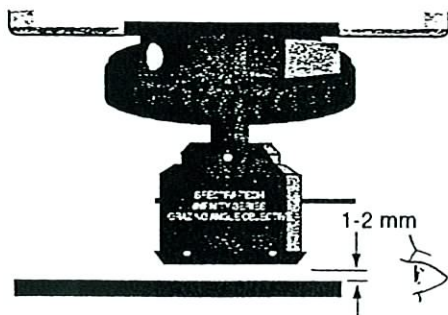
Put the microscope into the *Reflection mode*.

Place the gold mirror on the stage.

Using the Coarse Focus adjustment knob, raise stage so that it is 1-2mm from the bottom of the GAO.

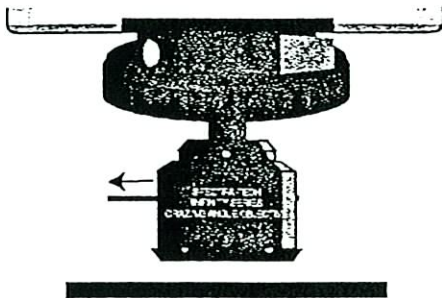
Verify the Reflex aperture is open to its maximum position. With the GAO, the aperture size is 75 μ m.

Using the Fine Focus knob, raise the stage and focus on the mirror.



USING THE GRAZING ANGLE OBJECTIVE

Initial Preparation



Adjust GAO for Grazing Incidence

Slide the Grazing/Viewing Selector (on the GAO) into the *grazing mode* position.

Refocus the sample using the fine focus adjustment knob.

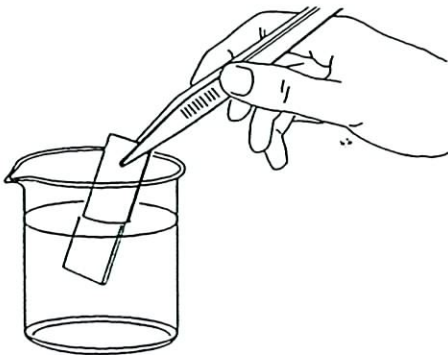
Measure signal throughput

Still in the Reflection mode, check the energy throughput. The energy should be comparable to the energy throughput at the time of installation.

Note throughput for later reference.

Performing an Experiment

Sample Preparation & Measurement



Prepare Reference Mirror

A gold-coated microscope slide is supplied for use in collecting background spectra. Before use it must be cleaned* to remove any organic material picked up from the atmosphere. Any residue remaining on the mirror could present problems in a grazing angle analysis.

*sulfuric acid-chromic acid cleaning solution (Glass-Terg†) is a suggested cleaning agent.

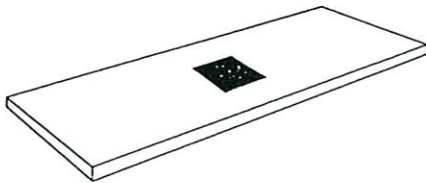
†Glass-Terg is a brand name of Brand Nu Laboratories Inc., Meriden, CT. 203-235-7989.

USING THE GRAZING ANGLE OBJECTIVE

Performing an Experiment *Sample Preparation & Measurement*

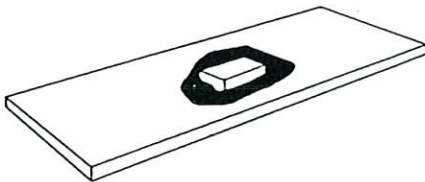
Mount Flat Sample

Place a sample onto a standard 1" x 3" microscope slide.



Mount Irregular Sample

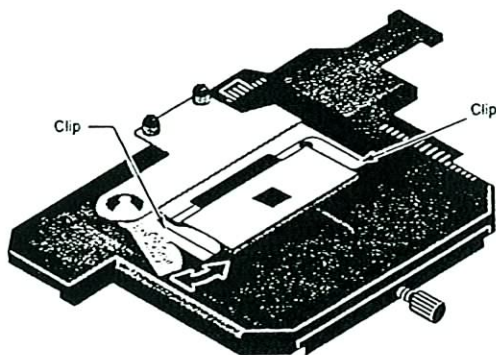
Press the sample into Plasticine® or modeling clay on a standard microscope slide. Position the sample so that the top surface is level.



