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CRADA Final Report
For
CRADA Number NFE-12-04041

Upgrade and Operation of the Second Generation Two-Modulator Generalized Ellipsometry Microscope (2-MGEM2)

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ORNL-27 (4-00)

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Abstract

The two-modulator generalized ellipsometry microscope (2-MGEM) is now the standard instrument for determining the crystallographic anisotropy of the IPyC and OPyC layers in tristructural isotropic (TRISO) nuclear fuel. This instrument was first developed in 2003-2004. Since that time, the operating system of the computer and much of the hardware has become obsolete. We have undertaken to update both the software and hardware to currently supported products so that the instrument can continue to support the nuclear fuel program at ORNL and DOE.

Statement of Objectives

The 2-MGEM instrument at ORNL and the Hinds instruments in the field will be upgraded to run on Windows 7 with upgraded hardware, and will now be called 2-MGEM2. Furthermore, the resulting ORNL instrument would be completely compatible with the Hinds instruments, and therefore Hinds could service the ORNL instrument.

1. **Electronics.**
   Electronic circuits and an electronics enclosure box will be constructed by Hinds Instruments. This box will perform the following functions:
   a. Control the photomultiplier (PMT) voltage to give a constant current out of the PMT.
   b. Generate a trigger pulse when the photoelastic modulators (PEMs) are in phase.
   c. Control the PEMs, allowing external setting of the retardation amplitude and the ability to read the frequency of the PEM.
2. **Hardware**
   Several pieces of obsolete hardware will be replaced with currently supported hardware. In addition, the optics will be optimized to improve the light collection efficiency of the instrument.
3. **Software development**
   The software used to run the 2-MGEM will be updated, written in Visual Basic.NET 2010 to operate on Windows 7.
4. **Software Development (Analysis Package).**
   The software used to analyze the 2-MGEM data will be written in Visual Basic.NET 2010 to operate on Windows 7. The new package will be independent of the software to run the 2-MGEM, so it can be put on any computer.

Benefits to the Funding DOE Office's Mission

At the end of this project, the Advanced Gas Reactor (AGR) program will have an updated 2-MGEM instrument to used for the characterization of TRISO nuclear fuel. In addition, the instrument at B&W in Lynchburg, VA may be updated via Hinds Instruments.

Technical Discussion of Work Performed by All Parties

See the attached Technical Discussion, *Upgrade and Operation of the Second Generation Two-Modulator Generalized Ellipsometry Microscope (2-MGEM2)*.

Subject Inventions

None

Plans for future Collaborations

Hinds Instruments and ORNL will continue to collaborate on applications and projects that involve polarization optical metrology.

Conclusions

The 2-MGEM2 has been completely updated, resulting in improved performance and reliability. In particular (See the Technical Discussion section for more details):

1. Software. The computer program running the 2-MGEM2 is now written in Visual Basic.NET 2010, using the Windows 7 operating system. The data analysis program is now separate from the main program, and also runs on Windows 7.
2. Algorithms. New algorithms for the 2-MGEM2 have been written to improve the speed at which data can be taken. The time required to take a registration scan has been reduced from 1 to 1 ½ hours to ~12 minutes, a reduction of a factor of 5-7. The time required to take a 2-MGEM2 data set on a single particle has been reduced from 3 ½ - 4 hours to 1-1 ¼ hours, a reduction of nearly a factor of 3.
3. Hardware. Several improvements have been made to the hardware, including:
    a. New rotators
    b. New controllers for the rotators and translators
    c. New CCD camera for imaging the particle during measurement
    d. Newly designed detector assembly, resulting in more light reaching the detector
    e. New data acquisition card

See the Technical discussion for details.

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April 2013

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Acronyms

2-MGE	Two-modulator generalized ellipsometer
2-MGEM	Two-modulator generalized ellipsometry microscope
2-MGEM2	Second generation 2-MGEM
AGR	Advanced Gas Reactor
B&W NOG	Babcock and Wilcox Nuclear Operations Group
CRADA	Cooperative Research and Development Agreement
CCD	Charge-coupled device
CMOS	Complementary metal-oxide semiconductor
DAQ	Data acquisition
GUI	Graphical user interfaces
IPyC	Inner pyrocarbon layer between the buffer and SiC in a TRISO particle
ITU	Institute for Transuranium Elements
NI	National Instruments
OPyC	Outer pyrocarbon layer
PCIe	Peripheral component interconnect express
PEM	Photoelastic modulator
PSA	Polarization state analyzer
PSG	Polarization state generator
PMT	Photomultiplier tube
Std. Dev.	Standard deviation
TRISO	Tristructural isotropic nuclear fuel particle, (Kernel, Buffer, IPyC, SiC, OPyC)
USAF	United States Air Force
VB6	Visual Basic 6

Introduction

The two-modulator generalized ellipsometry microscope (2-MGEM) [refs. 1–7] is now the premier instrument used to characterize the crystallographic anisotropy of the pyrocarbon layers in tristructural isotropic (TRISO) nuclear fuel. These layers are critical to the performance of the TRISO fuel — if the crystallographic anisotropy is too high, then anisotropic irradiation-induced dimensional changes in the inner pyrocarbon (IPyC) layer during operation in a nuclear reactor may lead to fracture. Fracture of the IPyC can possibly result in damage to the overlaying silicon carbide and the unwanted release of fission products from the TRISO particle.

At the beginning of the Advanced Gas Reactor (AGR) Fuel Development and Qualification program, it was decided that a revolutionary technique for the measurement of the optical anisotropy of pyrocarbon layers in TRISO nuclear fuels would be developed based on ORNL's two-modulator generalized ellipsometer (2-MGE) technology [refs. 1–4]. As a result, ORNL developed the two-modulator generalized ellipsometry microscope (2-MGEM). Application of the 2-MGEM to the measurement of pyrocarbon layers in TRISO particles was extremely successful, resulting in measurements that were at least a factor of 10 times more accurate than alternate polarimetry techniques, while being considerably faster. This has resulted in some recognition of ORNL and the AGR program, including an R&D 100 award and several publications. The technology has been transferred to Hinds Instruments and their versions of the device have been sold to the Institute for Transuranium Elements (ITU) in Karlsruhe, Germany, and to Babcock and Wilcox Nuclear Operations Group (B&W NOG) in Lynchburg, Virginia.

Work on the 2-MGEM started in 2003 and used software and electronics compatible with the existing 2-MGE, which had been developed under a seed money project in 1994–1995. In the interest of speed of development, it was decided to also use as much existing software as possible, changing components as needed. As a result, the 2-MGEM was programmed using Visual Basic 6 (VB6) from Microsoft. Unfortunately, this software has been obsolete since 2005; moreover, existing VB6 program files are incompatible with Windows 7, so the 2-MGEM must be run using the Windows XP operating system, which itself is now obsolete. In January 2012, the original version of the 2-MGEM was at the end of its useful life in that the computer and some of its boards would arbitrarily shut down, requiring a reboot of the entire system, losing any unsaved data. The origin of this intermittent shutdown was never determined, but it was getting worse as time went by.

As a consequence, a project was undertaken to upgrade and revitalize the 2-MGEM to make the instrument available for measurements for the next 10 years or longer. This modernization required that the computer operation of the instrument use a currently supported operating system and programming language, and that all the obsolete parts of the instrument be replaced with modern, currently supported hardware. In addition, the instrument and software was made more user-friendly, including consolidation of the electronics, implementation of a larger computer screen to present the data and to interact with the user, and improved graphical user interfaces (GUI). Different measurement schemes were designed to improve the speed of data collection and the optics were improved so that more light is collected at the detector. Finally, the data analysis program was separated from the operating software so that it can reside on computers other than the one running the instrument. This last improvement will mean that users can use the data analysis program to look at data at any time.

This technical manuscript consists of three parts. The first part is a report that summarizes the changes that have been made to produce the second generation 2-MGEM system (2-MGEM2), and quantifies the upgrades as much as possible. The first attachment is a manual for the operation of the 2-MGEM2 system, and the second attachment is a manual for the operation of the analysis software used specifically for the analysis of the data taken on TRISO nuclear fuel particles. Much of the operational detail of the 2-MGEM2 is included in these attachments.

Summary of the Upgrades

The next generation 2-MGEM2 is an updated and upgraded version of the original 2-MGEM. This significant revision impacts almost every element of the 2-MGEM2 system. The old computer system has been replaced, which also necessitated an upgrade of the operating system, replacement of obsolete software, and modernization of data communication protocols. Key optical and electronic components have also been replaced to take advantage of recent technological developments and ensure future vendor support. As a part of the software revision, improvements have been made to the analysis methods, user interface, and data output.

The operating software of the 2-MGEM2 has been rewritten using the Microsoft Visual Studio 2010 (Visual Basic.NET) development system. A stand-alone desktop analysis package has also been written in the new language, and has been tested out on previously acquired data. A new master electronics enclosure, housing controllers for most of the components, has been built by Hinds Instruments under a Cooperative Research and Development Agreement with ORNL (CRADA NFE-12-04041); this electronics box has been tested both by Hinds and ORNL and works as designed.

Two major operational improvements have been achieved as part of the hardware and software upgrade. First, the redesigned light source and detector head now result in nearly a factor of 10 times more light reaching the detector than typical for the 2-MGEM. This increase in light results in a decrease in the time required to collect data on a single particle from 3–4 hours to 1–1¼ hours. Second, a new technique has been employed to measure the initial registration image acquired at set-up, reducing the time required from ~90 minutes to ~15 minutes.

Table 1 provides a summary of the major upgraded elements included in the new 2-MGEM2.

Table 1. Summary of improvements to the 2-MGEM2

Improvement	Comments and Results
Upgrade from a Windows XP to a Windows 7 computer, and software migration from Visual Basic 6.0 to Visual Studio 2010	Visual Studio 2010 is presently supported by Microsoft and is compatible with Windows 7; Visual Basic 6.0 is not. The system now uses a modern computer monitor with 1920x1080 resolution or better, which improves the user interface.
Upgrade of the translator and rotator controllers	The new controllers (Newport SMC100C) are current technology, which is supported by the vendor and compatible with modern computer communication protocols. Using the same controllers for both rotators and translators simplifies code writing and maintenance.
Upgrade of the light delivery system, detector head and camera	The new light delivery system and detector head result in a factor of 10 times more light getting to the photomultiplier tube (PMT). This decreases the measurement time. The old charge-coupled device (CCD) camera was obsolete; the new complementary metal-oxide semiconductor (CMOS) camera performs better, providing an enhanced dynamic range and increased resolution.
Upgrade of the photoelastic modulators	The photoelastic modulators (PEMs) have been replaced with modern units that have automatic temperature control for improved stability.
Upgrade of data acquisition (DAQ) electronics	The old DAQ solution required two peripheral component interconnect (PCI) boards as well as a break-out box. The new DAQ board uses the more modern PCI-express motherboard interface and handles the requirements of both previous boards with a simplified break-out box, which is incorporated into the Hinds electronics package.
Consolidation of cabling and component controllers in a single master electronics enclosure	The Hinds Instruments master electronics enclosure provides a serviceable unit with simplified connections to the computer. This enclosure gathers together the numerous individual electronics pieces into one organized package.
New algorithm to measure the particle registration	The new technique for defining particle position during analysis set-up is a factor of ~5–6 times faster than the previous technique.
New algorithm to perform the 2-MGEM measurements	A new technique for performing the scanning measurements has been developed that decreases the measurement time from 3½–4 hours to ~1–1¼ hours per TRISO nuclear fuel particle.
Data analysis package is now separate from the 2-MGEM operation package	Separating the data analysis package from the operating program allows the user to put the analysis program on any desktop PC for added efficiency and convenience.
Optical resolution	The optical resolution of the present instrument is ~6–7 microns. This is comparable to the resolution of the previous version, and can be modifed by changing the large-diameter objective.
The 2-MGEM2 is now in an enclosure box.	This makes the system neater, eliminates external light sources, and reduces the amount of dust that accumulates on the optics.

Details of the Upgrades

Software Development

The 2-MGEM2 operating and analysis software packages are now written in Visual Studio 2010 using Visual Basic.NET, which is compatible with the Windows 7, 64 bit operating system. The programs require a monitor with 1920x1080 resolution or better. Visual Studio 2010 is currently supported by Microsoft. There is a newer version now available (Visual Studio 2012), but this newer version does not offer any additional features that would be useful to the operation of the 2-MGEM2. The present software is entirely compatible with Visual Studio 2012, and could be easily upgraded.

The newer version of the 2-MGEM2 operating software has separate panels that operate the translators, rotators, photoelastic modulators, and the data acquisition card. Separate windows are written to examine the acquired waveform, calibrate the instrument, set up the XY scan, and acquire the 2-MGEM2 data. The photomultiplier tube voltage and current are continuously displayed in a separate panel, and other instrument and measurement data are also displayed in a separate panel when appropriate (this depends on the measurement being performed). See the operation manual (Attachment 1) for details.

One major data acquisition method improvement included in the new operating software is a revised technique to acquire the registration image of the particle array. This is usually performed at a reduced resolution and is used to select out regions of the sample to be examined at a higher resolution during set-up. The old technique would first move the examination point using the XY translators and then measure the intensity of light reflected from a small part of the sample (typically ~5 microns) using the measured PMT voltage. The electronic circuitry controlling the PMT is designed such that the PMT direct current is kept constant by dynamically changing the PMT voltage. In this configuration, more light results in a lower PMT voltage, and the reflected light intensity is proportional to $V^{-7.5}$. The new technique sets the Y-position of the translator, and then continuously scans the X-position, recording the PMT voltage continuously. The intensity is then determined using a spline fit of the PMT voltage data. This new technique eliminates the start and stop times of the translator controlling the X position, thereby speeding up the acquisition of the registration image. If the user desires, the older point-by-point registration technique can be selected. Additionally, the arrays of TRISO particles now used are hexagonal close-packed, reducing the total area of the sample that must be scanned, further reducing the time required for the registration measurement. Using the older system and technique, a registration scan could take ~1 to 1½ hours for a 20,000 to 30,000 point scan. The present system and acquisition technique measures ~12,000 points in ~12–15 minutes, resulting in an improvement of a factor of ~5–6.

A similar technique has also been employed to measure the 2-MGEM parameters of the TRISO particles. As with the registration measurement, the y-axis is first set and the x-axis position is set to the beginning of the scan. The translation stage is then continuously moved to the end x-axis position. During this move, measurements are continuously made of the PMT voltage and current (to measure the reflected light intensity), the position of the stage, and the 2-MGE waveform. After each waveform capture, the waveform is analyzed and the eight 2-MGE parameters are determined. These measured parameters are then grouped according to the set resolution for the x-scan, and the grouped parameters are then analyzed to determine the average and standard deviation of the group. The velocity of the x-scan is adjusted so that approximately n measurements are made in each pixel. Typically, 35–40 waveform captures and analyses can be done per second, so if the number of measurements per pixel is n=5, then ~7–8 pixels can be measured per second.

The data analysis package has also been rewritten, and is now separate from the program operating the 2-MGEM2 instrument. As a result, separate compiled versions of the analysis package can be placed on any PC operating under Windows 7, making data analysis much more convenient.

Hardware Development (ORNL and Hinds Instruments)

Several changes to the hardware have been incorporated into the new version of the 2-MGEM. This was necessary to make the instrument compatible with the newer software, and to replace older and now obsolete hardware. These new components include:

a) Computer. We have purchased a new HP elite 8200 computer using the Windows 7 (64 bit) operating system to run the 2-MGEM2. In addition, a new HP L2445 monitor has been purchased that has 1920x1200 pixel resolution.
b) Rotators and translators. The 2-MGEM2 requires two rotators to automatically set the positions of the polarization state generator (PSG, input) and the polarization state analyzer (PSA, output), and two translators to control the position of the sample. The older rotators have been replaced with Newport PR50CC rotators; the older Newport VP25XA translators were retained, because they are still supported by the vendor. All four automatic motion control instruments are controlled using Newport SMC100C controllers, which are daisy-chained to operate together, these are controlled from the computer using an RS-232 interface, and are incorporated into the Hinds electronics box.
c) Computer controlled data acquisition device. Eight (8) inputs/outputs are required to operate the 2-MGEM2:
   i. Two 0–5V digital-to-analog outputs to control the amplitudes of the two modulators,
   ii. Two inputs to measure the frequencies of the two modulators,
   iii. Two analog-to-digital inputs to measure the photomultiplier tube voltage and direct current,
   iv. A trigger input to trigger the waveform acquisition, and
   v. A high-speed analog-to-digital input at 2 MHz for waveform acquisition.

   All of these functions are now accomplished using a single National Instruments NI 6361 card, connected to the computer via the PCIe bus. The connection box for the NI 6361 card in incorporated into the Hinds Instruments electronics package.

d) Light Source. While the light source is still a mercury arc lamp in an Oriel/Newport Series Q housing, the light is now channeled to the input collimating microscope objective using a 400 micron to 100 micron optical fiber. Additionally, the original interference filter has been replaced with another interference filter. Both of these improvements have resulted in a factor of 2 more light reaching the detector head.
e) Detector head. After the light passes through the PSA, it is imaged using a CCD camera, and simultaneously detected using the PMT to obtain the intensity waveform that is then analyzed to

determine the diattenuation. This has been redesigned using a new ThorLabs DCC1240M monochrome CMOS camera, where the beam is split off using a pellicle beam splitter. The improved detector head, along with the improved light source, has resulted in nearly a factor of 10 more light reaching the PMT detector and CCD camera.

f) Enclosure box. A new enclosure box has been constructed using ThorLabs extrusion hardware and black panels. While the improvement from darkening of the sample during measurement is expected to be minor, this enclosure will primarily help in keeping dust and air-borne contaminants off the optics.

g) Hinds Photoelastic modulators. The older version of the 2-MGEM utilized older photoelastic modulators and the PEM-90 control boxes to control the photoelastic modulators. The revised 2-MGEM2 now includes the latest photoelastic modulators that have automatic temperature control, as well as newer controllers, which are included in the Hinds electronics box. The newer modulators exhibit less drift than the previous version.

h) Hinds electronics enclosure. All of the electronics for the 2-MGEM2 are now incorporated into a single electronics enclosure. This box has two connections to the computer: 1) An RS-232 cable from the serial port of the computer to the Hinds enclosure, and 2) the connection cable from the NI 6361 PCIe card to the Hinds enclosure. The RS-232 interface allows the computer to communicate directly with the Newport SMC100CC controllers used to interact with the two rotators and the two xy translators, and the NI 6361 card allows the computer to set and read various voltages and frequencies used in the 2-MGEM2. The enclosure also includes the two boards used to control the PEMs, and a control circuit for the PMT. The PMT control circuit is designed to dynamically change the voltage on the PMT dynode chain depending on the light level on the PMT. If the light level decreases, then the PMT circuit will increase the dynode chain voltage (thereby increasing the PMT gain) to keep the current coming from the PMT constant.

The PMT control circuit board included in the Hinds electronic enclosure has just recently been built and works, but we are still testing the board to determine its specifications. As a result, the tests described below were carried out using our older PMT control circuit.

Testing of the Upgraded 2-MGEM2

Optical Resolution of the 2-MGEM2

As noted in the 2-MGEM2 Operation Manual (Attachment 1), the 2-MGEM2 is focused by observing the image on the CCD camera and adjusting the sample height by raising or lowering the sample stage. While this focuses the sample image onto the CCD camera, it is also critical that this focal plane be in conjunction with the focal plane for the pinhole in front of the PMT. To obtain this condition, images of a 1951 United States Air Force (USAF) resolution target were taken using the XY scan setup program normally used for determining the registration of the sample. The position of the pinhole was raised or lowered until the best image was obtained. Figure 1 shows that, at the best imaging conditions for the optics in the system, the 1951 USAF target element 6-3 (group 6, element 3) could just be resolved using the PMT (indicating a resolution of ~6.2 microns). For comparison, the best resolution obtained with the CCD camera was ~4.9 microns (element 6-5). The primary limiting factor for PMT resolution is the pinhole diameter (currently 50 microns). While decreasing the pinhole size would result in some improvement in resolution, the improvement would not be significant, compared to the increase in signal to noise. To further improve the PMT resolution effectively, it is necessary to replace the large-diameter objective with a smaller focal length objective; this was done for the work described in ref. 7.

