CHARACTERIZATION OF SEMICONDUCTORS
BY PHOTOLUMINESCENCE USING MICROSCOPY

Semiconductor characterization depends on photoluminescence.

Photoluminescence of Semiconductors

Characterization of Semiconductors with Photoluminescence Measurement System

Photoluminescence Characterization of GaN Alloys and Other Semiconductor Microstructures

High-Resolution Low-Temperature PL of Semiconductors

III-V Wafer Characterization through Photoluminescence Mapping

Room-temperature Micro-electroluminescent Characterization of Ge-based IR Sources

The MicOS Microscopespectrometer

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**Integrated Micro and Macro Photoluminescence**

**Micro-PL measurements**

**Macro-PL measurements**

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You couldn’t live without semiconductors these days. It’s the engine that drives LED lights, computer displays and other technologies. Most electronics employ semiconductors, most notably computer chips.

Photoluminescence (PL) is a powerful tool for semiconductor characterization in the various stages in its life cycle. That includes development, testing, quality control, and failure analysis. Most modern semiconductor devices are engineered materials made from multilayered structures fabricated on wafers. These are then diced up into individual devices. The process of engineering the base material, fabricating the wafers and characterizing the devices made from these wafers all depend on techniques like PL.

Photoluminescence

Photoluminescence phenomena result from materials absorbing excitation light photons and getting raised into an excited state. In the case of semiconductors, these levels are typically above the bandgap of the material. When the excited species relax, it releases this excess energy in the form of luminescence or emission of photons. The emitted light is often characteristic of either the material or its surrounding environment, and can even provide information about local dynamics around emitting species.

PL is entirely a photon mediated process, so it is a non-contact and non-destructive method of probing materials. Therefore, manufacturers can integrate PL into the production process without destroying or contaminating the tested sample.
Semiconductor characterization depends on photoluminescence

Photoluminescence investigations

Modern semiconductors are highly engineered materials designed to exhibit certain defined behaviors. These materials are usually made on various substrates by epitaxial processes that stack different material layers one over another. Engineers build semiconductors from stacks of various atoms. This process has very tight tolerances, and PL is a tool that can be used to verify what is being made will perform as expected. The way you stack up the atoms, and which atoms you stack up, determines the function of what you ultimately get, from solar cells to LEDs.

For example, the LED lights in a home supply store have different colors. Some are bright white, and others have a yellowish tint. That is all materials engineering, and PL helps determine which parts of the wafer are fit for the various light sources.

PL offers unique signatures, providing operators with information on quality and other characteristics. It allows device designers and manufacturers to determine, prior to the manufacturing process, which parts of the wafer meet the functional requirements of the intended device before they are actually made. Once made, failure of a device to meet performance expectations can be very costly – so it is a lot cheaper to determine and weed out non-performing devices early on in the process. PL is a key technique for doing this.

Photoluminescence is also widely used for defect analysis in semiconductor analysis. Defects are usually foreign dopants that are embedded into a host material matrix – by design or accident, or, they can be structural deformations of the material itself. In either case, these defects affect the band structure of the material in which they occur. Since PL is really a measure of the band structure of the material, it therefore serves as a useful tool for defect analysis both in material engineering and device fabrication and quality control...
A typical PL mapper, such as the HORIBA MicOS PL mapper, works by scanning a focused excitation laser beam over a wafer or device and collecting the full PL spectrometer at thousands to millions of points across the structure. Various parameters of the PL emission can be displayed, as shown in figure 1.
Semiconductor characterization depends on photoluminescence

Photoluminescence at various fabrication stages

Foundational to any semiconductor device endeavor is the material engineering effort, to ensure that the materials used in the device exhibit the right properties that support the expected device performance. This work often boils down to band structure engineering, so PL is obviously a key analytical technique at this stage.

At the pre-production level, manufacturers use PL to fine tune the actual wafer fabrication process by characterizing such considerations as the homogeneity of the deposition process or presence and location of defects introduced intentionally or otherwise.

Technicians use PL at the end stage to do quality control to ensure that device performance is consistent across different fabrication batches. PL is also used to check and maintain the stability and robustness of the fabrication process itself – a tool to monitor and ensure the often tight tolerances required for correct device fabrication is maintained.

Finally, PL is also key in the analysis of failed devices, still part of quality control. A device may fail in the field. A section of your monitor might not appear correctly. It could happen on an industrial level, or even for an agency like NASA, where device failure can be very expensive. Researchers use PL to understand why the component failed, so they can correct it in the development or manufacturing stages.