Note: Be careful not to touch the analysis area.

Place Sample on Stage

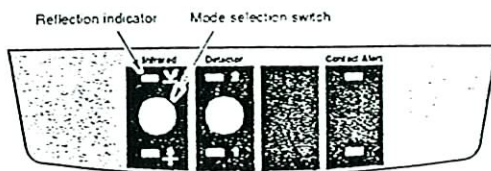
Place sample securely on the stage with the stage clips (mounting plates should be used with the Motorized Stage).



USING THE GRAZING ANGLE OBJECTIVE

Performing an Experiment

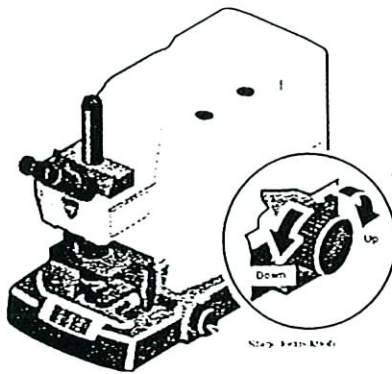
Sample Preparation & Measurement



Adjust Controls for Reflection Viewing*

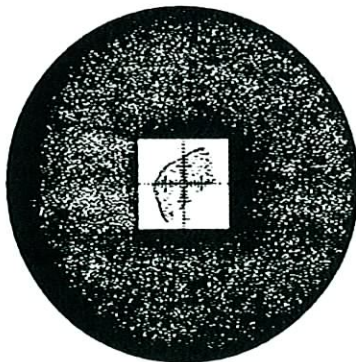
Ensure the microscope is in the *Reflection mode*.

Adjust the reflection illumination to a comfortable viewing level.



Locate Sample

Use the stage positioning controls to position the sample so the area to be analyzed is centered on the crosshairs



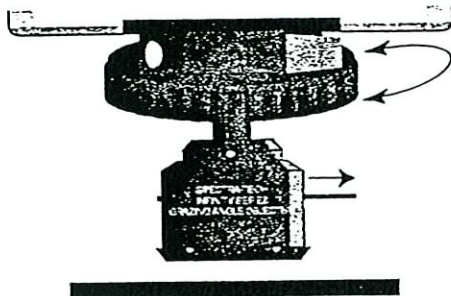
Note In the case of very small sample areas, you may want to switch to the reflachromat 15X or 32X objective or a glass objective of higher magnification to focus on the sample.

Although it might not be possible to visualize some thin layer deposits and contaminants, measurement in the infrared is still possible.

CAUTION: It is imperative that the stage is lowered before attempting to move the GAO into position.

USING THE GRAZING ANGLE OBJECTIVE

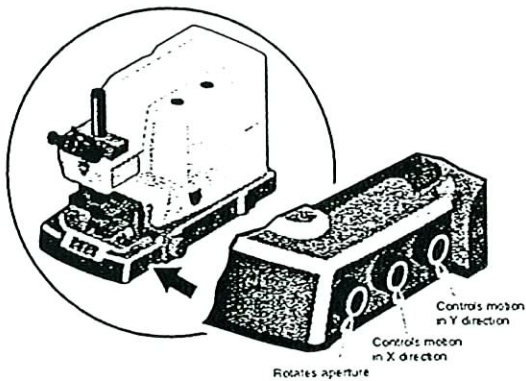
Performing an Experiment *Sample Preparation & Measurement*



Adjust GAO for Viewing

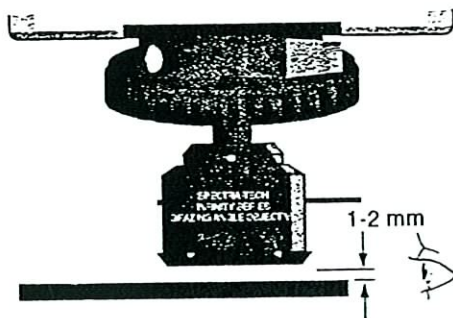
Firmly grip the knurled black nosepiece ring and rotate the GAO into position.