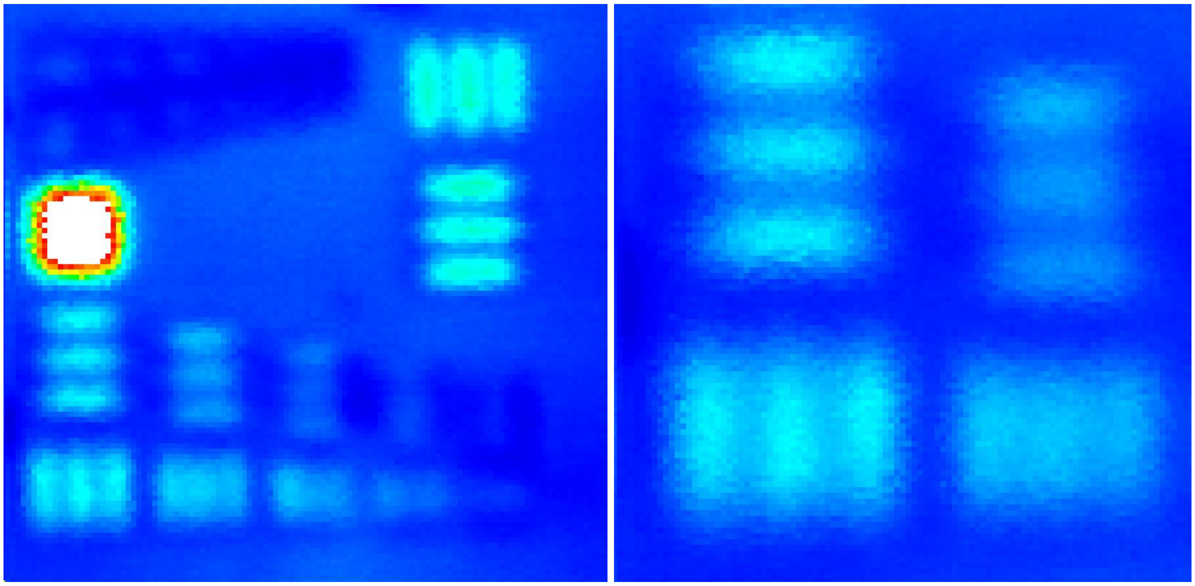

Figure 1. Optical images of a 1951 USAF resolution target. The image to the left (taken using a step size of 2 μm) shows group 6 in the central part of the 1951 USAF target. The image to the right shows elements 6-2 and 6-3 (~7.0 and ~6.2 μm resolution, respectively), also located in the lower left of the other image.

Comparison of 2-MGEM2 Results Using Different Data Collection Techniques

Since several data collection techniques are now available with the 2-MGEM2, it is useful to quantitatively compare both the accuracy of the results as well as the time performance. To do this, the central particle of the sample mount M11110801 was measured (M11110801 contained surrogate TRISO particles with a zirconia kernel). Comparison measurements were performed on just the central particle using three different techniques: 1) Continuous Scan, 2) Point-by-point 1 (5 measurements and 1 trace), and 3) Point-by-point 2 (4 measurements and 3 traces). The number of traces is the number of waveforms that are signal averaged before the waveform is analyzed to determine the 2-MGE parameters. The signal averaged waveforms are then analyzed to determine the eight 2-MGE parameters for each measurement. (See the 2-MGEM2 Operation Manual (Attachment 1), for more details.) Since the same XY Scan setup was used, the total number of pixels (31,840) was the same for all three measurements.

The resulting images of the diattenuation (Figure 2) show that visually, there is no significant difference between the three measurements. All three images show that the diattenuation of the IPyC is significant, and that there are regions within the IPyC where the diattenuation is considerable. Moreover, the diattenuation of the OPyC is very small.

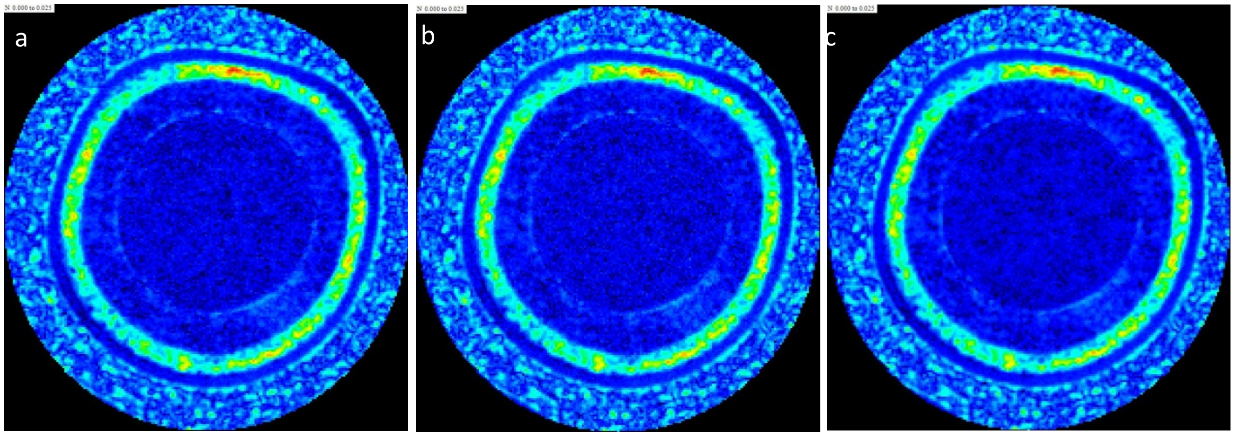

Figure 2. Diattenuation images of the central particle of M11110801. a) Continuous Scan. b) Point-by-Point 1 (5 measurements, 1 trace). c) Point-by-Point 2 (4 measurements, 3 traces).

The three data sets were analyzed using the standard method described in the 2-MGEM2 Data Analysis Manual (Attachment 2), and the results are summarized in Table 2. All three data sets result in insignificant differences in the average diattenuation N for the IPyC, the standard deviation (Std. Dev.) and the relative direction of the fast axis. A similar statement can be made for the results shown for the OPyC, although the diattenuation of the OPyC is considerably less than the diattenuation of the IPyC. While the average error of the Continuous Scan measurement is somewhat higher than the other two techniques (0.0011 versus 0.0010 and 0.0007), it is clearly less than the standard deviation. This, coupled with an examination of the diattenuation images shown in Figure 2, results in a conclusion that the variation in diattenuation is a function of the material and not a result of stochastic fluctuation.

Table 2. Comparison of the different data collection techniques with the 2-MGEM2

	Continuous Scan	**Point-by-Point 1**	**Point-by-Point 2**
# Measurements	5	5	4
# Traces	---	1	3
IPyC diattenuation N	0.0132	0.0132	0.0132
Std. Dev.	0.0036	0.0036	0.0035
Ave. Err.	0.0011	0.0010	0.0007
Rel. Fast Axis ± Std. Dev.	7.6°±2.8°	7.7°±2.5°	7.5°±1.6°
OPyC diattenuation N	0.0053	0.0053	0.0051
Std. Dev.	0.0028	0.0028	0.0027
Ave. Err.	0.0012	0.0011	0.0008
Rel. Fast Axis ± Std. Dev.	43.8°±10.5°	42.5°±9.2°	43.0°±7.6°
Measurement time (minutes)	71	99	139
Average points per second	7.45	5.36	3.83

While the average error of the IPyC diattenuation is somewhat higher for the Continuous Scan method of data collection, the time required to perform a measurement is considerably less. For the test shown in Table 2, the average error of the Continuous Scan method was nearly the same as that for the Point-by-Point 1 technique, but the time required to complete the measurement was nearly 30% less. The

Point-by-Point 2 technique resulted in lower error in the IPyC diattenuation, but it took nearly twice as long to perform the measurement.

Comparison of 2-MGEM2 Results to Measurements with the Previous Version

Sample mount M11110801 was also used to compare results from the second generation 2-MGEM2 to data obtained with the previous version of the 2-MGEM. This sample was one of the last to be measured reliably before the upgrade. This sample was a close-packed hexagonal grid of particles encapsulated in a new, harder epoxy (thermosetting Struers Isofast replacing thermoplastic Struers Specifast), and polished with a new cloth (Struers Plan replacing Struers Dac). The new materialography procedure was designed to allow for a better polish while maintaining flatness. The result of the new method was reduced relief in the OPyC layer and fewer scratches or pits in the polished surface. While the diattenuation of the OPyC of M11110801 is unusually low, the diattenuation of the IPyC is significant and can be used for a valid comparison.

Table 3 shows how the data compares. The older measurements were taken with the previous version of the 2-MGEM and analyzed with both the old 2-MGEM analysis software and the new 2-MGEM2 analysis software. The newer measurements were taken with the second generation 2-MGEM2 and analyzed with the new 2-MGEM2 software. The absolute value of the difference between the new and old analysis of the older data is shown, as well as the absolute value of the difference between the newer and older data, both analyzed using the new 2-MGEM2 software.

Table 3. Comparison of the average IPyC diattenuation determined for seven particles in M11110801

	Older Data (OD)			Newer Data (ND)	
	Old Analysis (OA)	New Analysis (NA)	Difference |NA-OA|	New Analysis (NA)	Difference |ND-OD|
Particle 1	0.0135[a,b]	0.0135	0.0000	0.0132[c]	0.0003
Particle 2	0.0150[b]	0.0144	0.0006	0.0142[d]	0.0002
Particle 3	0.0137[a]	0.0134	0.0003	0.0137[d]	0.0003
Particle 4	0.0139[a]	0.0140	0.0001	0.0141[d]	0.0001
Particle 5	0.0138[a]	0.0133	0.0005	0.0136[d]	0.0003
Particle 6	0.0145[a]	0.0143	0.0002	0.0143[d]	0.0000
Particle 7	0.0145[b]	0.0145	0.0000	0.0144[d]	0.0001
Average	0.0141	0.0139	0.0002	0.0139	0.0002
Ave. Std. Dev.	0.0037	0.0037		0.0034	
Ave. Error	0.0006	0.0006		0.0011	
Average measurement time (minutes)	215			66	

[a] Measurements taken on 12/27/2011
[b] Measurements taken on 1/5/2012
[c] Measurements taken on 4/5/2013
[d] Measurements taken on 4/8/2013

The previous version of the 2-MGEM that was used to take the old measurements was having severe problems at the time, in that the PEMs would turn off during the measurement, or the computer would hang up requiring a reboot. Consequently, measurements of 7 particles were often not completed in a single measurement session, but rather required multiple sessions to collect the required data. For the case of these measurements, Particles 1, 3, 4, 5, and 6 were completed on 12/27/2011, but the instrument failed to complete the entire series of measurements. Consequently, the additional measurements were completed on 1/5/2012 (see Table 3).The new measurements were taken using the new 2-MGEM2 Continuous Scan option on April 5 and 8, 2013.

In Table 3, the average value of the IPyC diattenuation for the older data is nearly the same for both versions of analysis software. The observed difference is likely due to variation in selected regions; the new analysis was performed on a wider donut shaped region (0.015 versus 0.010, see Attachment 2). Similarly, a comparison of the older and newer data, both analyzed using the new 2-MGEM2 analysis software yielded an average difference of only 0.0002. The observed differences are less than the average error, and it can be concluded that both systems measure comparable values of the IPyC diattenuation.

It is also important to consider the standard deviation over the analyzed region of the IPyC layer. As discussed in the 2-MGEM2 data Analysis Manual (Attachment 2), if the standard deviation is considerably larger than the average error, then the standard deviation is due to the sample itself and not just stochastic error. As can be seen from Table 3, the average standard deviation of the older data was 0.0037, analyzed with either the old analysis program or the new analysis program, and the average standard deviation of the newer data was 0.0034. Since both of these values are larger than the average error, this indicates that the systems are measuring a real variation of the diattenuation within the IPyC layer. This variation can be seen in Figure 3 for multiple measurements made on the same particle, and the local variation in the diattenuation compares well in all images.

Finally, it can be seen in Table 3 that, while the average error of the newer data was somewhat larger than the average error obtained with the older 2-MGEM, the time required for the 2-MGEM2 measurement was about 31% of the time required for the 2-MGEM measurement. In both cases, the magnitude of the average error is acceptable, considering the local variation, so the improvement in analysis time is beneficial.

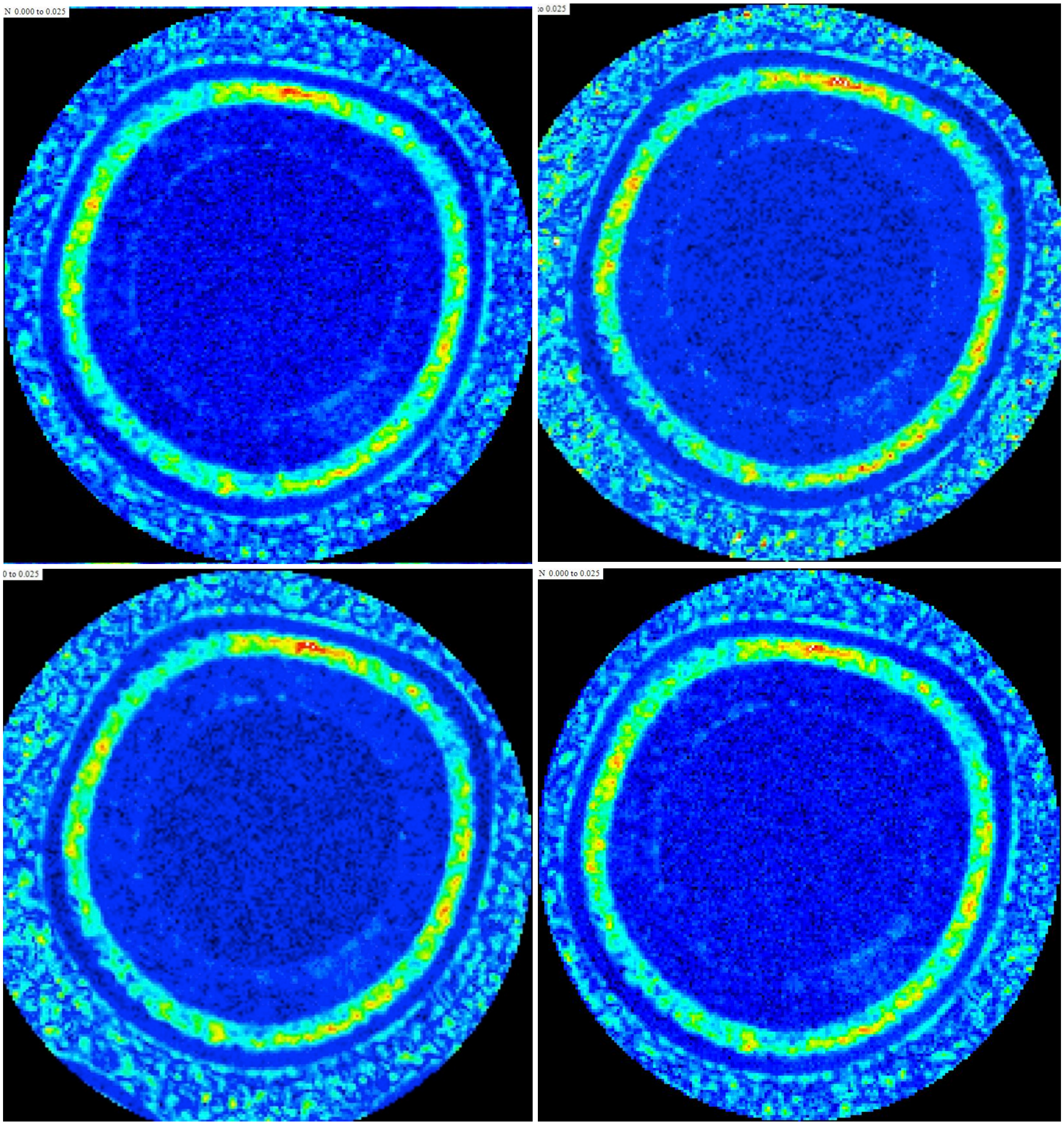

Figure 3. Diattenuation images of the central particle of M11110801 taken on several different days using the old 2-MGEM system (top) and the new 2-MGEM2 system (bottom). The two data sets shown at the top were taken on 12/27/2011 (upper left) and 1/5/2012 (upper right). The bottom data sets were taken using the Continuous Scan (lower left) and the Point-by-Point 1 (lower right) techniques.

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2-MGEM2 Operation Manual

Operating Instructions

The two-modulator generalized ellipsometry microscope (2-MGEM2) is the 2nd generation of an instrument that measures generalized ellipsometry parameters for near-normal incidence light. The 2-MGEM2 uses two photoelastic modulator (PEM) polarizer pairs, operating at different resonant frequencies (50 kHz and 60 kHz). The 50 kHz PEM-polarizer is placed on the input arm, and acts as the polarization state generator (PSG), while the 60 kHz PEM-polarizer pair is placed before the detector and acts as the polarization state analyzer (PSA). The optical operation of the 2-MGEM2 is similar to the previous two modulator generalized ellipsometer (2-MGE), in that 8 elements of the sample Mueller matrix are measured simultaneously. If the angles of the PSG and PSA are set at 0° and 45°, then one can determine the diattenuation N, the retardation δ, the direction of the fast axis γ, the circular diattenuation CD, and the depolarization $1-\beta$ with a single measurement. The primary difference between the 2-MGEM2 and the 2-MGE is that the 2-MGEM2 is designed as a microscope at near-normal incidence, where a very small part of the sample is examined at any one time. Maps of the sample are then obtained by scanning the sample and taking 2-MGEM2 measurements at each selected point on the sample surface. The typical optical resolution of the instrument is ~6-7 microns, but this can be improved using different optical elements.

4/22/2013

Table of Contents

Acronyms

2-MGE	Two-modulator generalized ellipsometer
2-MGEM2	Two- modulator generalized ellipsometry microscope, revision 2
ATC	Automatic temperature control
CCD	Charge-coupled device
DAC	Digital to analog converter
DAQ	Data acquisition
NI	National Instruments
PCIe	Peripheral component interconnect express
PEM	Photoelastic modulator
PSA	Polarization state analyzer
PSG	Polarization state generator
PMT	Photomultiplier tube
SWL	Single wavelength
USAF	United States Air Force

Introduction

This manual describes the operation of the two-modulator generalized ellipsometry microscope, revision 2 (2-MGEM2). Figure 1 shows a schematic of the new instrument, which has been significantly updated. These updates include migration to a modern operating system and computer programing language (Windows 7 and Visual Studio 2010), and many of the components have been replaced with currently supported components, making the instrument more robust and easier to service. Improvements have been made in the optics to improve light collection at the detector, where the improvement is about a factor of 20. Additional improvements in the algorithms used to perform the measurements have resulted in nearly a factor of four reduction in measurement time over the previous version.

The first section of this manual gives a brief description of the instrument, summarized from ref. 1. The second section deals with the operational instructions for the software running the 2-MGEM2. The instrument is mostly computer-controlled, so the software is essential to the operation of the instrument. The third section discusses operational instructions for the hardware that is not computer controlled. The theory of operation of the 2-MGEM2 is discussed primarily in the references, but a summary relating directly to this version of the 2-MGEM2 is discussed in the appendices.

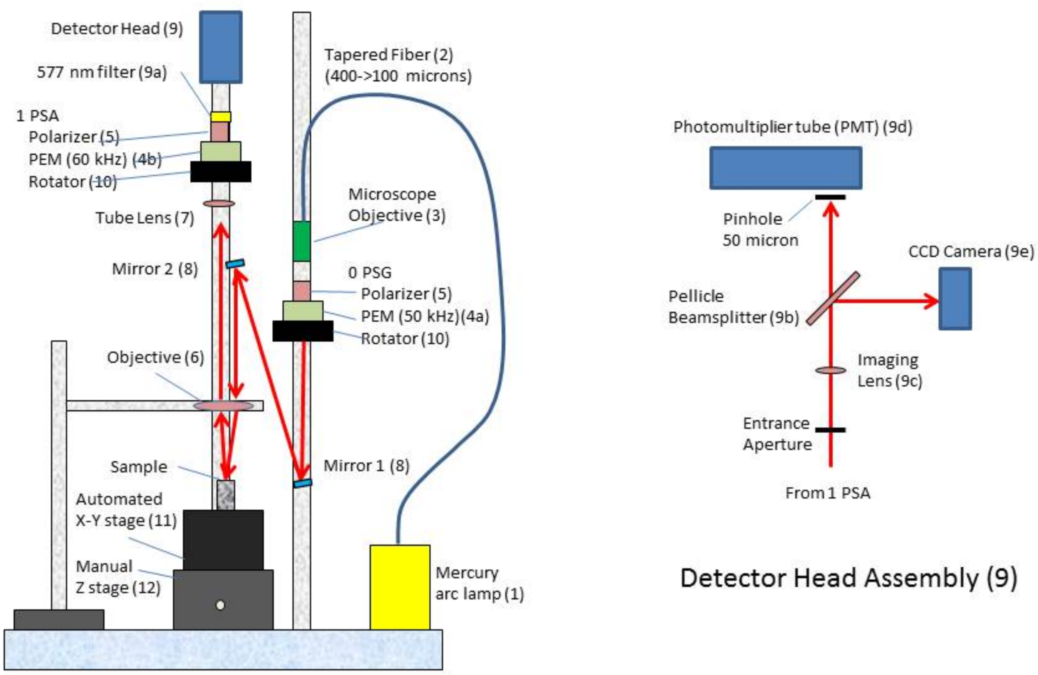

Figure 1. Schematic diagram of the new 2-MGEM2 (left) with a schematic of the detector head assembly (right). The numbers in parentheses refer to components listed in the equipment list.

Description of the 2-MGEM2

This section provides an updated summary to that appearing in ref. 1 for the previous 2-MGEM. The reflection two-modulator generalized ellipsometry microscope (Figure 1) is very similar to the reflection two-modulator generalized ellipsometer (2-MGE) described in refs. 2–4 and the transmission 2-MGE described in ref. 5. The instrument is illuminated with quasi-collimated light originating from a mercury arc lamp imaged onto a tapered fiber with a 400-micron-diameter core transitioning to a 100-micron-diameter core. The light is transmitted through the fiber exiting the 100-micron-diameter end at the focal point of a microscope objective, resulting in the light emerging from the entrance aperture of the objective being nearly collimated with a diameter of 3–5 mm. The collimating microscope objective is a 10X objective with a working distance of ~1 cm. By finely adjusting the position of the optical fiber with respect to the focal point of the microscope objective, the emerging light can be focused at large or small distances, with the diameter of the focused light spot depending on the core diameter of the fiber near the microscope objective (a larger core diameter results in a larger focused spot).