HORIBA offers the MicOS Microscope Optical Spectrometer, part of its Standard Microscope Spectroscopy Solutions (SMS), to carry out these and other analyses. It is a modular, versatile and cost effective microspectrometer platform for steady state and lifetime photoluminescence.

The MicOS combines a custom high throughput microscope head with a high-performance, triple grating, imaging spectrometer that can accommodate up to three different detectors. HORIBA can customize the MicOS with various light sources and detectors to meet the requirements of the research at hand. The MicOS merges microscopy and photoluminescence spectroscopy, to provide optimal coupling from the sample, all the way to the detector.
Introduction

Photoluminescence spectroscopy (PL) is a powerful optical method used for characterizing materials. PL can be used to find impurities and defects in silicon and group III-V element semiconductors, and to determine semiconductor band-gaps. A material absorbs light, creating an electron-hole pair; an electron from the valence band jumps to the conduction band leaving a hole. The photon emitted upon recombination corresponds to the energy-difference between the valence and conduction bands, and is hence lower in energy than the excitation photon.

Features

- 500 to 2000 nm operation
- Integrated sample optics and viewer
- Precision xy sample positioning
- Specifically designed for use with cryostat
- Integrated data-collection and analysis

Benefits

- Unique system for continuous monitoring from visible to IR without realignment
- Optimize collected signal and accurately target sample
- Automated mapping facility
- Ambient and low-temperature capabilities
- Completely automated PL system
Experimental setup

In order to measure photoluminescence of semiconductors, there are various requirements: (a) a stable, powerful monochromatic light source, (b) optics to focus light on the sample, (c) sample holder, (d) collection optics, (e) monochromator, and (f) detector for spectral analysis. Actual sample excitation and collection optics used depend on the type of samples and experimental conditions required. For some samples, excitation and collection are optimal at 90°. In some cases it is also important to map an entire semiconductor wafer to analyze impurities found in different areas of the sample. Computerized mapping can be done with a precision x-y stage, and here usually the optics are positioned at zero degrees to the sample.

Various experimental parameters derived from the spectra such as peak symmetry, FWHM of the peak, center position of the peak, and fine structure, can give information about the structure and composition of doped semiconductors. This type of analysis can also be used to evaluate growth methods using different input gas mixtures.

Figure 1 shows the optical and mechanical arrangement of the photoluminescence system for analysis and mapping of a semiconductor sample. The sample is placed on the MapMax x-y translational stage and fixed in position in the sample holder. The HORIBA Scientific Macro Illuminator allows light to be focused on the sample while the image is displayed on the monitor. An Ar+ laser is used as the excitation source. Light from the laser is focused into a fiber, which goes into the input channel of the HORIBA Scientific Macro Illuminator. The photoluminescence from the sample is collected, goes through the output channel of the HORIBA Scientific Macro Illuminator, into a fiber, and finally into the TRIAX550 monochromator for analysis.

The excitation light is chopped, so that a lock-in amplifier can be used with an InGaAs detector for data-acquisition. After the initial system alignment is fixed, the experiment itself is controlled by computer using SpectraMax for Windows® spectroscopy software.
Results

An example of a typical doped semiconductor photoluminescence spectrum obtained with a HORIBA Scientific system is shown in Fig. 2. Here, a liquid-nitrogen-cooled InGaAs detector was used, for optimal sensitivity and low noise. The entire experiment is controlled by SpectraMax for Windows® software, which provides easy data-acquisition and analysis.

Conclusions

A complete, nearly turn-key system for photoluminescence measurements from HORIBA Scientific is shown to produce spectra with high signal-to-noise ratio, for diagnostic testing of semiconductors and other materials. A HORIBA Scientific photoluminescence system provides a comprehensive solution for characterization of semiconductor materials.

Figure 2. Photoluminescence spectrum of doped GaAs.
Introduction
Photoluminescence is the optical emission obtained by photon excitation (usually a laser) and is commonly observed with III-V semiconductor materials. This type of analysis allows non-destructive characterization of semiconductors (material composition, qualitative investigations, etc.

Description
An Argon laser beam is focused on the sample which is located in the center of the sample compartment. If the energy of photons coming from the laser source is greater than the energy gap of the semiconductor, the sample emits photons. These are collected and analyzed with a dual flat field spectrograph. Two detectors are used, a CCD and an InGaAs array. This system allows investigations from 0.75 to 2.4 eV.