Move the Grazing/Viewing Selector Slide (on the GAO) into the *viewing mode* position.



Adjust Aperture

Verify the Reflex aperture is open to its maximum position. With the GAO, the aperture size is 75 x 75 μm .



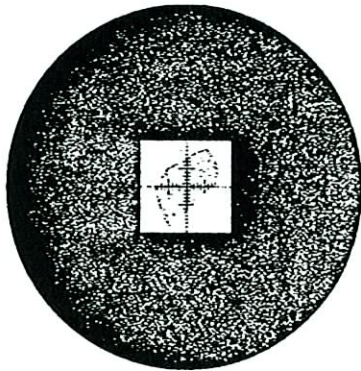
Raise Sample

While viewing the objective and sample from the side, use the Coarse Focus Adjustment knob to raise the sample to within 1-2mm of the Grazing Angle Objective.

USING THE GRAZING ANGLE OBJECTIVE

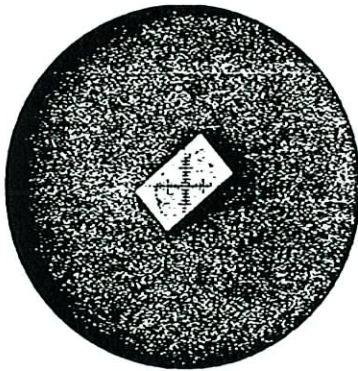
Performing an Experiment

Sample Preparation & Measurement



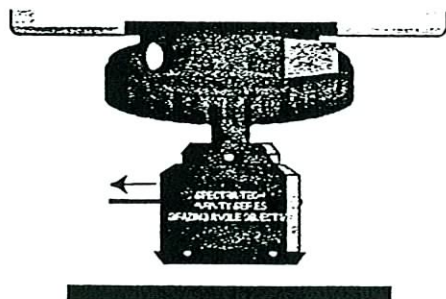
Bring Sample into Focus

While looking through the microscope bring the sample into sharp focus by using the Fine Focus Adjustment knob. (The edges of the aperture are sharp and the field is at its brightest when the sample's surface is in focus).



Adjust Aperture to Sample Area

Rotate the aperture and adjust the blades to conform with the sample's geometry and dimensions.



Set the GAO for Grazing Angle Mode

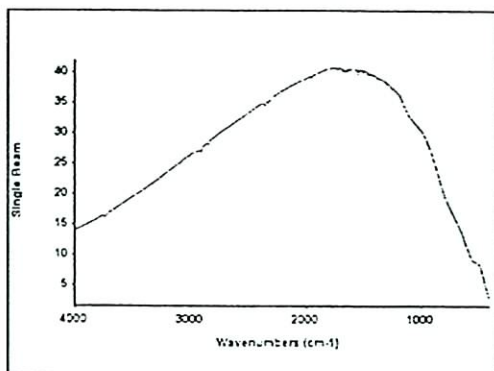
Move the Grazing/Viewing Selector Slide (on the GAO) to the *grazing mode* position.

Using the Fine Adjustment, ensure that the sample and aperture are in sharp focus.

Note: Because of the low angles of incidence in *grazing mode*, the image is frequently sharper in the *viewing mode*.

USING THE GRAZING ANGLE OBJECTIVE

Performing an Experiment Sample and Background Measurement



Collect a Sample Single-Beam Spectrum

- Check energy
- Set gain
- Collect long enough to achieve adequate signal to noise - this may require more than 1000 scans when band intensities are weak.
- 8 cm^{-1} resolution is recommended for initial GAO experiments.