The collimated light first passes through the polarization state generator (PSG), which is mounted on an automated rotator. The PSG consists of a polarizer attached to a photoelastic modulator (PEM), which generates dynamically elliptically polarized light oscillating at the resonant frequency of the PEM (~50 kHz). The polarizer is attached to the PEM using a manual rotator, which allows the user to adjust the azimuthal angle of the polarizer with respect to the PEM (θ_b—normally set to +45°). The polarizer-PEM pair is attached to an automated rotator so that different incoming polarization states can be incident onto the sample.

The light emerging from the PSG is reflected from directing mirrors 1 and 2. Generally, reflections from mirrors are avoided in polarization measurements, since this reflection can change the polarization state of the light beam and introduce a systematic error into the measurement. However, if the light beam is at normal incidence to an isotropic reflector, then there is a small reduction in the beam intensity (~10%) but no perturbation to the polarization state (other than a possible change of sign). If the angle of incidence is small, there will be some contribution to the change in the polarization state, but the change will be small (ref. 1).

The two directing mirrors are placed in standard kinematic mirror mounts so that the direction of the reflected light beam can be adjusted. Generally speaking, one would like to have the light beam a constant distance from the supporting poles shown in Figure 1. The light reflecting from the second directing mirror is directed to a large diameter, long-working-distance objective (Leica model 10411597 with 100-mm focal length), which focuses the light beam onto the sample. Since the incoming light beam is nearly collimated, the distance from the sample surface to the large diameter objective will be close to the focal length of this objective. The large-diameter objective acts as a condenser that increases the light intensity per unit area by about a factor of 50. This light is then specularly reflected from the sample and passes through the objective again, transforming the diverging light beam coming from the sample back into a quasi-collimated beam. It is generally best if the light beam entering the large diameter objective is parallel to the outgoing beam, and if the center of the large diameter objective is between the two beams; this insures that the refraction of the light beam is the same for the entering and exiting beams.

Under these conditions, the light beam incident onto the sample is near normal incidence. Typically, the separation of the incoming and outgoing beams is <10 mm, which results in an angle of incidence of ~3° for a 100-mm working distance.

The most significant systematic error in the 2-MGEM2 measurement comes from the perturbations of the light beam polarization state by the large-diameter objective. Apart from the normal stress-induced birefringence of the optical elements in this objective, the light is not passing along the axis of the objective, which can introduce a significant change in the polarization state. This will be discussed below.

If the system is properly adjusted, the light beam exiting from the large diameter objective should be collimated. This beam is then directed to a 200-mm focal length tube lens, which focuses the light onto the entrance aperture of the detector (Figure 1, right), with a magnification of $f_{tube}/f_{objective} = 2$. The tube lens will also introduce a small systematic error, but this perturbation is significantly less than that from the large diameter objective, since the light beam passes along the axis of the lens.

The polarization state analyzer (PSA) consists of another PEM-polarizer pair, placed between the tube lens and the detector. The PSA PEM operates at a resonant frequency of 60 kHz, which is different from the frequency of the PSG PEM (50 kHz). As with the PSG PEM-polarizer pair, the polarizer is oriented at +45° with respect to the PEM, and the PSA is also mounted on an automated rotator so that different polarization states coming from the sample may be analyzed.

The detector assembly (Figure 1, right) includes a lens that images the aperture onto both a charge-coupled device (CCD) and the entrance pinhole of the photomultiplier tube (PMT). The CCD (Thor Labs model DCC1240M) is a monochromatic camera connected to the computer using a universal serial bus (USB), and is used for examination of the sample image and for fine focusing, while the 2-MGEM2 signal is obtained by digitizing the intensity waveform from the PMT. An achromatic lens with 30-mm focal length is positioned to image the entrance aperture onto the CCD camera and onto the pinhole in front of the PMT with a resulting magnification of ~3:1. Small focusing adjustments can be made by changing the axial position of either the aperture and/or the imaging lens. Since all the detector head assembly optics shown in Figure 1 are placed after the last polarizer, changes in the polarization state from these optics do not perturb the measurement. This is because the emerging light from the PSA is linearly polarized from the last polarizing prism and the waveform analysis of the 2-MGEM2 (see below) depends only on the ratio of the intensity of various frequency components to the average intensity.

As can be seen from Figure 1, the sample is placed on a 5-axis sample stage. The vertical axis manual adjustment is used to position the sample in the focal plane of the instrument, thereby imaging the sample surface onto the CCD camera and the PMT pinhole. The sample can be moved using the automated xy linear translation stage, and the sample can be tilted with the manual tilt stage (not shown). Using the described set of optics, the magnification of the system is roughly 2 × 3 = 6. The resolution is also limited to first order by the size of the aperture before the PMT; using the 50 micron pinhole, the resolution should be ~50/6 or ~8 microns. The resolution was evaluated using a 1951

United States Air Force (USAF) resolution target, and found to be ~7 microns. As presently configured, the 2-MGEM2 is a serial system, where xy images are made by moving the sample via the automated xy stage and taking 2-MGEM2 data at each point.

The 2-MGEM2 must operate at a single wavelength. This is obtained by using a mercury arc lamp, where the 577-nm line is selected using a Semrock FF01-576/10-25 narrow-pass interference filter placed in front of the detector head assembly (Figure 1).

Operational Instructions: Software

This part of the instructions deals with the operation of the computer program designed to run the 2-MGEM2. Refer to Appendix A and to the references for a more detailed description of the theory and operation of the instrument.

In this part of the instructions, the word “panel” will refer to the narrow side windows that are used to control specific instruments, located to the left of the screen, and to similar windows containing instrumental data, located to the right of the screen. These panels are used in several parts of the program. The word “window” will refer to the window placed at the center of the screen, which is used to perform specific functions. The entire monitor area is referred to as the “screen.” Whenever a push button, label, text box, etc. is indicated, its name will be in italics. For all peripheral panels, the “Hide” button to the lower right of the panel will hide the panel. However, it still will be active, so that when it is re-activated, the values in the panel will still be valid.

Opening Screen

Upon the initialization of the 2-MGEM2 program, the opening screen (not shown) will be presented, which will give access to the other windows of the program. These additional windows are accessed by the push buttons located on the right (*Measure 2-MGEM2*, *Calibrate Swl*, *XY-Translation Setup*, and *Waveform Analysis*).

All of the individual instruments can be operated directly from the opening window. These are activated by pressing the *Instruments…* push button, which will expose the group box *Select Inst* above the *Instruments…* push button. There are four different panels that are used to operate the various instruments associated with the 2-MGEM2. When the appropriate button is pressed, a panel will appear on the left of the screen. In most of the windows described below, many of these instrument panels are also available, and they will all appear, one at a time, at the left of the screen. In addition, the user can select two different *User Levels*: *User* or *Engineer*. The *Engineer* level will allow the user access to more features of the program that interact directly with the instrument, while the *User* level will allow the user to perform all the tasks required to perform 2-MGEM2 measurements on TRISO particles.

Peripheral Panels

The program contains several panels that are used in more than one of the main parts of the program. These common panels can be used to control individual instruments of the 2-MGEM2 or to display the 2-MGEM2 data. The common panels for the instruments will appear to the left of the screen, while the displayed data will be shown to the right of the screen. When the opening screen is finished initializing, the panel displaying the PMT operating voltage and current will be displayed. This panel will be refreshed every 0.5 s, allowing the operator to monitor the light level incident on the PMT detector.

PEMs and PMT

The panel shown in Figure 2 allows control of the photoelastic modulators used to control the polarization state of the polarization state generator (*0 PSG*) and the polarization state analyzer (*1 PSA*), as well as control of the photomultiplier tube. When the panel is opened, the present values of the modulator amplitudes and the control voltages are shown by the *Ampl.* and *Volt* labels, respectively. The voltages are supplied by a digital to analog converter (DAC), which can be accessed by the *Digitizer/DAQ* button described in the next section. Each modulation amplitude is shown in radians (*Rad.*), with equivalent values shown in degrees (*Deg.*) and waves (*Wave* = radians/2π). The amplitudes are converted to the expected voltage using the most recently defined calibration constants. The value of the wavelength of light is also used to determine the voltage, this is saved in the calibration file and should not be changed, except on the rare occasion that the wavelength selection filter in the instrument is changed.

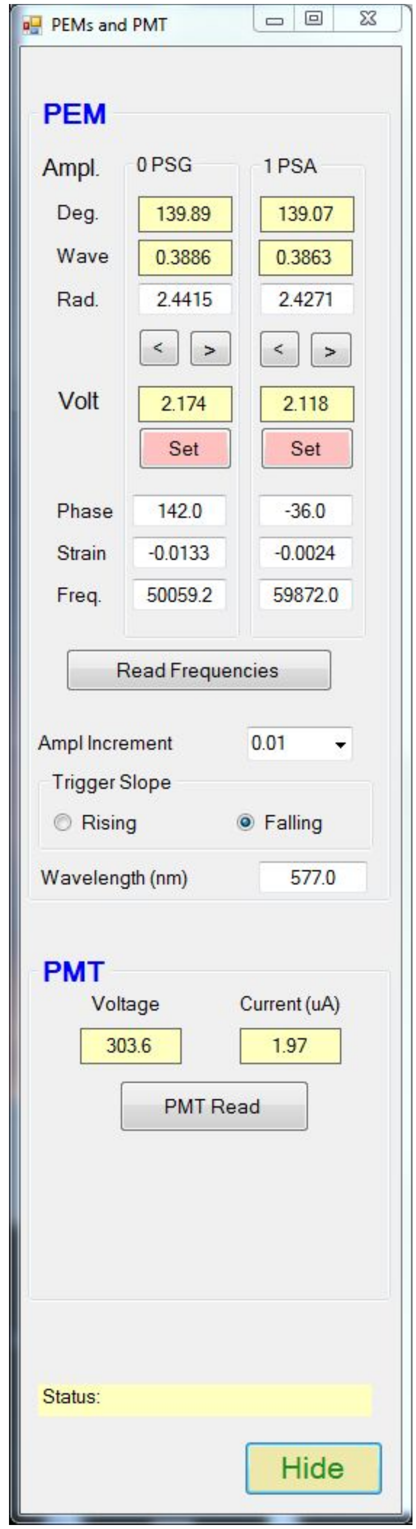

Figure 2. PEMs and PMT control panel.

The amplitude can be set in two ways:

a) The amplitude can be incremented (in radians) by pressing the < and > buttons beneath the displayed value for the desired modulator. This will increment the amplitude by the amount shown beside *Ampl Increment.* The amplitude increment can be changed using the pull-down selection tool.

b) The amplitude can also be changed by selecting the amplitude box, entering an amplitude (in radians), and then pressing *Set*.

There are three other characteristics of the modulators that affect the operation of the PEMs, and should only be changed by the engineer in charge of the 2-MGEM2. The values are stored on the calibration files, but can be changed by clicking on the text box and entering the correct number.

a) *Phase*: This is the actual optical phase of the PEM with respect to the electrical signal generated by the PEM controller.

b) *Strain*: This is the static strain of the PEM in radians. The strain will be updated whenever the calibration routine is run.

c) *Freq.*: (Frequency): This is the actual frequency of the PEM in Hz. The frequency will be updated whenever the calibration routine is run, as well as during the measurements. Additionally, the operator can initialize a frequency measurement by pressing the *Read Frequencies* button below. The frequencies are read using the DAQ card, described below.

The *Trigger slope* refers to the output of the triggering circuit, which puts out a trigger pulse whenever the phases of both modulators are in sync. The trigger pulse can be generated either by the positive-going or negative-going part of the waveform from the PEM, which is selected on this panel. This is saved in the calibration file, and should not be changed except by the maintenance engineer.

The *PMT* group box shows the current value of the PMT *Voltage* and PMT *Current*, and this can be updated by pressing the *PMT Read* button. On some instruments, it is possible to set these parameters by the computer. If this is the case, then there will also be a “Set” group box, allowing the user to set these values.

Digitizer/DAQ

The Digitizer/Data Acquisition (*Digitizer/DAQ*) panel (Figure 3) allows the user to access parameters that control the waveform capture utilizing the digitizer and other data acquisition functions of the card. The present DAQ card that is used is the National Instruments NI 6361 card utilizing the PCIe interface.

The *Setup* box shows 4 different parameters, each accessible using a pull-down menu. These are:

a) *# Samples*: The number of samples measured by the digitizer. This is restricted to factors of 2, as reflected in the pull-down menu.
b) *Sample Rate (MS/s)*: This is the number of mega samples collected per second. The time between samples is the inverse, so a 2 MS/s sample rate is equivalent to a sample time of 0.5 microseconds.
c) *# Measurements*: The number of measurements collected at each data point. A measurement consists of collecting N waveforms that are signal-averaged, and then performing the data analysis on the signal averaged waveform, which will determine the 8 parameters measured by the 2-MGEM2. If there is more than one measurement at each data point, then the estimated error is also determined.
d) *# Waveforms SA* (traces): The number of waveforms captured and signal-averaged before the data analysis is performed.

Figure 3. Digitizer and DAQ control panel.

The *Digitizer Specs* box shows the details of the digitizer used. These cannot be changed by the user. These include

a) *# bits*: The number of bits of the digitizer
b) Volts full scale
c) *Polarity*: Unipolar or Bipolar

The *Waveform Analysis Technique* pull-down menu shows the various types of analyses that can be performed on the waveform at the completion of the measurement cycle. These will be described in more detail below, but generally refer to the number of waveform parameters that are calculated from the captured waveform, which will then be converted into the eight 2-MGEM2 parameters. The default for this selection is

12 measured frequencies, shown as *T2_12parasSD*. This is restricted to the maintenance engineer.

The *Waveform apodization* pull-down menu shows the various types of window functions that can be applied to the signal waveform before any analysis is performed on the waveform to obtain the eight 2-MGE parameters. Generally speaking, the *Rectangular* window, where there is no apodization, gives the best results, and this is the default. This is restricted to the maintenance engineer.

The *Digitize…* button allows the engineer to perform a single digitization and the program records the time that it took to perform this operation. This is restricted to the maintenance engineer.

The *DAQ* box also allows the user to set and read voltages, as well as perform the frequency reads on either PEM. These interact directly with the DAQ card, and are not set specifically to any element of the 2-MGEM2 instrument. For the case of a voltage read, one can access any of the 8 channels available on the DAQ card, although most of these will not be connected and will give unreliable results. This is restricted to the maintenance engineer.

Rotators

The Rotators panel (Figure 4) controls the rotation stages attached to each PEM: *0 PSG* for the polarization state generator and *1 PSA* for the polarization state analyzer. The angles of rotation can be set either by selecting the appropriate radio button (in increments of 22.5°), or by jogging the stage, where the jog increment is selected by the pull-down menu. During motion, the present position of the rotators is updated every 0.5 s in the yellow boxes below the radio buttons. The *Angle Offset* is determined during a calibration run, but can also be changed by the user by clicking and entering in the appropriate text box. If the user wishes to NOT use the angle offset, then uncheck the *On* checkbox. The rotators can both be homed (Set both the 0 PSG and 1 PSA to 0°) by pressing the *Home* button. However, the rotators currently used cannot set a home position, so this option is not available. The *All Stop* button will stop the motion of both rotators instantly.

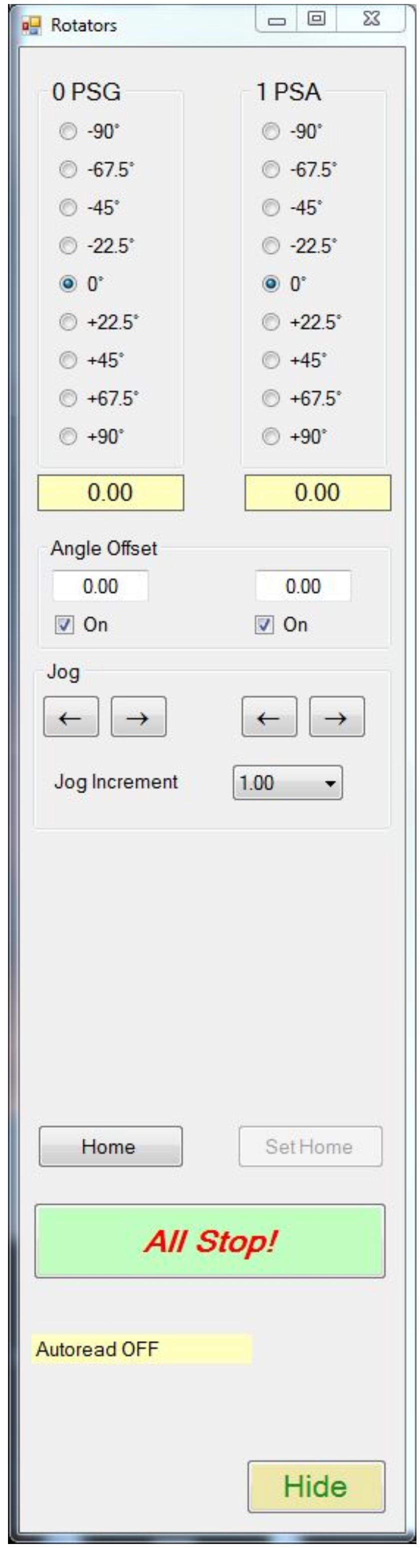

Figure 4. Rotators control panel.

Translators

Two (or three) axes can be controlled using the Translators panel (Figure 5). The x- and y-axes control the position of the xy translation stages that change the position of the spot imaged onto the detector, used in the measurement of the 2-MGEM2 map of the sample. The z-axis is used to control the height of the stage, thus imaging the sample onto the CCD camera and the pinhole in front of the PMT. Depending on the system, this axis control can be manual or automatic; if the system is automatic, then a z-axis control will also be shown on this panel. The present *Position* of each axis is shown in the green label box and the *Target* position is shown in the text box. The *Home* button can be pressed to return each stage to 0. The positions of the two (or three) stages can be changed by either entering the desired position in the target box, or by using the jog buttons. Each press of a jog button will move the appropriate axis the number of millimeters shown in the *Jog Increment* box.

For some systems, the axes cannot be moved manually while the motors are activated. To deactivate the motors, press the *Off* radio button in the *Motors* group box.

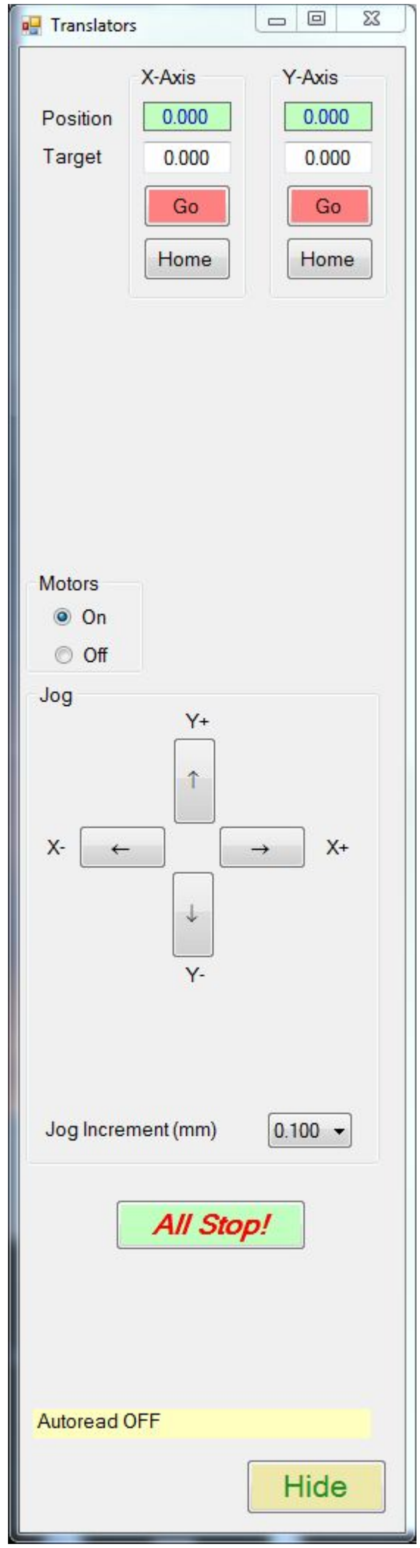

Figure 5. Translators control panel.

Instrument Data and PMT Display

The panels shown in Figure 6 appear to the right of the screen, and show a summary of all the important data being taken using the 2-MGEM2. The upper panel shows the present value of the PMT voltage and current, and is normally updated every 0.5 seconds. This panel is displayed for all measurements. The normal updating is suspended when the instrument is performing a continuous scan.