Measurement Procedure
We identify the position of one or several photoluminescence lines, and with the help of mathematical models, determine the composition of the material. Example: GaAlAs sample. The following expression gives the AlAs composition as a function of the photoluminescence line position (*):

\[ E(x) = 1.424 + 1.247 \times (\text{at } T = 300\text{K}) \]

with

\[ E(x): \text{line position (in eV units)} \times: \text{AlAs composition} \]

Measured line position: 1.643 eV
then \( x = 0.18 \) (18% of AlAs)

Applications
- Qualitative investigations for semiconductors.
- Fast determination of alloy composition in ternary compounds (GaAlAs, GaInAs, AlInAs...)

System Advantages
The two array detectors, CCD and InGaAs, offer high sensitivity and speed. They can both be mounted simultaneously on a Dual Flat Field Spectrograph allowing full characterization of the sample with ease.
MicOS characterizes III-V semiconductor microstructures GaN and related alloys are important materials used to build short-wavelength light sources (lasers and LEDs). Room- and low-temperature photoluminescence (PL) are used to characterize these materials as well as device performance. Parameters such as IQE of quantum wells (QW) can be measured for patterned structures using selective optical excitation of microstructures made from these materials. Selective excitation means fine control of laser-excitation beam size and positioning, and visualization of the sample under measurement.

In many such measurements, important electronic structure information can only be revealed at low temperatures. Therefore the PL measurement system also must be compatible with a cryostat. Fig. 1 shows a typical configuration of a HORIBA MicOS measurement system, and Fig. 2, the resultant PL spectra. Our MicOS (Fig. 3) also has the flexibility to accommodate different user-selectable excitation laser wavelengths for III-V material excitation, and includes vision so that the user can readily see excitation position and areas of interest on the sample (Fig. 3).

Fig. 1. Typical low-temperature, direct-coupled micro-PL setup. Direct coupling of the microscope’s front end increases throughput to the spectrometer (for low-light samples). System also has flexibility to measure sample via the side window of an upright cryostat or in a down-looking configuration.

Fig. 2. GaInN PL spectrum taken at 10 K after 405 nm laser excitation at different excitation power-levels.

Fig. 3 (Top) Down-looking version of HORIBA MicOS with mapping stage. (Bottom) Representative image of a patterned sample showing laser excitation on a region of interest.

References
2. Proprietary—semiconductor manufacturer.
**MicOS Specifications**

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<tr>
<th>Spectrometers</th>
<th>iHR320</th>
<th>iHR550</th>
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<td>800–1600 nm</td>
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<td>Microscope Objectives</td>
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<tr>
<td>Spot Size</td>
<td>100 µm</td>
<td>&lt;20 µm</td>
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**Sample Stage** | XYZ (manual or motorized) | |

<sup>1</sup> Depends on choice of objective, filters, and detectors.

<sup>2</sup> For 1200 gr/mm grating and open-electrode CCD

<sup>3</sup>BIV, BIVS, and BIDD formats available for specific quantum-efficiency requirements.

<sup>4</sup> Needs two detectors to cover entire range.

<sup>5</sup> Other options are available upon request.

*Specifications are subject to change without notice.*
High-Resolution Low-Temperature PL of Semiconductors

Introduction

Temperature-dependent photoluminescence (PL) spectroscopy is a powerful optical method for characterizing materials. PL can be used to identify defects and impurities in Si and III-V semiconductors, as well as determine semiconductor bandgaps. At room temperature, PL emission is usually broad—up to 100 nm in width. When samples are cooled, structural details may be resolved; a small spectral shift between two samples may represent a difference in structure. For cooling, two types of cryostat typically are used: a cryostat using liquid N2 or liquid He, or a closed-cycle cryostat in which cryogenic liquid is included as part of the cooling system. The cooled sample is excited by a laser, and the PL is coupled to a spectrometer via an optical interface. In this Technical Note, sample data are shown from a high-resolution PL system.
Experimental setup

A 1 m focal-length monochromator (1000M Series II, 600 gr/mm grating) scanned from 800–1650 nm. The detection system was a thermoelectrically cooled InGaAs detector with chopper and lock-in amplifier. Semiconductor samples were mounted in a closedcycle cryostat mounted above the optical table, and cooled to 4.5 K. A HORIBA Scientific Low Temperature Cryostat Interface optimized the optical coupling into the monochromator. The excitation source was a 10 mW HeNe laser ($\lambda = 632.8$ nm). Fig. 1 shows the experimental apparatus.
Results

Fig. 2 shows spectra of the same sample measured at room temperature and at 4.5 K. In PL spectroscopy, a material absorbs light, creating an electron-hole pair. An electron from the valence band jumps to the conduction band, leaving a hole in the valence band. The photon emitted upon recombination corresponds to the energy difference between the valence and conduction bands (bandgap), and is hence lower in energy than the excitation photon, so that the emission is red-shifted with respect to the excitation light.