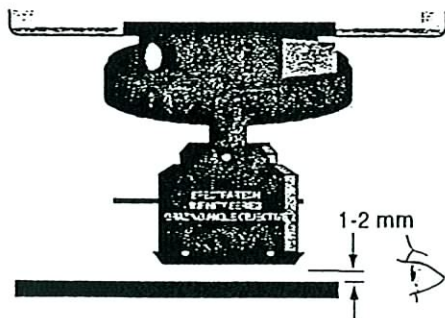
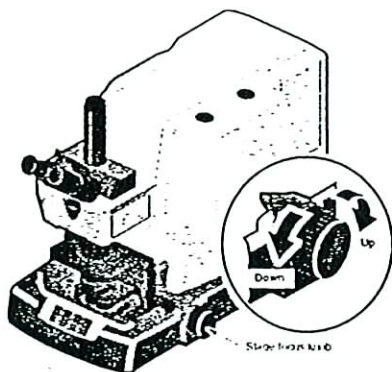
Reference Mirror

Without adjusting or changing the aperture, use the Coarse Focus Adjustment to lower the sample stage.

Remove the sample from the stage.

Place a pre-cleaned gold reference mirror securely on the stage.

Note: A background spectrum can be recorded prior to the sample spectrum if desired but it must be collected with the same size aperture.



Raise Reference Mirror

While viewing the position of the stage from the side, use the Coarse Adjustment knob to raise the reference mirror to within 1-2mm of the Grazing Angle Objective.

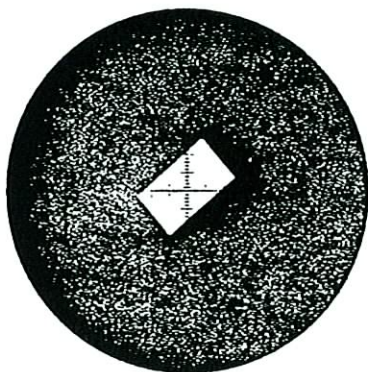
Check the Grazing/Viewing Selector (on the GAO) to ensure that it is in the *grazing mode* position.

Using the Coarse and Fine Adjustment knobs, ensure that the reference mirror and aperture in sharp focus.

USING THE GRAZING ANGLE OBJECTIVE

Performing an Experiment

Sample and Background Measurement

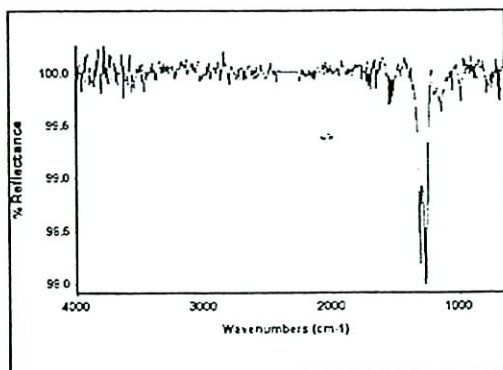


Bring Reference Mirror into Focus

While looking through the microscope, bring the reference mirror into sharp focus by using the Fine Focus Adjustment knob.

Collect a Background Spectrum

Collect a Background single-beam spectrum.



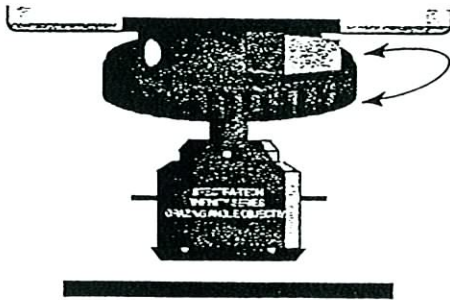
Ratio Spectra

Ratio background single-beam spectrum to previously acquired sample single-beam spectrum.

Note: This function is usually done automatically in the software by selecting the final format as %R.