The *Instrument Status* panel shows the various parameters of the digitizer and the PEMs, as well as the present values of the 2-MGEM2 parameters. The *Digitizer* parameters are read-only, and are not continuously updated, but the PEM Frequency (*Freq.*) data will occasionally get updated as it changes through a measurement. The present values of the 2-MGE parameters are shown, along with the calculated stochastic error if the number of measurements is greater than 1. Either the raw 2-MGE parameters (*Raw*) or the parameters corrected for instrumental systematic offsets (*Corr.*) are displayed. Normally, the 2-MGE parameters are continuously updated as new measurements are made. However, these parameters are not updated during a continuous scan measurement.

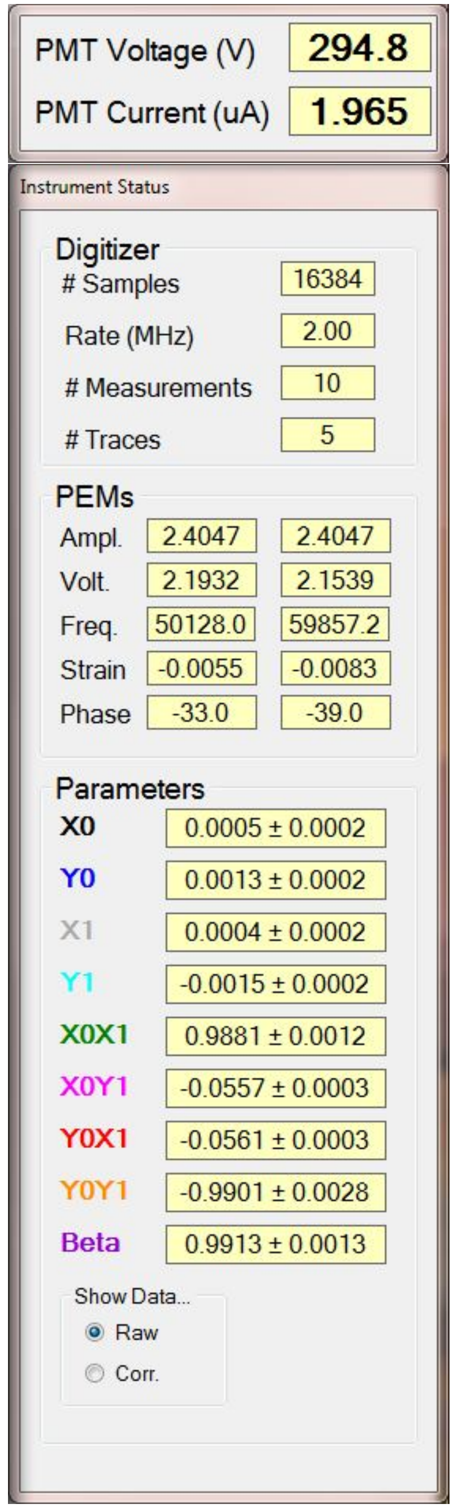

Figure 6. Data for the instruments used in 2-MGEM2, shown at the right of the screen.

XY Scan Setup

Pressing the *XY Scan Setup* button on the opening screen brings up the window in Figure 7 used to perform a registration scan of the sample and to select the regions that will be measured using a finer scan. When this window is activated, the translator panel is automatically placed to the left of the window. The purpose of this window is to measure the intensity of the light reflecting from the sample as a function of the xy position of the sample. This is usually done at a lesser resolution than the actual measurements, and will therefore not take as long. The coordinates of the intensity scan are determined using the parameters in the group box labeled *Intensity Scan Parameters* in the upper right of the window. Here, the user enters the minimum and maximum values for the x- and y-axes, as well as the increment, all in millimeters. The total number of points is shown dynamically in the yellow box.

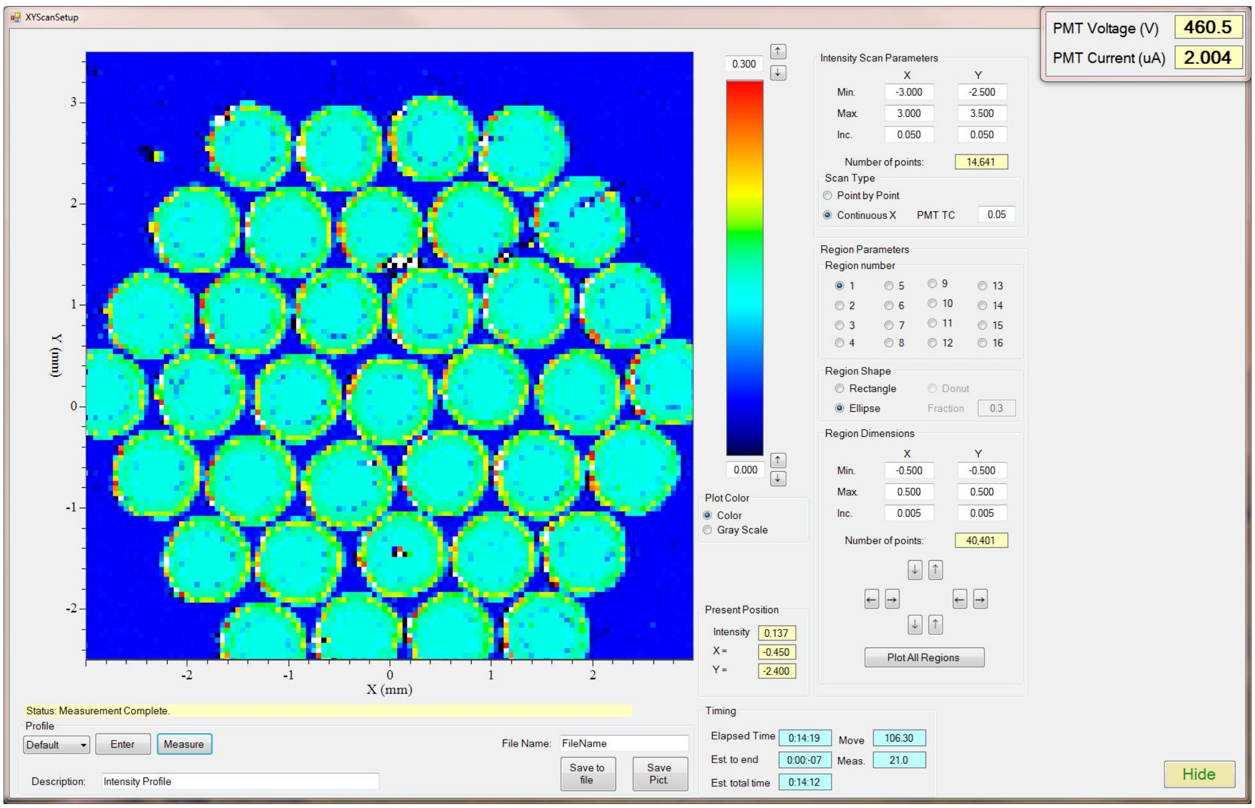

Figure 7. The control panel for performing the XY scan setup.

The intensity of the reflected light is determined by measuring the current and voltage of the PMT; this intensity is proportional to $V^{-7.5}$ at constant PMT current, where V is the PMT voltage. The recorded intensity is the ratio of the measured current divided by the intensity as determined during the most recent calibration scan, discussed below. The intensity scan can be performed in two ways:

a) *Point-by-Point*. With this scan, the positions of the xy stages are first set (first y, then x), the translators stopped, and then the intensity of the present position of the sample is measured by recording the current and voltage on the PMT. When the measurement is complete, the sample stages then move to the next point.

b) *Continuous X*. Here, the y-axis is first set and the x-axis is set to the first point of the scan, all done at the maximum stage velocity. The velocity of the x-axis translator is then changed to a smaller value (depending upon several factors) and then moved continuously to the end of the x-scan. During this continuous motion, PMT and x-position data are taken as fast as the electronics allow. At the end of the scan, the intensity at each point is determined by a spline interpolation routine. The velocity is set such that the PMT can respond to the changes in the light intensity; its time constant (*PMT TC* in seconds) is shown in the scan type box. The velocity increases as the *PMT TC* decreases, but the measurement time required to measure the x-position is also a factor.

Clearly, the *Continuous X* method is faster, since it eliminates the need to constantly stop and start the x-axis translator motor, but it is not as accurate. This loss of accuracy is due mostly to the PMT control circuitry not being able to keep up with the rapid changes in the light intensity, making the monitoring of the voltage less accurate. For the measurement of coated particle fuel pyrocarbon anisotropy, this loss of accuracy is not particularly important and is more than compensated by the improvement of the speed of measurement.

The picture box occupying most of the window is where the data is dynamically displayed using the color scale, shown to the right of the picture box. The pattern displayed in Figure 7 is an experimental image of a hexagonal array. The maximum and minimum intensity values are shown at the top and bottom of the color scale, and can be increased or decreased using the appropriate arrows. Once the appropriate scan parameters are set, the measurement is initiated by pressing the *Measure* button at the bottom of the window.

After a measurement has begun, the *Timing* group box shows the elapsed time, the estimated time to the end of the active measurement, and the estimated total time for the measurement. The *Abort* button will also appear once the measurement has begun. Pressing this will abort the present scan, but it may take a while for the scan to abort and it may be necessary to press the *Abort* again if the routine was not in a position to recognize the initial *Abort* button push. Once the scan ceases, the status line will update indicating that the scan has been aborted.

If the present intensity profile has already been measured, then it can be entered directly. Select *Default* from the pull-down list to the left of the *Enter* button and then press the *Enter* button. This will load the last profile that was successfully measured. Other profiles can also be loaded if the pull-down menu is set to *Enter File*. This will open a dialog that will allow the user to select an alternate intensity profile that has been previously saved. Two other *Enter* options are also included in the pull-down menu: *Gen Hex* and *Gen Rect*.; these two choices will generate an intensity profile based on a calculation, and can be used for demonstration purposes. The *Gen Hex* is a hexagonal array of TRISO particles, while the *Gen Rect*. is a rectangular array.

Once an Intensity profile has been measured, it can be saved using the *Save to file* button, and will use the File Name above the button. The image of the intensity profile can also be saved as a *.jpg file by pressing the *Save Pict.* button.

Once the intensity profile has been measured or entered, it is possible to select regions around each particle, or area of interest, that will be later measured using a finer xy increment. As the user places the cursor over the image, the group box will give updated data of the X and Y positions (as an integer), as well as the intensity at that point. A region is selected by left-clicking the mouse at the upper left of the region to be selected, dragging the cursor to the lower right of the region to be selected, and releasing the left mouse button. While the cursor is being dragged, a dynamic image of the selected region will be shown on the plot and the XY coordinates will be shown in the group box labeled *Region Dimensions*. Once a region has been selected, it can be altered using the arrow buttons in the *Region Dimensions* group box. The increments (in mm) of the X- and Y-scans are defaulted to 0.005 mm, but can be changed in the text box of *Region Dimensions*. Also, it is possible to manually enter the maximum and minimum coordinates of the selected region. As these parameters are changed, the number of points will be updated.

The coordinates of the selected region are rectangular, but it is possible to also use an elliptical region. A donut-shaped region may be included in the future, but is presently disabled. The elliptical region will draw an ellipse within the rectangular coordinates of the selected region. The elliptical region shape is particularly useful for TRISO particles, since it ignores the corner parts of a rectangular region which are of no interest, thus reducing the measurement time.

In selecting regions, it is possible to select 16 separate regions, as indicated by the radio buttons in the group box *Region Number*. If the region number is not changed, then the newer coordinates will over-write the older numbers. However, changing the region number will save the region data for the unselected regions. When the region number is changed, then the plot and data from the previous region will disappear from the plot and the *Region Dimensions* data, but not from memory. When the user is finished, then pressing the *Plot All Regions* button will generate a plot of all the selected regions using the most recent coordinates.

Calibrate Reflection Transmission SWL

Figure 8 shows the window used to perform a calibration of the 2-MGEM2 instrument at a single wavelength (SWL). To perform this calibration, an aluminum mirror must first be inserted into the 2-MGEM2 and the surface of the mirror imaged onto the detector. This is done by observing the image from the CCD array camera and by bringing that image into focus by changing the height of the sample, either using the automated or manual z-axis control.

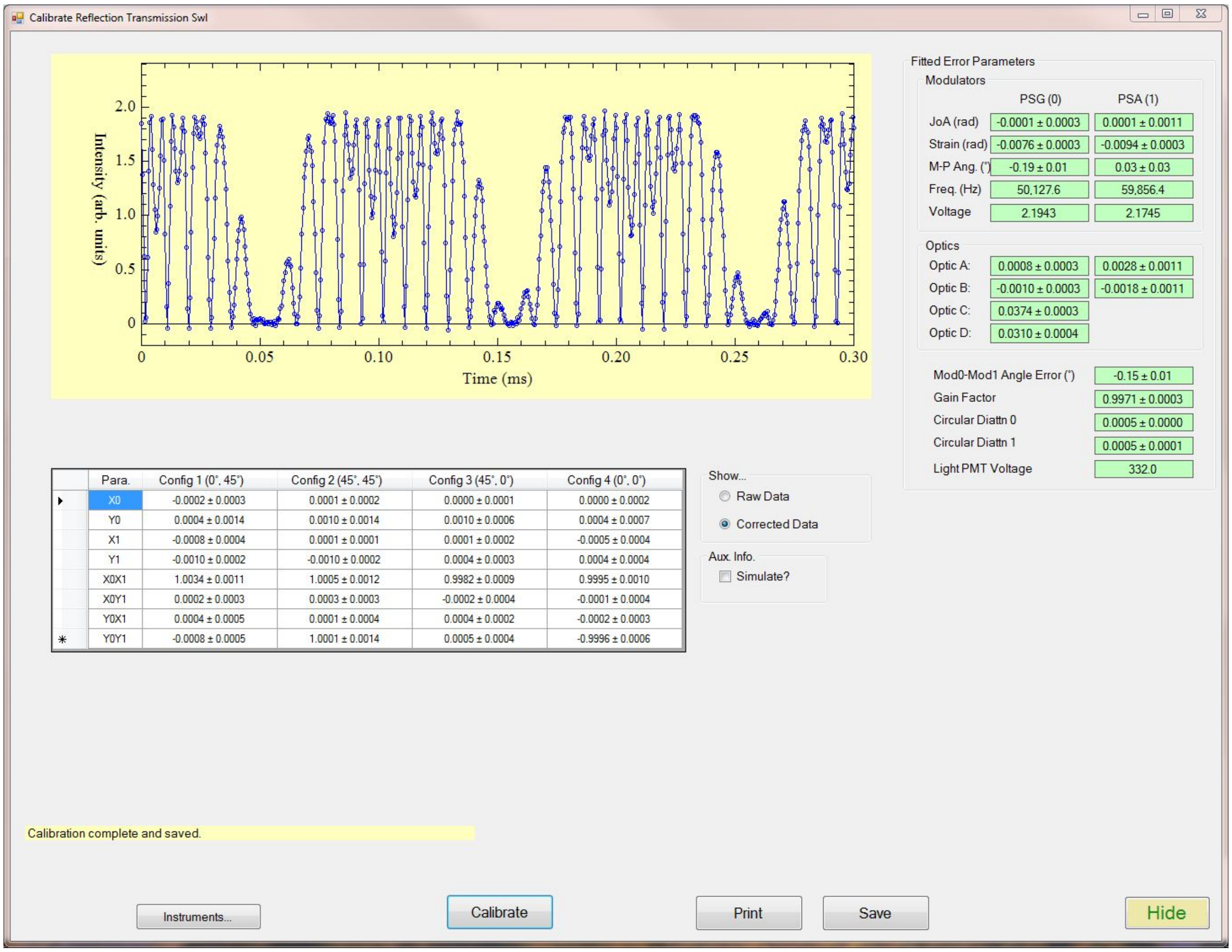

Figure 8. The Calibration routine.

The individual instrument panels (described above) can be viewed by first pressing the *Instruments…* button, and the pressing the appropriate button in the *Select Inst.* group box. Note that the Translators panel is not included, since there should be no need to alter the xy position of the sample during the calibration.

The calibration is then performed by pressing the *Calibrate* button. The instrument will then automatically perform a 4-zone calibration by moving the rotators to the appropriate configuration and acquiring the 2-MGE data. As the data is collected, a representative waveform will be plotted in the plot field at the top of the window and the values of the 2-MGE parameters will updated in the data table below the plotted intensity. Once the raw data has been collected, the program will calculate the various offsets determined by the calibration and then correct the raw data. These offsets are discussed in more detail in the Appendices, but this is a summary of the parameters:

Modulators:

a) *Jo(A)*: This is a measure of the magnitude of the 0^{th} order Bessel function, which is a direct measure of the amplitude of the PEM modulation. If the amplitude is 2.4048 rad, then $J_o(A) = 0$ and any deviation from 0 can be used to correct the set voltage of the PEMs.
b) *Strain*: This is a measure of the static strain of the modulators.
c) *M-P Ang.*: This is the error of the Modulator-Polarizer angle from 45°.
d) *Freq.*: The measured frequency of the Modulators.
e) *Voltage*: The applied voltage on the Modulators.

Optics:

The optics, particularly the large diameter objective, will alter the polarization state of light reflecting from or passing through them. These can be summarized using the A, B, C, and D parameters, discussed in the Appendices. Generally speaking, the A and B terms are quite small and affect the measurement of the diattenuation, while the C and D terms can be quite large and affect the retardation.

General parameters:

a) *Mod0-Mod1 Angle error*: The error of the angles between the PSG and the PSA. This should be close to 0.
b) *Gain factor*: The parameter X0X1 should be close to 1. This parameter corrects for any small discrepancy between the measured values of X0X1 and 1.
c) *Circular Diattn 0 and 1*: The circular diattenuation parameters (X0 and X1) should average to 0; these corrections are any small deviations from 0.
d) *Light PMT Voltage*: The average PMT voltage as determined during the calibration. This parameter is used in the determination of the relative intensity.

Once the calibration is complete, the raw data or the corrected data are listed in the table below the plotted waveform. The user can switch between the two representations by pressing the representative radio button to the right of the data table. The calibration is NOT automatically saved; to do this, press the *Save* button at the bottom of the window. Pressing the *Print* button will result in printing a summary of the calibration, which is shown in Figure 9.

4 Zone Calibration

Date: 3/25/2013 11:05:09 AM
Version: 1.0.0.0

Wavelength (nm): 577.0
Measurements: 10
Traces: 5

Raw Data:

Element	Config 0 (0°, 45°)	Config 1 (45°, 45°)	Config 2 (45°, 0°)	Config 3 (0°, 0°)
X0	0.0007 ± 0.0001	0.0005 ± 0.0001	0.0005 ± 0.0001	0.0005 ± 0.0002
Y0	0.0006 ± 0.0002	0.0020 ± 0.0003	0.0014 ± 0.0002	0.0013 ± 0.0002
X1	0.0005 ± 0.0002	0.0007 ± 0.0002	0.0006 ± 0.0002	0.0004 ± 0.0002
Y1	0.0015 ± 0.0001	-0.0006 ± 0.0002	0.0005 ± 0.0003	-0.0015 ± 0.0002
X0X1	1.0029 ± 0.0009	0.9911 ± 0.0007	0.9979 ± 0.0008	0.9881 ± 0.0012
X0Y1	0.0222 ± 0.0004	0.0279 ± 0.0004	-0.0506 ± 0.0003	-0.0561 ± 0.0003
Y0X1	-0.0479 ± 0.0003	-0.0277 ± 0.0004	-0.0364 ± 0.0003	-0.0557 ± 0.0003
Y0Y1	-0.0027 ± 0.0003	0.9964 ± 0.0022	-0.0021 ± 0.0003	-0.9901 ± 0.0028
PMT V	301.7	302.3	293.5	296.7
Mod0 Freq	50,128.0	50,128.0	50,128.0	50,128.0
Mod1 Freq	59,857.1	59,857.1	59,857.2	59,857.2

Average Error(X0, Y0, X1, Y1): 0.00017

Corrected Data:

Element	Config 0 (0°, 45°)	Config 1 (45°, 45°)	Config 2 (45°, 0°)	Config 3 (0°, 0°)
X0	0.0001 ± 0.0001	0.0000 ± 0.0001	-0.0001 ± 0.0001	-0.0001 ± 0.0002
Y0	-0.0002 ± 0.0002	0.0013 ± 0.0003	0.0013 ± 0.0002	-0.0002 ± 0.0002
X1	0.0000 ± 0.0002	0.0001 ± 0.0002	0.0000 ± 0.0002	-0.0002 ± 0.0002
Y1	-0.0013 ± 0.0001	-0.0013 ± 0.0002	-0.0002 ± 0.0003	-0.0002 ± 0.0002
X0X1	1.0080 ± 0.0009	0.9960 ± 0.0007	1.0030 ± 0.0008	0.9933 ± 0.0012
X0Y1	-0.0002 ± 0.0004	0.0000 ± 0.0004	-0.0001 ± 0.0003	-0.0002 ± 0.0003
Y0X1	-0.0002 ± 0.0003	0.0001 ± 0.0004	-0.0003 ± 0.0003	0.0002 ± 0.0003
Y0Y1	-0.0003 ± 0.0003	1.0012 ± 0.0022	0.0003 ± 0.0003	-0.9948 ± 0.0028

Results: Modulators

	PSG (0)	PSG (1)
Frequency	50128.0	59857.2
Eps b	-0.06 ± 0.01	0.02 ± 0.01
Strain	-0.0055 ± 0.0003	-0.0083 ± 0.0002
JoA	-0.0001 ± 0.0002	-0.0001 ± 0.0002
Voltage	2.1932	2.1539
CD Offset	0.0006 ± 0.0000	0.0005 ± 0.0000
PEM Azi. Angle	0.07 ± 0.00	
Gain Factor	0.9951 ± 0.0002	

Results: Optics

	0	1
A	-0.0005 ± 0.0002	0.0021 ± 0.0002
B	-0.0015 ± 0.0002	-0.0012 ± 0.0002
C	0.0307 ± 0.0004	
D	0.0423 ± 0.0003	

Figure 9. Example of a calibration print-out.