At low temperatures, a PL peak is quite sharp. As the temperature increases, the peak broadens and shifts to lower energy. This red-shift, typical for shrinkage as a function of temperature. The decrease in peak intensity indicates that electrons escape via non-radiative processes.

Fig. 3 compares PL spectra from a sample of Nd:YAG laser-glass, using the 1000M Series II (1 m focal length) and the iHR320 (0.32 m focal length) spectrometers. Note the sharper peaks recorded with the 1000M system.
System components

In order to measure photoluminescence of semiconductors, the following are needed: a stable, powerful monochromatic light source, optics to focus light on the sample, a sample holder, collection optics, a spectrometer, and a detector for spectral analysis. The Low Temperature PL Optical Interface from HORIBA Scientific provides a stable collection-optics system, to collect the maximum amount of light from the sample inside either type of cryostat, and couple it efficiently into the spectrometer.

Benefits include:

- Reflective optics for maximum light collection
- Compatible with M-Series, iHR320/550, and FHR640/1000 spectrometers
- Mounts directly on the spectrometer entrance slit
- Compatible with most cryostats with 90 mm dia. bodies
- Input f/1.5, output f/7.5
- Filter-holder included [standard 1 inch (2.5 cm) filter]

HORIBA Scientific component | Part number
--- | ---
Low Temperature PL Optical Interface | ACC-CRYO-1000M
1000M Series II, 2 entrance and 1 exit slits | 1000M II
Optical Chopper | ACH-C
Lock-in Amplifier | SR810
Solid-state detector interface | 1427C
Thermoelectrically cooled InGaAs photodiode | DSS-IGA020T
SynerJY® spectroscopy software | CSW-SYNERJY
Closed-cycle He cryostat | Contact us
Cryostat-mounting hardware | Contact us
MicOS for mapping semiconductor wafers

III-V semiconductors are important to the fabrication of active photonic devices such as light sources and detectors. Successful fabrication of such devices relies on the high quality of the underlying materials and precise deposition of intended geometries on a wafer substrate. Defective materials and imperfections in geometries adversely affect yield, and usually increase cost and development times. The cost and delay penalties are further compounded when such defects in either material or device-geometry are not caught early enough in the cycle.

Photoluminescence (PL) spectroscopy is a robust, noncontact, non-destructive optical technique for determining material quality and geometrical accuracy for many III-V semiconductor-based components. Quality of the material is often obtained by measuring point PL on the bulk material, but geometrical accuracy of the device requires mapping PL over the entire device—or at least a region of interest on the device. Fig. 1 shows a typical PL emission near-IR spectrum of a III-V semiconductor measured at a point on a wafer, using the HORIBA MicOS PL wafer-mapper (Fig. 2).

Fig. 1. Typical room-temperature PL spectrum of a III-V semiconductor in the near-IR region of the spectrum following laser excitation at 532 nm. This measurement was carried out on the HORIBA MicOS system (Fig. 2, see right).

Fig. 2. Down-looking version of HORIBA MicOS with mapping stage.
The versatile HORIBA MicOS PL wafer-mapper micro-PL system includes a vision camera so the user always sees the region of the wafer under excitation, useful when the wafer has patterned structures. The MicOS head is directly coupled to a triple-grating spectrometer, ensuring the highest throughput and wide spectral coverage (200–1600 nm). The MicOS can also use different excitation-laser wavelengths, and includes an assortment of motorized xyz-stages for mapping wafers up to 300 mm. It can measure in a down-looking configuration for standard wafers or side-looking configuration for facet-emitting samples.

Included with the MicOS wafer-mapper is LabSpec software, which not only automates data-collection but offers an array of analytical tools for data-processing and interpretation. LabSpec offers a data-collection mode called SWIFT, in which the stage serves as the controller, and triggers detector-acquisition, bypassing the computer. This mode can collect data at high speed so that 2500 spectra can be collected over a two-inch wafer in under three minutes. LabSpec also offers an array of analytical and display tools, including peak identification and fitting, background subtraction, and multivariate analysis.
Fig. 3 displays various PL parameters for wafers (peak intensity, peak wavelength, and FWHM of the emission), all of which can be correlated to material properties of the wafer.