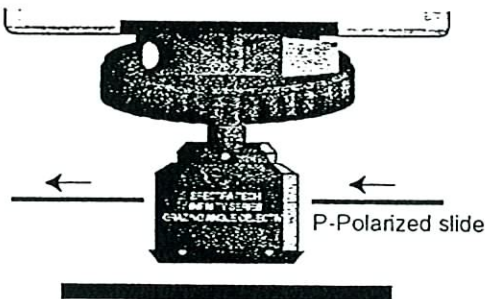
USING THE GRAZING ANGLE OBJECTIVE

Using the IR Polarizer *Sample Preparation & Measurement*



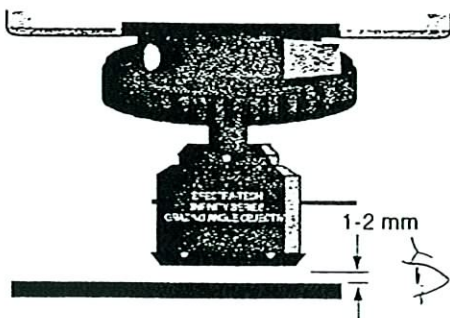
Adjust GAO for Viewing

After lowering the stage, firmly grip the knurled black nosepiece ring and rotate the GAO into position.



Change GAO Selector Slide

Remove the original selector slide and insert the P-Polarized Selector slide.



Focus on Sample

Move the P-Polarized Selector slide into the *viewing mode*. While viewing the position of the stage from the side, use the Coarse Adjustment knob to raise the reference mirror to within 1-2mm of the Grazing Angle Objective.

Using the Coarse and Fine Adjustment knobs, ensure that the sample is centered and in sharp focus.

USING THE GRAZING ANGLE OBJECTIVE

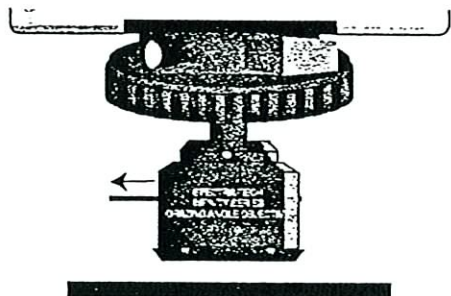
Using the IR Polarizer

Sample Preparation & Measurement



Adjust Aperture to Sample Area

Rotate the aperture and adjust the blades to conform with the sample's geometry and dimensions.



Set the GAO for Grazing Angle Mode

Move the P-Polarized Selector Slide to the *grazing mode* position.

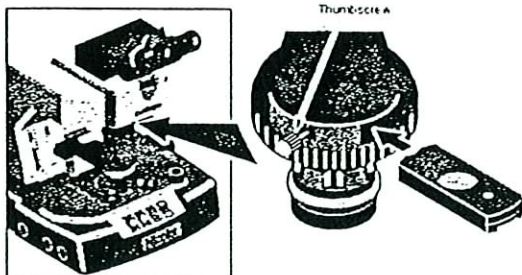
Using the Fine Adjustment, ensure that the sample is in sharp focus.

Note: Because of the low angles of incidence in *grazing mode*, the image is sharper in the *viewing mode*.

Install Infrared Polarizer

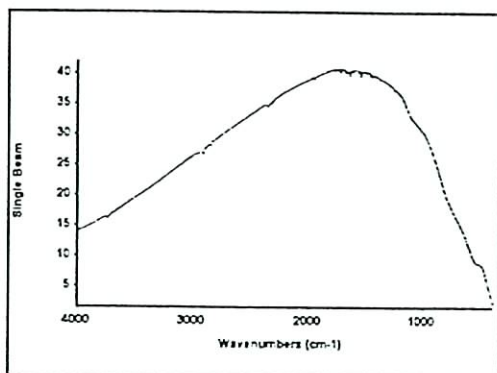
Insert the infrared polarizer into the nosepiece slot above the objective with the increment dial facing down.