Measure 2-MGEM2

The Measure 2-MGEM2 window (Figure 10) is where the actual 2-MGEM2 measurements are displayed. It should be activated only after the calibration routine has been run and the regions have been selected from the XY Scan Setup routine. Once those two routines have been run, the user need only enter an appropriate file name and any description string that the user would like to associate with the file.

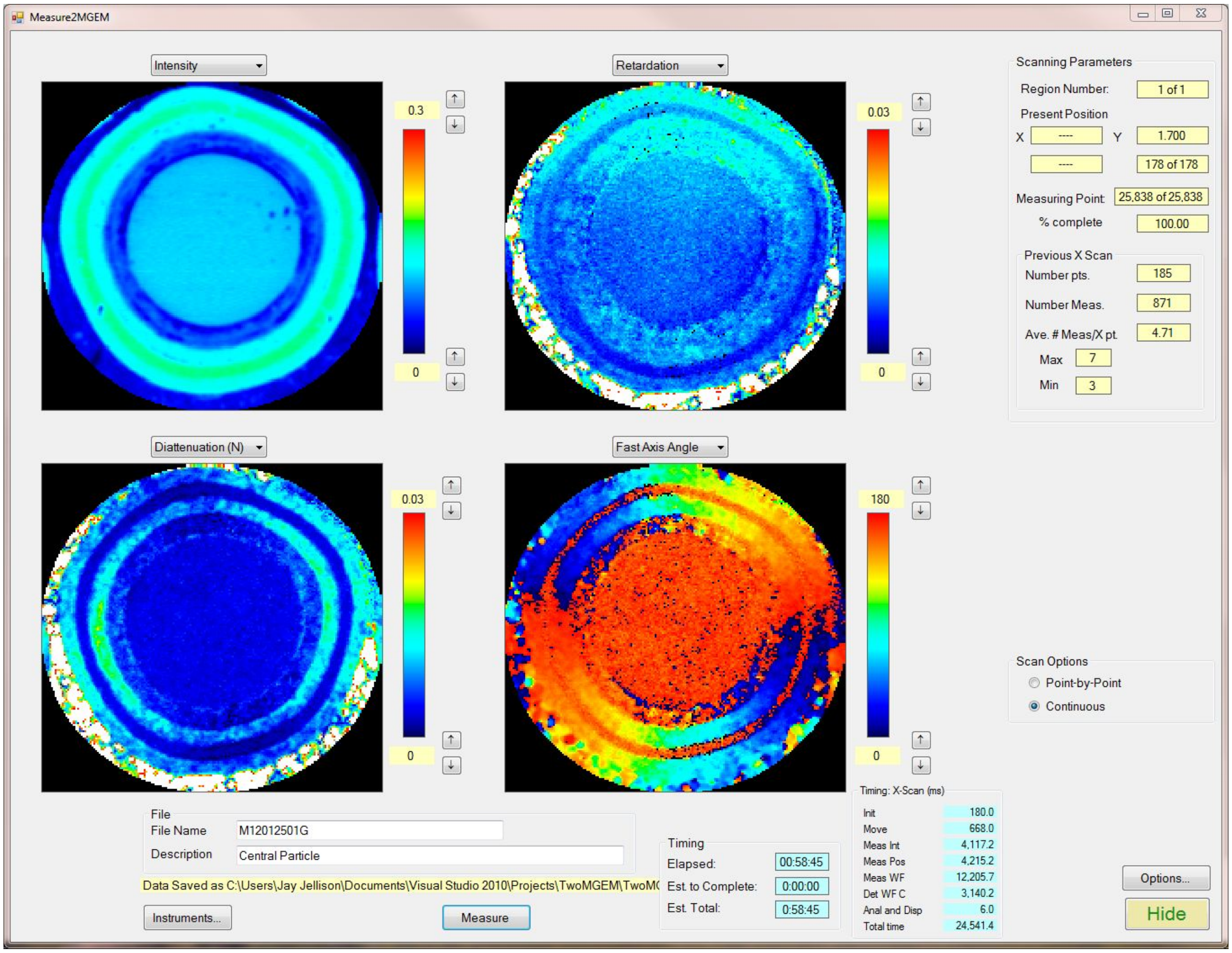

Figure 10. The measurements window.

As with other windows, the individual instrument panels can be displayed to the left of this window by pressing the *Instruments…* button, and the PMT data and instrument data panels will be displayed to the right. The scanning can be done point-by-point or continuous, where the continuous technique is similar to that described in the XY Scan Setup routine.

a) Scanning *Point-by-Point* first moves the translation stage to the next measurement spot and then takes the number of measurements as specified on the Digitizer panel. Since the data are available real-time, the updated data are available in the instrument data box, and the updated timing information data are displayed in the timing group box (see below).
b) A *Continuous* scan will first move to the next y-point, beginning x point, and then move continuously to the ending x point, taking data along the way. The data recorded are the average of the points recorded that have x-values closest to the x-point being recorded, as determined

from the XY setup window. For example, if the x-parameters to be measured (that is, the x-points determined in XY Scan Setup) were (…., 0.025, 0.030, 0.035, …) and the actual measured points were (…, 0.02685, 0.02777, 0.02868, 0.02957, 0.03048, 0.03139, 0.03230, 0.03335, 0.03425, 0.03515, …), then the points measured at (0.02777, 0.02868, 0.02957, 0.03048, 0.03139, 0.03230) would be included in the average for X=0.030, while the 0.02685 point would be included in the average for X=0.025 and the 0.03335, 0.03425, 0.03515 points would be included in the average for X=0.035. The primary advantage of the continuous scan is that the measurements can be accomplished faster than with the point-by-point scan, but more data is available real-time with the point-by-point scan.

Four plots of the data are presented real-time to the user. These can be selected by the user using the drop-down list above each of the four plots. As shown in Figure 10, the defaults are: *Intensity*, *Diattenuation*, *Retardation*, and *Fast Axis Angle*. The other possibility is the *Depolarization*.

Real-time display of data occurs during the measurement, but is different for the point-by-point and continuous scans. Since a real-time data point is taken at each xy point for a point-by-point scan, it is possible to calculate and display parameters such as the diattenuation, retardation, etc. in real time. Also, the instrument data panel to the right is updated real-time for the PMT and PEM parameters, as well as the values of the 2-MGE parameters. This is not possible with the Continuous scan option, since the final determination of 2-MGE parameters takes place at the end of the x-scan.

During the continuous scan, there are several parameters that are available for continuous updating. These include the y-axis position, the number of points measured, as well as the total number of points in the scan, and data concerning the previous x-scan. These are included to give the user some confidence that the measurement is proceeding properly. For example, the average number of points per X-point should be approximately the number of measurements set on the digitizer/DAQ panel, and displayed to the right in the Instrument Data panel. In the present case shown in Figure 10, the average number of measurements is 4.71, which is reasonably close to the set number of measurements (5). Statistics are also included for the maximum and minimum number of measurements made per X-point. For this case, there were at least 3 and no more than 7 measurements made for each X-point.

The continuous scan option also includes several internal options to reduce the measurement time. In particular, the object is to spend as much time as possible recording and analyzing waveforms at the expense of other, less important measurements. For example, the measurement of the relative intensity is accomplished by reading the current and voltage of the PMT, calculating (Current / Voltage^7.5), and comparing this with the relative intensity determined during the calibration routine using the aluminum mirror. However, this does take some significant amount of time if it were done every time the waveform were captured and analyzed. Similarly, the exact position of the translator stage can be measured by querying the translator controller, but this is an RS-232 operation, and is not fast. To compensate for this, measurements of the intensity and the x-stage position are taken only every Nm times, and the values between measurements are estimated using interpolation. Presently, we are using Nm=3; that is, measurements of the intensity and position of the X-stage are taken whenever N mod Nm =0 (0, 3, 6, 9, 12, etc.), where N is the number of the waveform capture and analysis.

To monitor this, a timing group box is displayed (displayed only for maintenance engineers) that shows the time spent (in milliseconds) performing several activities during an X-scan. The *Init* time is the time spent in initialization of the scan and *Move* is the time spent in initializing and executing the continuous X-axis move; these are generally small. The *Meas Int*, *Meas Pos*, and *Meas WF* are the total times during the X-axis move taken to measure the intensity, position, and waveform (this does not include the time to analyze the waveform). Clearly, most of the time during the scan is taken by these three processes. If Nm were 1 (measuring the intensity and position every time a waveform is measured), then the *Meas Int* and *Meas Pos* times would be considerably larger. The *Det WF C* is the time taken to analyze the waveforms and then to convert the analyzed waveform coefficients into the 2-MGE parameters and parameters such as N, S, C, Fast Axis Angle, depolarization, retardation, etc. This calculation is done for all the points at the end of the X-scan. The *Anal and Disp* time is the time required to update the plots and the display. The *Total time* is the time for the entire x-scan process to take place, and is not simply the sum of the times shown above. Since the sum of the times in Figure 10 is 24,532.3 ms and the entry in the *Total time* box is 24,541.4 ms, only 9.1 ms is not accounted for.

Waveform Analysis

The Waveform Analysis window (Figure 11) is primarily a diagnostic tool for the 2-MGEM2, allowing the user to capture and analyze a waveform directly. As with other windows, panels are available to access the appropriate instruments by pressing the *Instruments…* button, displaying the panel to the left of the window (note that the translator panel is not available here), and the *Instrument Data* and *PMT* data panels are displayed automatically to the right. The *Capture Waveform* and *Stop* (the stop button is displayed only after the waveform capture has been initialized) are initiated using buttons at the bottom of the window. If the capture is not stopped manually, it will automatically stop after *Max. Captures* have been captured. The intensity waveform is displayed at the top of the window, and its Fourier transform is displayed just below. Scales for either the intensity or the Fourier Transform can be changed using the appropriate arrow buttons.

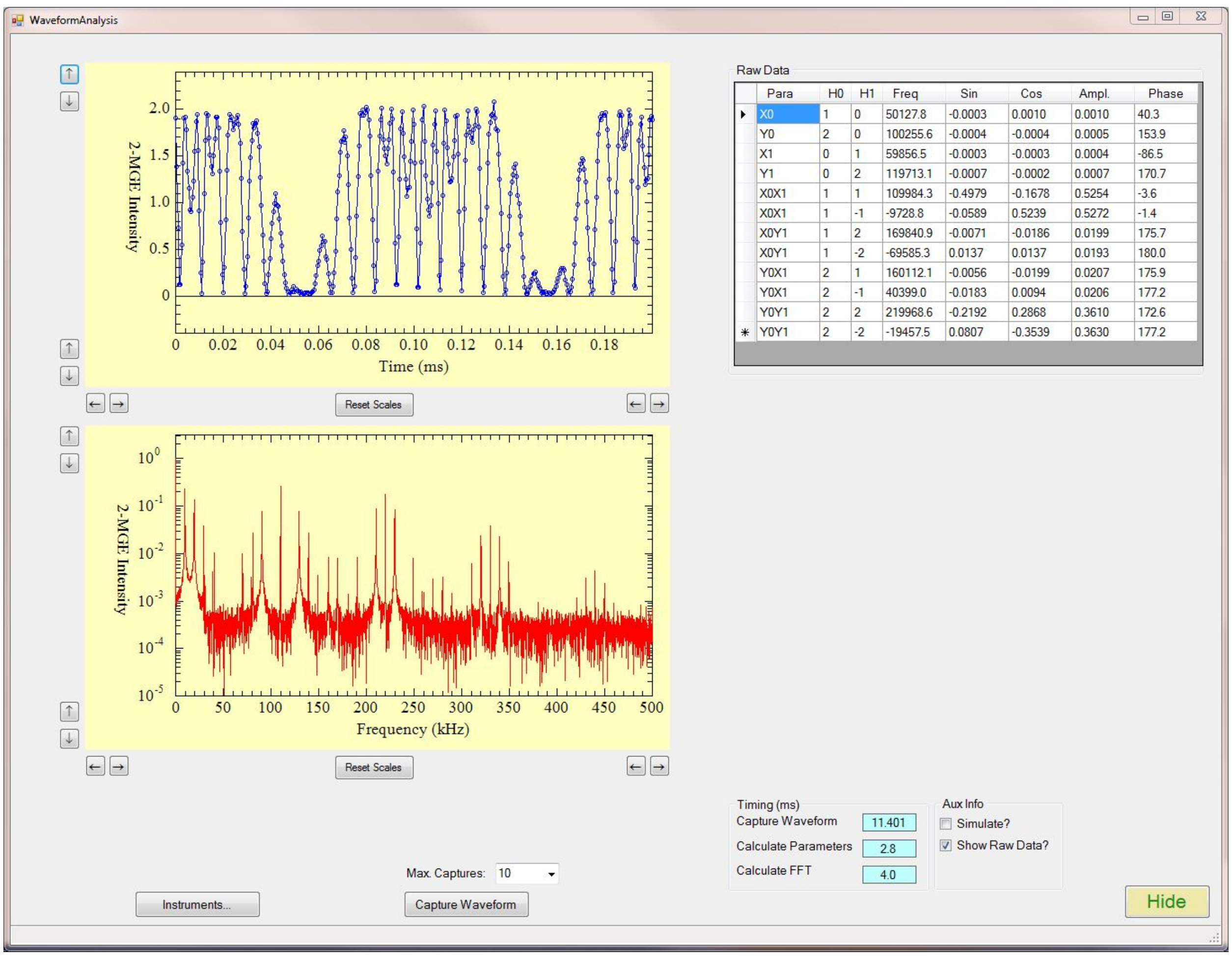

Figure 11. The waveform analysis window

The analyzed data for each waveform is displayed in the data table for each of the waveform coefficients, which is set on the *Digitizer/DAQ* panel. The normal default is 12 frequencies, one each for X0, Y0, X1, Y1, and two each (sum and difference frequencies) for X0X1, X0Y1, Y0X1, and Y0Y1. However, other options include 8 frequencies, 16 frequencies, and 24 frequencies. If there are more than 8 frequencies measured, then some of the 2-MGE parameters may be determined from more than one frequency. The table displaying the data consists of 8 columns:

a) *Para* The 2-MGE parameter determined
b) *H0* and *H1* The harmonic of PEM0 and PEM1 used to determine the parameter
c) *Freq* The frequency = H0*freq(PEM0)+H1*freq(PEM1). Negative frequencies just indicate a 180° phase shift.
d) *Sin* and *Cos* The sine and cosine coefficients determined from the waveform analysis
e) *Ampl* The amplitude of the coefficient = sqrt(Sin^2 + Cos^2)
f) *Phase* The final phase of the coefficient, after all corrections. These phases should be close to 0 or 180° for coefficients with large amplitudes.

Timing information is also given for the capture of the individual waveforms. In Figure 11, it takes 11.4 ms to capture a 16,384 sample waveform at 2 MHz (0.5 microseconds per sample point). Thus, 8.2 ms is taken capturing the waveform and 3.2 ms is taken transferring the data into the computer and putting the data into a useful form. In performing waveform captures, it will be found that the total capture and display time is considerably longer than this. This is because the time required to plot the Fourier transform is considerable, since it contains 16384/2=8192 points.

Hardware

Electronics

The new version of the 2-MGEM2 now uses an electronics box made by Hinds instruments. This box contains the following equipment:

Power supply and control electronics for the PEMs

These are custom electronic circuits made by Hinds Instruments that control the photoelastic modulators. The amplitudes of the PEMs are controlled by voltages set by the computer using the digital to analog converters of the National Instruments (NI) 6361 DAC card, which utilizes the peripheral component interconnect express (PCIe) bus. One of the outputs from each PEM control card is a square wave at the present frequency of the PEM. This signal goes to the counter-timer of the NI 6361, which is configured to measure frequency very accurately. This frequency square wave also goes to the trigger circuit, from which a trigger pulse is generated.

Power supply for the automatic temperature control of the PEMs

The 2-MGEM2 uses up-to-date Hinds PEMs with automatic temperature control (ATC); the power to the ATC of the PEMs is supplied by the electronic box.

The controllers for the rotators and the translators.

The controllers of the rotators and translators are contained in the electronics box, and are powered by a power supply within this box. These controllers are Newport SMC100CC single-axis controllers, which are ganged together, so that they can all be addressed using a single RS-232 interface. The DC power required for these controllers is supplied by the Hinds Instruments electronics box.

The trigger circuit for waveform data acquisition.

Waveform acquisition is initiated by a trigger pulse generated by a trigger circuit within the Hinds Instruments electronics box. A trigger is initiated whenever the phases of both PEMs cross 0° at the same time (within the specifications of the electronics, generally ~0.3° phase or 14 ns).

The PMT control circuit that dynamically controls the voltage to the PMT

The photomultiplier tube is controlled by a circuit that dynamically controls the applied voltage so that the average current coming from the PMT is constant. If the light level is smaller, then the PMT voltage will automatically increase to compensate for this, resulting in a constant signal out, but with decreased signal-to-noise. If the light level is very small, then the voltage will increase to a maximum value before the current level will decrease. The output current is then converted to a voltage so that the waveform can be digitized using the waveform digitizer capability of the NI 6361. Since the average output signal is constant even in the presence of wide variations in light, the gain on the digitization circuitry is constant, considerably simplifying the waveform acquisition. While the limiting current and voltage of the PMT is manual for the 2-MGEM2, the operating software allows for use of automatic systems, such as that used in the Hinds Instruments version.

Electronic connection to the National Instruments NI 6361 data acquisition card

The NI 6361 card provides the following controls and inputs for the 2-MGEM2.

a) Waveform digitization. This is normally done at 2MHz, but can be done at lesser frequencies by changing the sample rate on the Digitizer/DAQ panel. During the digitization, only a single channel can be read at 2 MHz; this is a limitation of the NI 6361 card.
b) PMT current and voltage. These are voltages transmitted from the PMT to the NI 6361, and are used to monitor the PMT and to determine the relative light level from the sample.
c) PEM frequencies. The output waveform from the PEM control circuitry is fed into the counter/timers of the NI 6361, where the frequency of the PEMs is measured. For the frequencies of the PEMs (~50 and 60 kHz), this measurement can be performed to an accuracy of ~0.1 Hz.
d) PEM amplitude set. Two digital-to-analog converters supply voltage signals to the PEM control circuitry to set the amplitude of the PEM modulation. These are 16 bit, 0-10 V DACs.
e) Trigger input. Output from the trigger generation circuitry goes into a trigger input of the NI 6361, which is used to initiate the waveform digitization.
f) PMT current and voltage sets. With some systems, the PMT current and voltage can be set from the computer; this requires a NI 6363 card, which has an additional two DAC outputs.

The Optics and the Optical Alignment Procedures

In general, once the optics are appropriately aligned, very little change needs to be made to the optical elements. However, it is helpful to keep some general principles in mind if the system needs to be re-aligned. The numbers in parentheses indicate the numbers in Figure 1 and the equipment list.

a) Alignment of the input collimator (3). The input collimator is a microscope objective attached to the 100-μm-diameter end of the tapered fiber. This optical element is operated inversely to the traditional application of a microscope objective, in that it takes the diverging light from the end of the optical fiber and collimates it. For best alignment, the end of the fiber must be near the focal point of the microscope objective. The position of the end of the fiber with respect to the objective is controlled by manual Thor Labs zoom and xy translators. If the position of the fiber end is exactly at the focal point, then the light will image at infinity; if the end of the fiber is further away, the light will image at a shorter distance. With the 2-MGEM2, the optimum distance (set by the zoom device) will result in a focused image of the fiber end at the top of the large aperture objective (6); this in turn will result in the smallest possible light spot at the sample.
b) Large aperture objective (6). This optic serves as the condenser for incoming light and as the objective for outgoing light. For the incoming light, the primary purpose is to maximize the light intensity on the sample. For the outgoing light, this optical element serves as the objective, directing the light diverging from the analysis spot back into a quasi-collimated beam, which can be imaged by the tube lens (7) and imaging lens (9c) onto the CCD array (9e) and the pinhole/PMT (9d) with an intermediate focal point at the entrance aperture of the detector head assembly (9). Generally speaking, adjustments to this optic should be limited to being sure that the incoming and out-going light beams are symmetric about the center of the objective, these can be adjusted by using the xy translator stage attached to the objective. The actual imaging is performed by changing the z-distance from the sample to the objective using the manual Z stage (12).

c) Tube lens (7). This lens forms part of the imaging optics; light passing through this element will be imaged onto the entrance aperture of the detector head assembly. This lens is attached to a Thor Labs xy translator, so that small adjustments can be made to alter the image point on the CCD camera and the pinhole in the detector assembly. This is the most common minor adjustment to be made to the optics of the 2-MGEM2.

d) Detector Head Assembly (9). Light from the sample through the large aperture objective (6) and the tube lens (7) is imaged onto the entrance aperture of the detector head assembly. The entrance aperture is then imaged onto the CCD camera and the pinhole in front of the PMT by the Imaging lens (9c), making the entrance aperture an intermediate focal plane. The beam splitter is a pellicle, so that there is virtually no distortion of the light path from the entrance aperture to either the pinhole or the CCD camera. (The beamsplitter used in the previous version of the 2-MGEM was an ~1 mm thick fused quartz plate with a beam splitting film, distorting the path to the pinhole.) By adjusting the z-position of the pinhole, it is possible to make the distance from the entrance aperture to the pinhole exactly the same as to the CCD camera. Therefore, if the system is focused to the CCD camera, then it will also be focused to the pinhole. This adjustment is done using a USAF-1951 resolution target.

Phasing of the PEMs

Each of the modulators will have an optical phase that is somewhat different from the electronic phase, as indicated by the waveform coming from the PEM electronic circuitry. As a result, there needs to be a correction to the phase to take this into account. This phase correction enters into the determination of the final phase in a complicated way and is described in detail in ref. 3. The signs of the waveform coefficients are determined using the expression: Sgn(Cos(ϕ_i)), where Sgn is the sign operator, and ϕ_I is the phase of the waveform coefficient I; therefore, it is not particularly important to have a precise value of the phase, but it must be correct to ~10°. The final phase is shown on the table of the Waveform Analysis window, and should be either ~0° or ~180°, where 0° corresponds to a sign of +1 and 180° corresponds to a sign of -1. (Note that the phases are reported in the table from -180° to +180°, so a value of 181° would be converted to -179°.) The present value of the phase correction for each modulator is shown on the PEMs and PMT panel, and can be changed if the engineer privileges are activated.

The data from the Waveform and Analysis window can be used to correct the phases of the PEMs. If the rotators are placed in the (0°, 0°) configuration and the aluminum mirror sample is used, then the X0X1 term should be near +1, and the Y0Y1 term should be near -1. If the Analysis type is set to 12 parameters, then the two measurements of X0X1 should show a phase near 0°, and the two measurements of Y0Y1 should show a phase near 180°. If δP(0) and δP(1) represent the phase corrections for the 0 PSG and 1 PSA modulators, and MP(4) and MP(5) represent the measured phases for the two measurements of X0X1, then corrections can be made to the phases of the two modulators using the expression: $\delta P(0)=-(MP(4) + MP(5))/2$, $\delta P(1)=-(MP(4) - MP(5))/2$. After the corrected phases are entered into the Modulators and PMT panel, and the waveform is again captured using the Waveform Analysis window, then the measured phases for the X0X1 and Y0Y1 terms should be closer to 0° and 180°, respectively.

Changing the Mercury Arc Lamp

The mercury arc lamp is particularly useful for this application in that it presents the smallest arc size of any standard arc lamp, and it has several emission lines that can be used as a quasi-monochromatic light source. However, these advantages come at a price: the lifetime of these mercury arc lamps is relatively short (~200 hours). As a result, they must be changed on a regular basis. The number of hours that the lamp is run must be routinely monitored, and the lamp changed once the 200-hr limit has been reached. As the lamps age, the metallic components of the anode and cathode evaporate onto the inside of the glass bulb, obstructing the light from getting out. Consequently, the light intensity will decrease with the age of the lamp with the absorbed light energy heating the bulb, so it may be advisable to change the bulb before the stated 200-hr lifetime. If the bulb is run too long, the buildup of the evaporated metals could distort the bulb, eventually causing it to explode. The explosion of the bulb is not particularly dangerous (the bulb is small and enclosed), but this will involve the cleanup of the glass fragments.

Changing the bulb procedure:

1. Turn off the bulb and wait for it to cool (~1/2 hr if the bulb has been running).
2. Disconnect the power to the lamp, as well as the interlock.
3. Wear the appropriate protective equipment:
   a) Gloves
   b) Face shield
   c) Long-sleeve shirt and pants
4. Disassemble the lamp housing; be sure to wear the face shield, since there is a very small chance that the old bulb may explode in the changing process. For the series Q housing, there are four set screws at the bottom corners that must be released before the top can be lifted off.
5. Unscrew the two set screws on the top contact and heat exchanger; lift off the top contact.
6. Unscrew the two set screws on the bottom contact. Lift out the old bulb and put it in a secure place so that if it should explode, no damage is done.
7. Insert the new bulb. The bottom and top contacts are different sizes, so there should not be any confusion concerning the orientation of the bulb. Note that the orientation and contacting of the mercury arc lamp is important; if this is not done correctly, the bulb will explode.
8. Screw in the set screws on the bottom contact and insert and screw in the top contact.
9. Replace the lamp housing, and screw in the four set screws at the bottom of the housing. In setting the lamp housing back onto its base, be sure that the interlock switch on the back panel is activated.
10. Re-connect the power cables. Be sure to get the orientation of the cables correct, since the cable orientation is different for mercury arc lamps than for xenon lamps. Also, connect the interlock switch.
11. Turn on the power to the power supply, and then press the start button on the front of the power supply.
12. If the lamp does not immediately ignite, there are several possibilities:
    a) The interlock cable is not connected at the lamp housing or at the power supply.
    b) The internal interlock switch has not been activated.
    c) The bulb has not been installed correctly, or the polarity of the power cables is wrong.

Equipment List (Partial)

This equipment list includes all the optical components and computer-controlled components. It does not include all the structural components, most of which were purchased from Thor Labs or machined in-house.

#	Component	Company	Part #	Comment
1	Light Source	Newport/Oriel (N/O)	----	----
	Housing	N/O Series Q	60010	
	Focusing lens	N/O	60080	
	Back Reflector	N/O	60005	
	Replacement mirror	N/O	60015	
	Power supply 50–200 W	N/O	68806	
	Mercury arc lamp 100 W	N/O	6281	200 hr. lifetime
2	Fiber	Fiberguide Industries	---	400 -> 100 μm taper
3	Microscope Objective	Edmund	?	10X objective
4a	PEM 50 kHz	Hinds Instruments	I/FS50 ARC-2 ATC	AR: 450–650 nm.
4b	PEM 60 kHz	Hinds Instruments	I/FS60 ARC-2 ATC	
5	Polarizer (2)	Karl Lambrecht	MGTYE10	calcite prisms (No AR)
---	Polarizer holder/rotator	Klinger/Newport	TR46SBL	
6	Large aperture objective	Leica	10411597	
7	Tube Lens	Thor Labs	AC254-200-A1	Achromat
8	Directing Mirrors (2)	Thor Labs	PF10-03-G01	
9	Detector Head	----	----	----
9a	Interference filter	Semrock	FF01-576/10-25	577 nm BP
9b	Beamsplitter	Thor Labs	CM1-BP108	Pellicle beam splitter
9c	Imaging Lens	Thor Labs	AC254-30-A	Achromat
9d	Photomultiplier tube	Hamamatsu	R3896	
9e	Camera	Thor Labs	DCC1240M	USB connection
10	Auto Rotators (2)	Newport	URS50BCC	
11	XY Auto Translators(2)	Newport	VP-25XA	25 mm travel
12	Z Manual translator	Newport	MVN80	
---	Controllers for Auto (4)	Newport	SMC100CC	RS-232 interface
---	Data Acquisition card	National Instruments	NI 6361	PCIe interface
---	Computer	Hewlett Packard	8200 Elite	Windows 7 64 bit
---	Standard Al mirror	Edmund	46649	4–6 λ, 19-mm dia

Appendices

A. Interpretation of the Light Intensity from the 2-MGEM (from Ref. 1)

The signal of interest is the intensity of the light beam that enters the PMT through the pinhole. As with any 2-MGE experiment, the intensity of this light beam is given by[1, 2]

$$I(t) = I_{dc} + I_{X0}X0 + I_{Y0}Y0 + I_{X1}X1 + I_{Y1}Y1 + I_{X0X1}X0\,X1 + I_{X0Y1}X0\,Y1 + I_{Y0X1}Y0\,X1 + I_{Y0Y1}Y0\,Y1, \quad \text{(A.1a)}$$

where the basis functions are:

$$X0 = \sin(A_{m0} \sin(\omega_{m0}\, t)), \quad \text{(A.1b)}$$

$$Y0 = \cos(A_{m0} \sin(\omega_{m0}\, t)), \quad \text{(A.1c)}$$

$$X1 = \sin(A_{m1} \sin(\omega_{m1}\, t)), \quad \text{(A.1d)}$$

$$Y1 = \cos(A_{m1} \sin(\omega_{m1}\, t)). \quad \text{(A.1e)}$$

The amplitude of the PEM oscillations is expressed by the A_{m0} and A_{m1} terms (in radians), while the resonant frequencies of the two PEMs are given by ω_{m0} and ω_{m1}. The values of X0, X1, Y0, and Y1 in Eqs. A.1b-e can also be expressed in terms of a converging infinite series of sines and cosines multiplied by integer Bessel functions. Normally, the 2-MGE is operated such that $A_{m0} = A_{m1} = 2.4048$ radians, since this is the value at which $J_0(A) = 0$. The measured quantities of any 2-MGE are the coefficients of the basis functions (I_{X0}, I_{Y0}, etc.) normalized to the average or "dc" intensity I_{dc}. These 8 parameters correspond to 8 elements of the normalized sample Mueller matrix, where the azimuthal orientations of the polarizer-PEM pairs of the PSG and the PSA determine which of the 15 Mueller matrix elements are measured. For example, if the PSG is set to 0° and the PSA is set to 45° (the normal operating configuration for the 2-MGEM2), then the measured elements of the Mueller matrix (ignoring signs) are

$$\mathbf{M}_{\text{sample+perturbations}} = \begin{bmatrix} 1 & \bullet & I_{Y0} & I_{X0} \\ I_{Y1} & \bullet & I_{Y0Y1} & I_{X0Y1} \\ \bullet & \bullet & \bullet & \bullet \\ I_{X1} & \bullet & I_{Y0X1} & I_{X0X1} \end{bmatrix}. \quad \text{(A.2)}$$

Note that the Mueller matrix that is measured contains contributions from ALL the optical elements between the PSG and the PSA. In this case, this includes the reflections from directing mirrors 1 and 2, the input and output perturbations from the large diameter objective, and the perturbation from the tube lens.

As with the case of transmission 2-MGE measurements, (ref. 5) a single measurement of the 8 parameters is usually sufficient to determine the entire sample Mueller matrix at normal-incidence reflection, once the system is calibrated (see below). The Mueller matrix from reflection from an anisotropic surface is given by

$$\mathbf{M}_J = \begin{bmatrix} 1 & -C_{2\gamma}N & -S_{2\gamma}N & 0 \\ -C_{2\gamma}N & C_{2\gamma}^2 - S_{2\gamma}^2 C & C_{2\gamma}S_{2\gamma}(1+C) & S_{2g}S \\ S_{2\gamma}N & -C_{2\gamma}S_{2\gamma}(1+C) & -S_{2\gamma}^2 + C_{2\gamma}^2 C & C_{2g}S \\ 0 & S_{2\gamma}S & -C_{2\gamma}S & C \end{bmatrix} \quad \text{(A.3)}$$

where N is the diattenuation, $|N| = (R_{max}-R_{min})/ (R_{max}-R_{min})$, and S and C are given by $S = (1-N^2)^{1/2} \sin(\delta)$ and $C = (1-N^2)^{1/2} \cos(\delta)$, where δ is the retardation. The principal axis angle is given by γ, and we have used the shorthand notation: $S_{2\gamma} = \sin(2\gamma)$, $C_{2\gamma} = \cos(2\gamma)$. By comparing Eqs. A.2 and A.3, it is easy to see that by measuring the 8 coefficients shown in Eq. A.2, one can determine the diattenuation -N, the retardation δ, and the direction of the principal axis γ. If the sample has a circular diattenuation component, this will be measured by the I_{X0} and I_{X1} terms.

In certain circumstances, the sample can act to partially depolarize the light beam, and this also can be measured with the 2-MGEM2 if the depolarization is simple. For the $(\theta_{m0}, \theta_{m1}) = (0°, 45°)$ orientation of the PSG and PSA, the degree of polarization β is determined by

$$\beta^2 = I_{Y0}^2 + I_{Y1}^2 + I_{X0X1}^2 + I_{X0Y1}^2 + I_{Y0X1}^2. \quad \text{(A.4)}$$

Totally polarized light has $\beta = 1$, while partially polarized light results in $\beta<1$.

As described in refs. 2 and 3, the PEMs each have a square wave output that corresponds to the waveform of the PEMs. This signal is used in two ways. First, the signal is read by the frequency counter of the DAQ to determine the working frequency of the PEM. Second, this signal is fed into a trigger circuit that generates a pulse whenever the waveforms of the two PEMs are within ~0.3° of each other. Depending on the design of the trigger circuit, the trigger pulse can trigger on the positive slope or the negative slope of the signal coming from the PEMs. Since the PEMs are free-running, the time between trigger pulses is not constant, but rather depends in a very complicated way on the precise phase of each modulator. Using our present system, where each waveform contains 16,384 samples at 0.5 microsecond intervals (total of ~8.2 ms), we can take and analyze 4045 waveforms a second. This is equivalent to a duty cycle of ~35%.

B. Calibration of the Reflection 2-MGEM (from Ref. 1)

Calibrations of the reflection 2-MGEM2 must be performed to optimize the accuracy of the instrument, and must determine several characteristics of the various components. These include:

a) The perturbations caused by the intervening optical elements between the PSG and the sample and between the sample and the PSA.
b) The modulator amplitudes (A_{m0} and A_{m1}).
c) The static strain-induced retardation introduced by the modulators (δ_{m0} and δ_{m1}).
d) The errors in the polarizer-PEM angle from 45° (ε_{b0} and ε_{b1}).

All of these calibration parameters are determined by a calibration experiment using a high reflectivity isotropic sample, such as an aluminum mirror. Here it is assumed that the Mueller matrix of the calibration aluminum mirror is given by

$$\mathbf{M}_{Almirror} = \begin{bmatrix} 1 & -A_{Al} & 0 & 0 \\ -A_{Al} & 1 & 0 & 0 \\ 0 & 0 & -1 & D_{Al} \\ 0 & 0 & -D_{Al} & -1 \end{bmatrix}, \tag{B.1}$$

where the perturbations A_{Al}, and D_{Al} are all very small.

The Mueller matrix for the change in polarization state from the optics between the PSG and the calibration sample (this includes two directing mirrors and the focusing objective) is determined by a Mueller matrix multiplication of the Mueller matrices for two directing mirrors operating at near-normal incidence and the focusing objective. This is given by

$$\mathbf{M}_0 = \begin{bmatrix} 1 & -A_0 & -B_0 & 0 \\ -A_0 & 1 & 0 & -C_0 \\ -B_0 & 0 & 1 & D_0 \\ 0 & C_0 & -D_0 & 1 \end{bmatrix}. \tag{B.2}$$

The Mueller matrix for the optics between the sample and the PSA includes the objective and the tube lens, and has the same form as Eq. B.2, except that the subscript 0 is replaced by 1.

The composite Mueller matrix (ignoring the effects of the calibration aluminum mirror, but including all the input and output optics) is given by

$$\mathbf{M}_{composite} = \begin{bmatrix} 1 & A_t & B_t & 0 \\ A_t & 1 & 0 & C_t \\ -B_t & 0 & -1 & -D_t \\ 0 & C_t & D_t & -1 \end{bmatrix}, \tag{B.3a}$$

where

$$A_t = -(A_0 + A_1), \tag{B.3b}$$

$$B_t = -B_0 + B_1, \tag{B.3c}$$

$$C_t = -C_0 + C_1, \tag{B.3d}$$

$$D_t = D_0 + D_1. \tag{B.3e}$$

In the calibration configuration, the perturbations from the Bessel angles (A_{m0} and A_{m1}), the static strain-induced retardations (δ_{m0} and δ_{m1}), and the errors in the positioning of the polarizers with respect to the PEMs at 45° (ε_{b0} and ε_{b1}), also affect the intensities of the measured parameters. If it is assumed that the Bessel angles are close to 2.4048 and the ε_{b0} and ε_{b1} are close to 0, then 8 normalized parameters are given by

$$I_{X0} = 0, \tag{B.4a}$$

$$I_{Y0} = -2S_{m01}\,\varepsilon_{b1} + S_{m0}\,A_t - C_{m0}\,B_t - C_{m01}\,J_0(A_{m1}), \tag{B.4b}$$

$$I_{X1} = 0, \tag{B.4c}$$

$$I_{Y1} = -2S_{m01}\,\varepsilon_{b0} + S_{m1}\,A_t + C_{m0}\,B_t - C_{m01}\,J_0(A_{m0}), \tag{B.4d}$$

$$I_{X0X1} = 1, \tag{B.4e}$$

$$I_{X0Y1} = -S_{m1}\,C_t + C_{m01}\,\delta_{m0} + \delta_{m1} + C_{m1}D_{t,} \tag{B.4f}$$

$$I_{Y0X1} = -S_{m0}\,C_t + C_{m01}\,\delta_{m1} + \delta_{m0} + C_{m0}D_{t,} \tag{B.4g}$$

$$I_{Y0Y1} = -C_{m01.} \tag{B.4h}$$

The azimuthal angles of the PSG and the PSA are θ_{m0} and θ_{m1}, and the following short-hand notation is used:

$$S_{m0} = \sin(2\,\theta_{m0}), \tag{B.5a}$$

$$C_{m0} = \cos(2\,\theta_{m0}), \tag{B.5b}$$

$$S_{m1} = \sin(2\,\theta_{m1}), \tag{B.5c}$$

$$C_{m1} = \cos(2\,\theta_{m1}). \tag{B.5d}$$

The composite difference angle gives

$$S_{m01} = \sin(2\,(\theta_{m0} - \theta_{m1})), \tag{B.5e}$$

$$C_{m01} = \cos(2\,(\theta_{m0} - \theta_{m1})). \tag{B.5f}$$

Calibration data is collected from 4 different orientations of the PSG and PSA [(0°, 0°); (0°, 45°); (45°, 0°); (45°, 45°)] from which the perturbations A_t, B_t, C_t, and D_t can be determined, as well as the modulator parameters A_{m0}, A_{m1}, δ_{m0}, δ_{m1}, ε_{b0} and ε_{b1}. In addition, the total gain factor of the system can be determined from the I_{X0X1} term and the relative angle (θ_{m0} - θ_{m1}) can be determined from the I_{Y0Y1} term.

These values of the perturbation matrix can then be used to correct a single zone Mueller matrix element measurement of an unknown sample, resulting in a complete determination of the sample Mueller matrix under the assumptions of the symmetries shown in Eq. B.3. Unfortunately, it is not possible to determine the individual perturbation elements of the input and output optics from these measurements; that is, one can determine A_t but not A_0 and A_1 separately. In this case, one is forced to make the assumption that $|A_0| = |A_1|$, etc. This is equivalent to assuming that the two directing mirrors have a negligible effect on the input polarization state (quite reasonable for small angles of incidence, see Table I), that the tube lens has a minimal effect on the reflected polarization state (quite reasonable if the light beam goes through the center of the tube lens), and that the large-diameter objective is cylindrically symmetric and the light beam enters and exits equidistant from the center of the objective. This last assumption is the most severe.

References

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2. G. E. Jellison, Jr. and F. A. Modine, "Two-Modulator Generalized Ellipsometry: Experiment and Calibration," *Appl. Opt.* **36**, 8184–8189 (1997).
3. G. E. Jellison, Jr. and F. A. Modine, "Two-Modulator Generalized Ellipsometry: Theory," *Appl. Opt.* **36**, 8190–8198 (1997).
4. G. E. Jellison, Jr. and F. A. Modine, "Two-modulator generalized ellipsometer for complete Mueller matrix measurement," U. S. Patent No. 5956147 (1999).
5. G. E. Jellison, Jr., C. O. Griffiths, D. E. Holcomb, and C. M. Rouleau, "Transmission 2-modulator generalized ellipsometry (2-MGE) measurements," *Appl. Opt.* **41**, 6555–6566 (2002).

2-MGEM2 Data Analysis Manual

Operating Instructions

The two-modulator generalized ellipsometry microscope (2-MGEM2) is the second generation of the 2-MGEM, which is a polarization optical microscope operating in reflection mode used to determine the light polarization characteristics of a reflecting surface. The instrument is capable of determining the optical linear and circular diattenuation, retardation, direction of the fast axis, and depolarization of a small area on a polished surface. Maps of the various parameters are made by rastering the analyzed area to cover region of interest. The primary application of this instrument is to characterize the structure of the inner and outer pyrocarbon layers of TRISO nuclear fuel. Data taken with the previous generation 2-MGEM is entirely compatible with this software.