Turn the increment dial to maximize infrared energy.



USING THE GRAZING ANGLE OBJECTIVE

Using the IR Polarizer *Sample Preparation & Measurement*



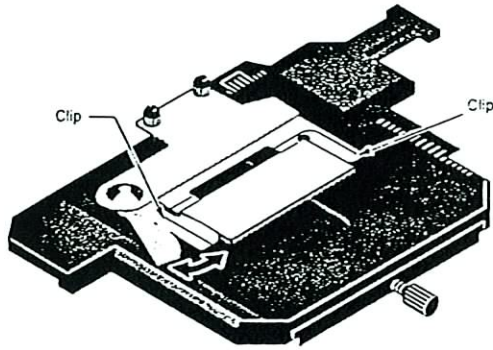
Collect a Sample Spectrum

Ensure the microscope is in the *Reflection mode*.

Collect a sample single-beam spectrum.

USING THE GRAZING ANGLE OBJECTIVE

Using the IR Polarizer *Background Measurement*

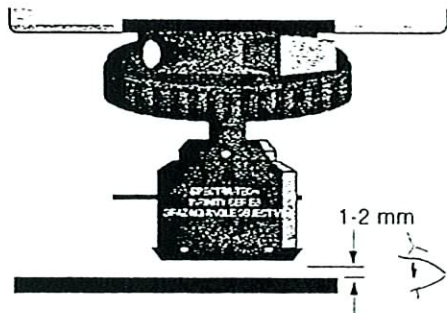


Place Reference Mirror

Without adjusting or changing the aperture, use the Coarse Focus Adjustment to lower the sample stage.

Remove the sample from the stage.

Place a pre-cleaned gold reference mirror securely on the stage.



Raise Reference Mirror

While viewing from the side, use the Coarse Adjustment knob to raise the reference mirror to within 1-2 mm of the Grazing Angle Objective.

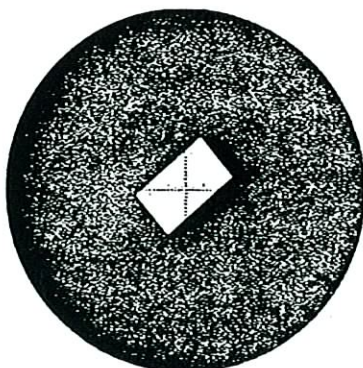
Check the P-Polarized Grazing/Viewing Selector (on the GAO) to be sure it is in the *P-Polarized grazing* position.

Using the Fine Adjustment knob, cautiously ensure that the mirror is in sharp focus.

Bring Reference Mirror into Focus

Looking through the microscope, bring the reference mirror into sharp focus by using the Fine Adjustment knob.

Note: Do not change the position of the Reflex aperture blades.



USING THE GRAZING ANGLE OBJECTIVE

Using the IR Polarization Background Measurement

Collect a Background Spectrum

Move the P-Polarized Grazing/Viewing Selector (on the GAO) to the *P-Polarized grazing* position.

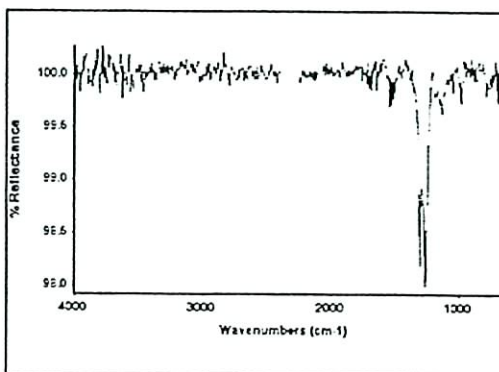
Refocus on the reference mirror.

Collect a reference single-beam spectrum.

Ratio Spectra

Ratio background single-beam spectrum to previously acquired sample single-beam spectrum.

Note: This function is usually done automatically in the software by selecting the final format as %R.



APPENDIX A

Cross-Section of the Grazing Angle Objective