4/22/2013

Table of Contents

Acronyms

2-MGE	Two modulator generalized ellipsometer
2-MGEM	Two modulator generalized ellipsometry microscope
2-MGEM2	Second generation 2-MGEM
Buffer	Inner layer of a TRISO nuclear fuel particle consisting of porous graphite
CD	Circular diattenuation
DePolF	Depolarization Fraction = 1 - β
FAA	Fast axis angle
IPyC	Inner pyrocarbon layer between the buffer and SiC in a TRISO particle
OPTAF	Optical anisotropy factor = (1 + N) / (1 - N)
OPyC	Outer pyrocarbon layer of a TRISO particle
PEM	Photoelastic modulator
PolF	Polarization factor
PSA	Polarization state analyzer
PSG	Polarization state generator
PMT	Photomultiplier tube
Rel FAA	Relative direction of the fast axis with respect to the radius of the TRISO particle
SiC	Silicon carbide. The structural layer of a TRISO particle
SL	Saved lists. Used to select regions to be analyzed
TRISO	Tristructural isotropic nuclear fuel particle, (Kernel, Buffer, IPyC, SiC, OPyC)

Symbols

I_{X0}, I_{Y0}, I_{X1}, I_{Y1}, I_{X0X1}, I_{X0Y1}, I_{Y0X1}, I_{Y0Y1}	2-MGE parameters
β	Polarization factor, $\beta = (N^2 + S^2 + C^2)^{1/2} <= 1$
N	Diattenuation, $N = (R_{max} - R_{min}) / (R_{max} + R_{min})$, a Mueller matrix element
δ	Retardation
S	$S = (1 - N^2)^{1/2} \sin(\delta)$, A Mueller matrix element
C	$C = (1 - N^2)^{1/2} \cos(\delta)$, A Mueller matrix element
γ	Direction of the fast axis

Introduction

This manual describes the operation of the data analysis software used to process data from the two-modulator generalized ellipsometry microscope. The generation 2 version (2-MGEM2) is described, but this software can also be used to process generation 1 version (2-MGEM) data. For the current system, data is acquired according to the instructions in the 2-MGEM2 Operation Manual. The data analysis software described in this manual may be run on a desktop computer independently from the software used to control the instrument.

Operation of the Program

The opening screen (Figure 1) shows the name of the program, the author, the last date of revision, and the present version number. Press *TRISO Data* to continue or *Close* to exit the program.

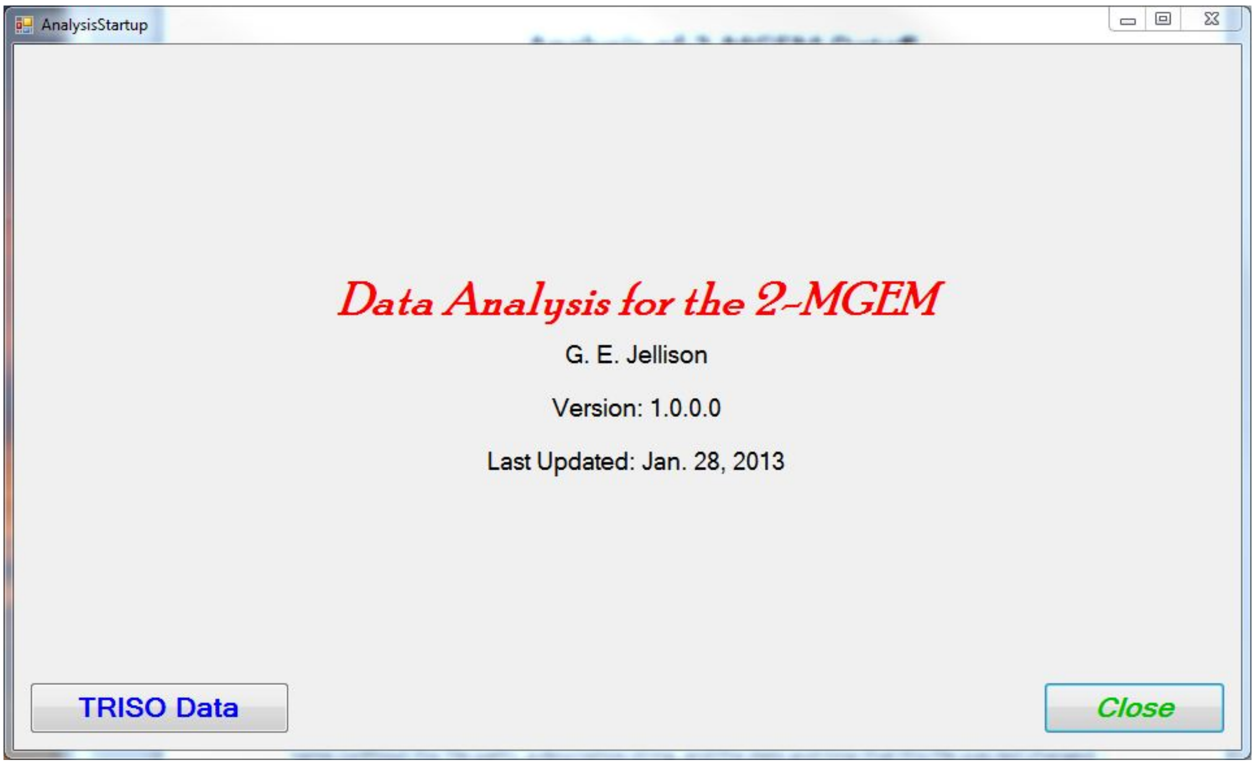


Figure 1. The opening screen for the Data Analysis software package.

Entry of Data and Plotting

The window entitled *2-MGEM2 Data Analysis* is presented next. Enter data for processing by pressing *Load File* at the bottom of the window (Figure 2). This will bring up the standard dialog box for loading a file. This dialog is defaulted to list only the header files (*.hdr) for the 2-MGEM2 data, although all the data will be loaded along with the header file. Once the file is selected, the data will be loaded into the computer, along with the file name (without the file path), a descriptive string, and the date and time that this file was last changed. The intensity map is also initially plotted in the window, using the color scale, shown to the upper right of the plotted data. If the cursor is placed over the plotted data, the

X and Y values of the position of the cursor will appear in the box labeled *Parameters*, as well as the value of that pixel for the selected parameter.

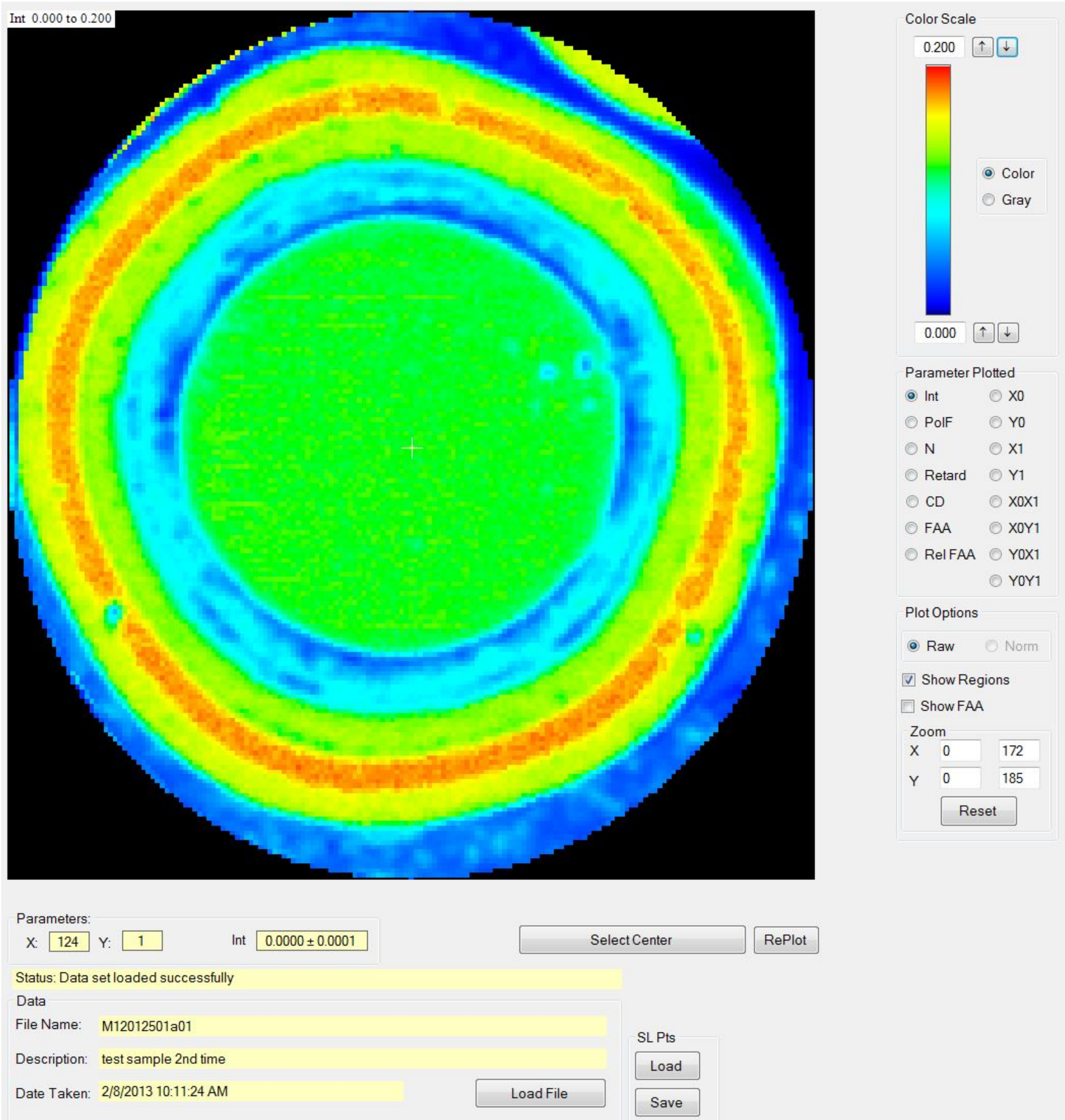


Figure 2. The left side of the 2-MGEM Data Analysis window, showing data entry and the initial intensity plot of the data file.

There are three different group boxes used to alter the calculation and/or the displayed map. These are described below.

Color Scale

This box shows the color scale used to plot the data. There are two options, *Color* or *Gray*, which can be selected using the associated radio buttons. The scale is initially defined by the maximum and minimum values of the associated parameter shown in the upper and lower text boxes, respectively. These can be changed by using the up and down arrows to the right of the text box.

Parameter Plotted

There are 15 different parameters that can be displayed in the plotted map, and later analyzed. Selection is made by pressing the appropriate radio button in the box. The parameters that can be selected are:

a) *Int* The intensity of light reflected from a given pixel
b) *DePolF* The depolarization factor for the given pixel. 0=> no depolarization.
c) *N* The diattenuation, $N = (R_{max}-R_{min})/(R_{max}+R_{min})$
d) *Retard* The retardation angle (in radians), $\delta = \delta_{Rmax}-\delta_{Rmin}$
e) *CD* The circular diattenuation
f) *FAA* The fast axis angle
g) *Rel FAA* The fast axis angle relative to the radius of the plot from the center
h) *X0, Y0, X1, Y1, X0X1, X0Y1, Y0Y1, Y0Y1*: The raw 2-MGE parameter data.

Plot Options

Several plot options can be selected. The raw data (*Raw*) or the normalized data (*Norm*) can be selected using the appropriate radio button (Figure 2). Likewise, the user can include the regions of the saved lists and/or a line representing the fast axis angle (*FAA*) on the plot by checking the appropriate box. These functions are described in more detail in the next section. There is also a *Zoom* option, which allows the user to select a smaller portion of the data, which can then be expanded in the plotting area. To do this, enter the pixel numbers for the maximum and minimum X and Y to be plotted, and press *RePlot*.

Once the data is entered, the default for the center of the data is the center of the figure. This can be changed by selecting the button *Select Center*. After selecting this button, the cursor can be placed over the plotted data, and the cursor arrow will change to a cross. Placing the cross over the pixel that is to be selected as the center of the data and left-clicking will change the center of the data.

Analysis of Data

To analyze the data, the regions of data that are to be to be grouped together must be selected. This is done by setting saved lists, which are then used to determine regions of data to be grouped and jointly analyzed. The data for the saved lists (*SL*) and regions is shown in a table to the right of the window (Figure 3). The first four regions are pre-named as *Buffer*, *IPyC*, *SiC*, and *OPyC* for the 4 primary layers of TRISO nuclear fuel. The names can be changed by clicking the appropriate box and typing in the new name. Three (3) additional regions can also be selected and names can be entered in the appropriate box. The saved lists are numbered 0-6, with the color code is shown.

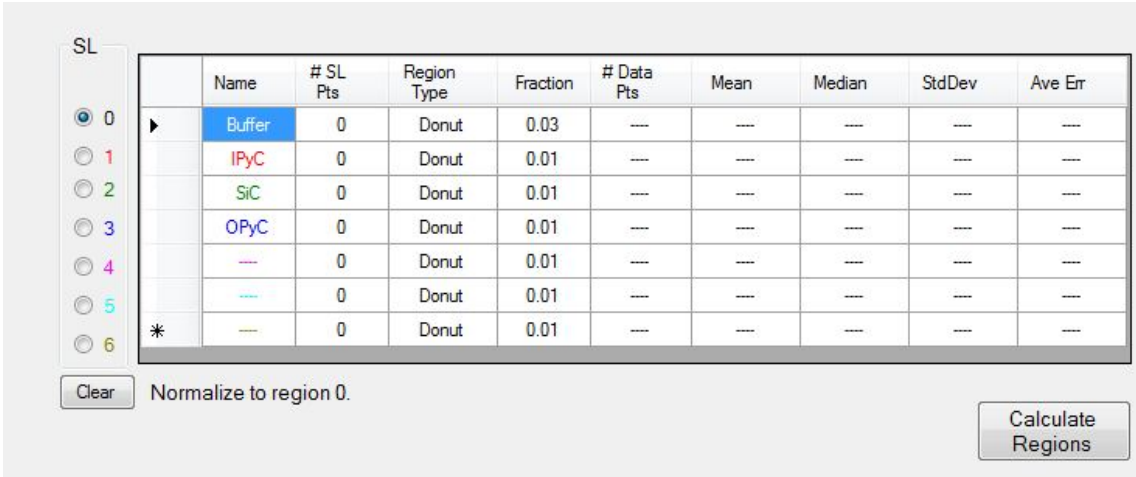


Figure 3. Saved Lists selection plus the table that will include the calculated data. These are Located on the right side of the 2-MGEM Data Analysis window.

Saved lists can be generated in two ways. If the data set has been previously analyzed, then the saved lists can be loaded by pressing the *Load* button in the *SL Pts* box (see the bottom of Figure 2). Alternately, the saved lists can be generated manually by individually selecting the points; this will always have to be done to analyze data not previously analyzed.

To generate a saved list manually, select the saved list using the appropriate radio button to the left of the table (Figure 3), then hover the cursor over the plot of the data and select a pixel by left-clicking; a small circle will appear around the pixel that has been selected and a number placed next to it. The pixel can be removed later by right-clicking inside the small circle. Continue to select points in this manner to define the region of data that will be grouped together for analysis; the number of points in each saved list will appear in the table. Saved List 0 is reserved for the region that will be used to normalize the data (usually the Buffer region for TRISO fuel).

The regions are determined using the Saved Lists points and by selecting the *Region Type* and the *Fraction* in the data table. The region type is changed by clicking on the appropriate box in the table, thereby cycling through the region type options. The *Fraction* is used only by the donut region type and is the value that determines the width of the donut as a fraction of full scale; this number can be changed by clicking on the appropriate box and typing in a different fraction. Presently, there are three region types that can be selected:

a) *Donut*. This region type resembles a distorted donut, such as might be used for one of the layers of TRISO nuclear fuel. The center of the donut is the selected center, shown by the white cross in the plot of the data. The central annulus of the donut is determined from the associated saved list, using a Spline routine to interpolate between the saved list points. The fraction parameter is used to draw the inner and outer boundaries of the donut, and is the fraction of full-scale.
b) *Points.* This region is defined only by the selected saved list points; no extra points are included in the region.
c) *Polygon.* This region is defined by a polygon connecting the saved list points. The polygon is closed, so any point within the polygon is included in the region.

Once the saved lists have been selected, the regions can be calculated using the *Calculate Regions* button below the table. This will draw the saved list points and the regions on the plotted data (Figure 4), update the table, and draw the appropriate histogram (Figure 5).

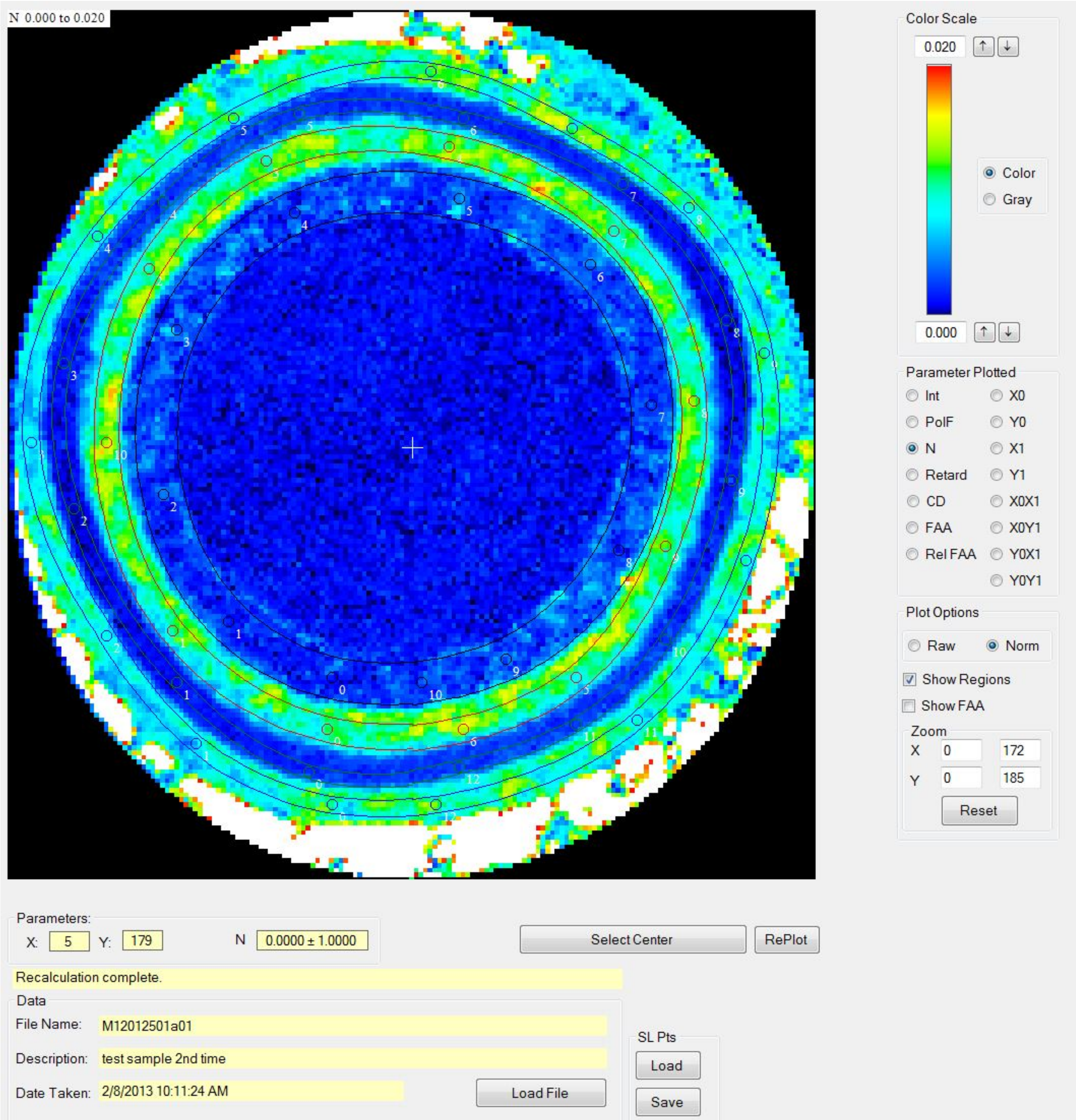


Figure 4. A map of the diattenuation with the selected list points indicated by small circles. The lines show the donut-type regions determined from the selected lists points.

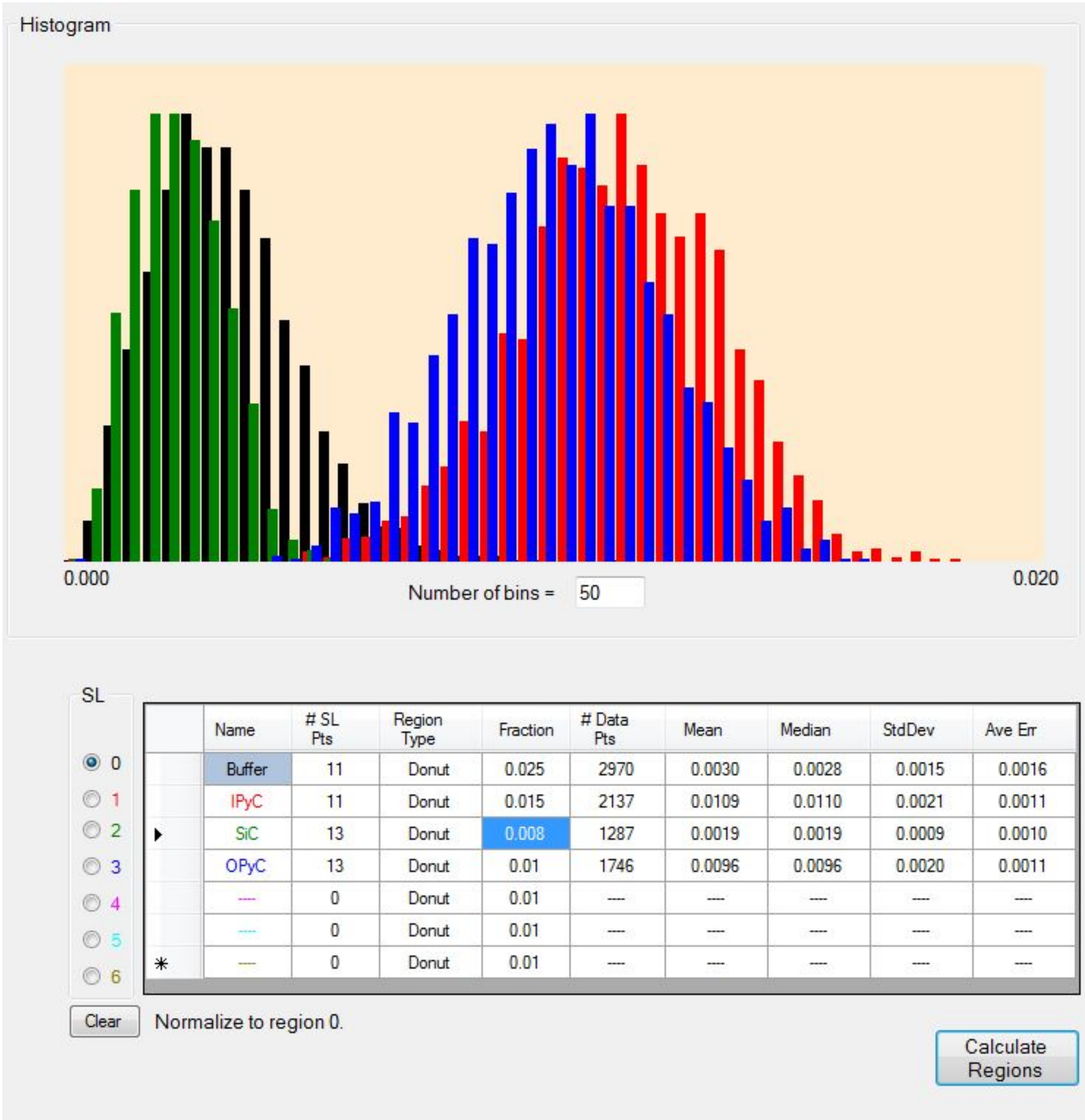


Figure 5. Histogram of the data and the table of data for the 4 selected regions.

The SL table (Figure 5) contains the relevant data for the selected regions for the parameter selected using the “Parameter” box. The elements in the data table are:

a) *Name* — The name of region
b) *# SL Pts* — The number of points in the saved list
c) *Region Type* — *Donut*, *Points*, or *Polygon*; described above
d) *# Data Pts* — The number of data points in the region
e) *Mean* — The average of all the points within the region for the parameter selected
f) *Median* — The median of all points within the region
g) *StdDev* — The standard deviation of all points within the region
h) *Ave Err* — The average error of the all the points in the region.

The histogram of the associated data in each region for the parameter plotted is shown above the data (Figure 5). Each region is color-coded based on the colors shown in the box labeled *SL*, and is consistent in the table, histogram, and the plotted regions in the map.

Once the regions have been calculated, the data can be normalized to region 0 and the *Norm* button becomes activated. At this point, it is possible to plot either the raw data without normalization or the normalized data. As this is changed, the histogram and the data in the SL table will be updated. Similarly, if the *Plot Options/Parameter* is changed, the data plot, the histogram, and the data table will be changed to reflect this choice.

There are two options for data output, selected by buttons at the bottom right of the window (not shown).

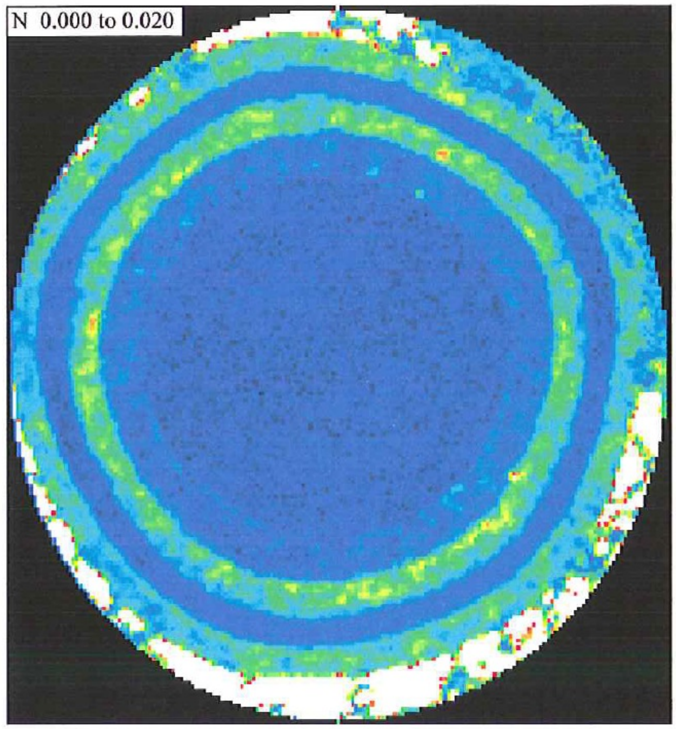
a) *Save Picture* Saves the present plotted data to a *.jpg file.
b) *Print Summary* Prints a summary of the data in the table to the default printer. An example is shown in Figure 6.

Figure 6 shows a sample printout from the data analysis. The file name, description and data taken date and time are the same as those in the Data Analysis window (Figure 2). The Analysis Date/Time entry is the date and time that the analysis was performed, and the present software version number is also given. The plotted data is the current selected parameter in the Data Analysis window, without the selected regions. The data table below the figure shows the selected regions, four for this case (Buffer, IPyC, SiC, and OPyC). The *NPts* are the number of points within the selected region. The averages of several parameters in each of the selected regions are also shown, as well as the standard deviations (*Std Dev*) and the average errors (*Ave Err*). The print out also lists the value of the optical anisotropy factor, *OPTAF* = (1+N)/(1-N) = R_{max}/R_{min}, and the direction of the relative fast axis (*Rel Fast Axis*). This last parameter is the direction of the fast axis relative to the radius of the particle, determined by the central point of the particle. This should be close to 90°, and the standard deviation is then a measure of the crystallographic organization of the region. If this value is close to ~52°, then there is no organization at all (the graphite nanocrystals are randomly oriented); if it is close to 0, the nanocrystals are preferentially oriented perpendicular to the radius of the TRISO particle.

TRISO particle anisotropy analysis

File Name: M12012501a01
Description: test sample 2nd time

Data Taken: 2/8/2013 10:11:24 AM
Analysis Date/time: 4/2/2013 10:17:50 AM
2-MGEM Version: 1.0.0.0



	Buffer	IPyC	SiC	OPyC
NPts	2970	2137	1287	1746
Diattenuation	0.0030	0.0109	0.0019	0.0096
Std Dev	0.0015	0.0021	0.0009	0.0020
Ave Err	0.0016	0.0011	0.0010	0.0011
OPTAF	1.0060	1.0221	1.0038	1.0194
Std Dev	0.0030	0.0042	0.0018	0.0041
Ave Err	0.0032	0.0022	0.0020	0.0022
Retardation	-0.0017	-0.0023	0.0001	-0.0027
Std Dev	0.0028	0.0022	0.0026	0.0024
Ave Err	0.0036	0.0032	0.0027	0.0037
Rel Fast Axis	87.3	89.7	86.6	89.4
Std Dev	25.3	6.2	30.5	7.0
Ave Err	10.2	3.9	8.2	4.1
DePol Fraction	0.0115	0.0073	-0.0059	0.0005
Std Dev	0.0125	0.0131	0.0105	0.0124
Intensity	0.0695	0.1333	0.1727	0.1400
Std Dev	0.0135	0.0066	0.0075	0.0098

Figure 6. Printout example of the analyzed data from a polished cross section of a TRISO nuclear fuel particle .

Background

The 2-MGEM2 is based on the two-modulator generalized ellipsometer (2-MGE) [refs. 1–4] and is described in detail in ref. 5. Briefly, the instrument is arranged as a reflection microscope with a single large-aperture objective acting as both the condensing lens and the objective. The input beam passes through the polarization state generator (PSG), which consists of a polarizer photoelastic modulator (PEM) pair placed before the objective. The reflected beam, after passing through the objective, passes through the polarization state analyzer (PSA), which also consists of a polarizer PEM pair. The two PEMs are operated at different frequencies (50 and 60 kHz in this case). The light intensity is detected as a function of time using a photomultiplier tube, which is then digitized at 0.5 μs/point. The light source is a mercury arc lamp, which is filtered at 577 nm (one of the primary emission lines of the mercury lamp). The intervening large-aperture objective does perturb the polarization measurement, but many of the effects can be calibrated out (see ref. 5). The optical resolution of the experiment is determined by the optical elements after the sample and the pinhole in front of the photomultiplier tube (PMT). The common resolution is 6–7 μm, but some data [refs. 6–7] has been taken using an optical resolution of ~4 μm.

The time-dependent intensity is a complicated function of time, but can be expressed as [refs. 1–4]

$$Intensity\,(t) = I_{dc} + I_{X0}\,\mathrm{X0} + I_{Y0}\,\mathrm{Y0} + I_{X1}\,\mathrm{X1} + I_{Y1}\,\mathrm{Y1} +$$

$$I_{X0X1}\,\mathrm{X0\,X1} + I_{X0Y1}\,\mathrm{X0\,Y1} + I_{Y0X1}\,\mathrm{Y0\,X1} + I_{Y0Y1}\,\mathrm{Y0\,Y1}. \qquad (1\text{ a})$$

The terms I_{dc}, I_{X0}, I_{Y0}, etc. are coefficients that multiply the basis functions:

$$\mathrm{X0} = \sin(A_0 \sin(\omega_0 t)) \qquad \mathrm{Y0} = \cos(A_0 \sin(\omega_0 t)), \qquad (1\text{ b, c})$$

$$\mathrm{X1} = \sin(A_1 \sin(\omega_1 t)) \qquad \mathrm{Y1} = \cos(A_1 \sin(\omega_1 t)). \qquad (1\text{ d, e})$$

The basis functions are not the common Fourier basis functions, but rather sines and cosines of sines. The modulator amplitudes (A_0 and A_1) are measured in angular units (usually radians) and the modulator frequencies are given by ω_0 and ω_1. The intensity is normalized to the average intensity (dc) level, and this is done both in hardware and software.

The coefficients of the basis functions are the measured quantities in the experiment, and are functions of the azimuthal orientation of the PSG and PSA. For the case where the PSG is oriented at 0° with respect to the measurement system and the PSA is oriented at 45°, then the 8 coefficients are

$$\mathrm{I_{X0}} = \mathrm{CD} \qquad \mathrm{I_{X1}} = -\mathrm{CD} \qquad (2\text{ a, b})$$

$$\mathrm{I_{Y0}} = \sin(2\gamma)\,N \qquad \mathrm{I_{Y1}} = -\cos(2\gamma)\,N \qquad (2\text{ c, d})$$

$$\mathrm{I_{X0X1}} = -C \qquad \mathrm{I_{Y0X1}} = -\cos(2\gamma)\,S \qquad (2\text{ e, f})$$

$$\mathrm{I_{X0Y1}} = \sin(2\gamma)\,S \qquad \mathrm{I_{Y0Y1}} = \cos(2\gamma)\sin(2\gamma)\,(1 + C). \qquad (2\text{ g, h})$$

For TRISO particles, the two most important parameters are the diattenuation N and the direction of the principal axis γ, which is referenced to the measurement system. Therefore, it is critical for the measurement system to determine the parameters I_{Y0} and I_{Y1} as accurately as possible. For other applications, such as Pockels effect measurements of internal electric fields (see ref. 8), the retardation δ is the important parameter. The retardation is determined from the *S* and *C* parameters, $S = (1-N^2)^{1/2} \sin(\delta)$ and $C = (1-N^2)^{1/2} \cos(\delta)$, which are determined from I_{X0X1}, I_{X0Y1}, and I_{Y0X1}. These three values, *N*, *S*, and *C,* are not independent, since $N^2 + S^2 + C^2 = 1$. However, if the sample depolarizes the light beam, then the sum $N^2 + S^2 + C^2 = \beta^2 < 1$. Often it is useful to show the Polarization Factor = 1 - β. If this is 0, then there is no depolarization of the light beam induced by the sample.

A typical TRISO coated particle sample will contain an array of particles placed in an epoxy holder, which is then polished down to expose the midplane of the TRISO particle. 2-MGEM2 measurements are then made point-by-point using a spatial resolution less than the optical resolution (typically 5 μm, but occasionally 2.5 μm). Since the particle is typically 0.8–1 mm in diameter, this will result in 25,000 to 40,000 data points, each of which will include the intensity of the light from the particle at that point and the 8 measured 2-MGEM2 parameters given in Eqs. 2a-h, and their errors. The derived parameters, such as the diattenuation N, the retardation δ, the fast axis angle γ, the circular diattenuation CD, and the polarization factor, are then determined from the raw data.

The most important parameter for TRISO particle characterization is the optical diattenuation N. For optically anisotropic materials, the reflectivity will depend on the polarization state of the light beam. The diattenuation is defined as

$$N = \frac{R_{\max} - R_{\min}}{R_{\max} + R_{\min}}, \tag{3}$$

where R_{max} (R_{min}) is the maximum (minimum) reflectivity for orthogonal polarization states of the incident light. The diattenuation is then directly related to the optical anisotropy factor:

$$OPTAF = \frac{1+N}{1-N} \sim 1 + 2N, \tag{4}$$

where the first-order Taylor expansion is used to obtain the second expression for small N. Clearly, these expressions represent an integration over the sampled region (~7 μm for the standard resolution).

For pyrolytic carbon, optical anisotropy originates from the preferential orientation of nanocrystalline graphite. Since the sampled region is considerably larger than the individual graphite nanocrystals, the optical response will be an integration over all the nanoparticles within the sampled region. If the graphene planes of the individual graphite nanoparticles are preferentially oriented, then the reflectivity will be polarization-sensitive (that is, optically anisotropic), while a totally random collection of graphite particles will result in no observable optical anisotropy after integration over the sampled region.

Normalization

The 2-MGEM2 is designed to operate at near-normal incidence, where the angle of incidence is 3–5°. The minor deviation from normal incidence results in a small residual diattenuation [ref. 5]. As a result, the two diattenuation-sensitive parameters (I_{Y0} and I_{Y1}) experience a small bias in their values, which must be subtracted out to get the most accurate diattenuation measurements.

For TRISO particles, any real preferred orientation is expected to be related to the growth direction (along the radius of the particle). Therefore, in the image of the particle, the preferred orientation will rotate 360° as one goes around the particle. In contrast, the bias for a non-zero angle of incidence will be dependent only on the angle of incidence and the complex refractive index of the material and will not be dependent upon the growth direction. This suggests a technique for compensating for the angle-of-incidence effect for materials that have near-circular symmetry: A large donut-like region of the buffer is selected and the averages of the measured values of I_{Y0} and I_{Y1} (as well as I_{X0}, I_{X1}, I_{X0Y1}, I_{Y0X1}, and I_{Y0Y1}) are calculated. The data for the entire image are then normalized for this small offset by subtracting off the average. The buffer region is selected because it is expected that its diattenuation will be considerably smaller than the diattenuation of the IPyC or the OPyC, but its complex refractive index will be similar to that of the IPyC and OPyC layers. The SiC layer may also be a candidate for this procedure, but the complex refractive index of the SiC may be considerably different from that of the pyrocarbon layers.

Diattenuation N

The diattenuation and the direction of the principal axis can be determined from only the I_{Y0} and the I_{Y1} terms in Eqs. 2c and 2d. The diattenuation and the direction of the fast axis are given by

$$N = \sqrt{I_{Y0}^2 + I_{Y1}^2}\,, \tag{5a}$$

and $$\tan(2\gamma) = -\frac{I_{Y0}}{I_{Y1}}\,. \tag{5b}$$

The determination of γ must use the sign values of I_{Y0} and I_{Y1} to allow a determination from 0° to 180°. Smaller values of N will necessarily result in more inaccurate determinations of γ, where in the limit of N = 0, γ becomes indeterminate.

For a typical pyrocarbon layer, the diattenuation will be 0.007–0.0140. The SiC layer will have a very low diattenuation (usually <0.002), while the buffer layer will have an intermediate diattenuation (typically ~0.003). The direction of the fast axis γ will be well-determined in the IPyC and OPyC, and will be perpendicular to the radius of the particle with a standard deviation of ~6° to 10°. The SiC and buffer layers will have a much larger standard deviation of the fast axis angle. See ref. 7 for more detail.

To get a quantitative measure of the quality of the diattenuation measurement, the depolarization factor (1 - β) is also determined. Significant depolarization occurs when this factor is greater than ~0.01. Surface imperfections, such as scratches, pits and pores can lead to significant depolarization, as can rounding of the outside edge of the OPyC. The procedures used to grind and polish a specimen are important for optimizing the measurement. Methods that produce a well-polished surface without sacrificing the flatness needed for collection of the reflected light are preferred.

The error of the diattenuation can also be calculated by propagating the individual errors of I_{Y0} and I_{Y1} using Eq. 5. This is important because the measured diattenuation N, as determined from Eq. 5, will always be biased toward a positive quantity, even though the true value is zero. Therefore, a measured value of N that is less than approximately twice the error cannot be considered different from zero.

Measurement of the Relative Direction of the Principal Axis

To quantify the orientation of the principal axis, we define the relative principal axis angle γ_r, which is the principal axis angle γ with respect to the radial vector of the TRISO particle. If the particle is spherical, then perfect alignment results in $\gamma_r = 90°$. If the fraction of the pyrocarbon layer producing the optical diattenuation is well-organized, then γ_r will be close to 90°, and the standard deviation of γ_r [SD(γ_r)] for a collection of points within the layer will be small. If the layer is not well organized, the average γ_r may still be close to 90°, but the SD(γ_r) for a collection of points within the layer will be large. (A similar increase in SD(γ_r) can be expected from the non-spherical nature of real TRISO particles.) It can be shown (ref. 7) that a totally uncorrelated collection of relative principal axis angles (corresponding to a random collection) will have a SD(γ_r) = 52.0°. Therefore, the SD(γ_r) becomes a quantitative measure of the degree of organization within the pyrocarbon layers: perfect organization in perfectly spherical particles results in SD(γ_r) = 0°, perfect disorganization results in SD(γ_r) = 52.0°. Care must be taken when interpreting data from a TRISO particle with a significantly non-spherical shape, since this can also contribute to SD(γ_r).

Assignment of Regions and Histograms

The data taken on each TRISO particle consists of ~25,000 to 40,000 points, and each pyrocarbon layer will consist of ~>1,500 data points. This large number of data points dramatically improves the reliability and accuracy of the 2-MGEM2 measurements over previous methods used to measure optical anisotropy, but this does require some significant statistical analysis. To do this, regions are defined over the sample surface to be analyzed as a single data set. There are three ways of determining regions allowed in the current 2-MGEM2 Data Analysis software: 1) Donut, 2) Points, and 3) Polygon. The donut region is used most often in the analysis of TRISO nuclear fuel particles. As the name implies, this region is in the shape of a donut, although the inner and outer regions are not quite circles. The points method simply analyzes all the points that the user selects, while the polygon method analyzes all the points included within a polygon as defined by the user.

TRISO particles are not perfectly spherical, so the cross-section will expose the layers as near-circles, but not as perfect circles. To take this into account, the following procedure has been developed to define a region to correspond to one of the layers in a polished cross section of TRISO nuclear fuel.

a) Select 8–15 points around each layer located near the center of that layer. Given the non-uniformity of the particles, this is best done by hand.
b) Convert these points to polar coordinates and sort the points by polar angle.
c) Perform a spline interpolation.
d) Select a width for the region of interest (usually 2–4% of the total image size for the buffer layer and 0.8–1.5% of the total image size for the IPyC, SiC and OPyC layers); this defines a region

between two modified concentric circles, where all the points within the region lie within the selected layer.

e) Plot the two modified concentric circles on the intensity image for a visual check that only points from the layer of interest are included.

In the current 2-MGEM2 Data Analysis software, step 1 is performed by hand, while steps 2–5 are performed by the computer, after certain defining parameters are input. It is then possible to perform a number of statistical manipulations on the groups of data thus selected.

One of the more useful presentations of the data is the histogram, illustrated in Figure 5. This figure shows the histogram of the diattenuation for all three layers. Clearly, the IPyC has the highest average diattenuation, followed closely by the OPyC. However, both regions show a rather large distribution of these values. While the average diattenuation of the IPyC is 0.0109, there are some pixels that register a diattenuation of close to 0.018, while others show a diattenuation of 0.004. Since the standard deviation of the diattenuation of the IPyC is 0.0021, while the average error is 0.0010, the distribution of the values of the diattenuation is due to fluctuations in the IPyC layer itself, and not due to stochastic variations in the measurement.

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