The PL measurements above are critical, and their implementation spans product design to fabrication and manufacturing quality-control, including failure-mode analysis for field failures. In quality-control, where time is critical, performing the mapping measurement quickly is important in order to create a statistically valid sample set, and thus increase the confidence level of the inferences drawn from the measurement.
MicOS characterizes Ge-based micro-LEDs

Monolithic integration of optical components on CMOS platforms is ongoing in the optical communications industry. CMOS offers a mature and robust platform, and therefore is logical for building optical-interconnect modules. These modules include light sources, modulators, multiplexers, and detectors on a single substrate. Silicon is the foundational material for CMOS technology, but as a material with an indirect bandgap, it poses a serious obstacle to building light sources via CMOS-based integrated photonics.

There is particular interest in integrated sources from 1300–1600 nm. Germanium has a bandgap exactly within this range. Like Si, Ge is a group IV element, so it is a likely candidate for integration on a CMOS device. Yet Ge is also an indirect-bandgap material but with an adjacent (higher-energy) well in the conduction band capable of direct transitions. Various mechanisms have been developed to engineer population of this direct-bandgap path in SiGe alloys leading to integrated light sources.1,2

One class of such SiGe light sources uses an electrical bias to populate the direct bandgap path. Electroluminescence (EL) is an appealing method for characterizing such devices. Figs. 1 and 2 show the EL spectra of one such device (Fig. 3, left) measured using the HORIBA MicOS microscope spectrometer (Fig. 3, right).3

References
2. S. Chen, et al., Optics Express, 17(12), 10019 (June 2009).
HORIBA Scientific’s MicOS (Fig. 1) microscope-spectrometer is a versatile instrument designed and optimized specifically for spectroscopy using a direct-coupled microscope. Such streamlining is required because this field traditionally has been served by a coupling between a standard imaging microscope and a spectrometer. This combination in many cases has proven to be suboptimal for spectroscopy. For one, the design considerations that often make a microscope very good for imaging (aberration-correction using glass optics) often prove to be a hindrance for spectroscopy, especially in the UV and IR regions of the spectrum. This hindrance is not only undesirable from a spectroscopic point of view, but also can be expensive. Furthermore, the coupling between the microscope and the spectrometer is often implemented using optical fibers, which is not optimal, for fibers can have significant losses in transmission and coupling for some regions of the spectrum. Not least is the fact that many traditional microscopes do not offer an easy method to couple an excitation laser while maintaining a port for vision, or the flexibility to accommodate different configurations for measuring samples.

The MicOS is a specially-built instrument that places an emphasis on spectroscopy in its design considerations. It offers a direct-coupled microscope with more than a ten-fold improvement in optical throughput compared to some fiber-coupled systems. It offers the flexibility to couple different laser excitation wavelengths without the need for factory- or field-servicing. The MicOS also provides the flexibility to measure the sample in different configurations: down-looking for flat samples such as wafers, and side-looking for such samples such as facet-emitting diodes or samples in upright cryostats.

In this application note, we provide examples of how the MicOS can advance your micro-luminescence research.
Examples

The first example shows how the MicOS may be used to study luminescence of III-V semiconductor materials. GaN and related alloys are important materials used to build short-wavelength light sources (lasers and LEDs). Room- and low-temperature photoluminescence (PL) are used to characterize these materials as well as device performance. These samples usually consist of micrometer-sized structures requiring selective laser-excitation in order to observe the PL emission. Selective excitation means fine control of laser-excitation beam size and positioning, as well as ability to see the sample under measurement. In addition, for many such measurements, important electronic structure information can only be revealed at low temperatures. Therefore a PL measurement system must also be compatible with a cryostat. Fig. 2 shows a typical configuration of a HORIBA MicOS measurement system, and Fig. 3, the resultant PL spectra. The MicOS also has the flexibility to accommodate different user-selectable excitation laser wavelengths for III-V material excitation, and includes vision so that the user can readily see excitation position and areas of interest on the sample.

References
2. Proprietary—semiconductor manufacturer.
For the second example, we use the MicOS to observe electroluminescence from a micro-LED (Fig. 4). The MicOS is particularly suited to this type of measurement because it allows measurement in a down-looking configuration with long working distance objectives, for samples such as VCSELs, in which the user may need to introduce probe pins under the objective, as well as a side-looking configuration for planar waveguide structures such as SiGe integrated IR waveguide sources. These latter structures are facet-emitting and suited to measuring in a side-looking configuration.3,4

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Finally, the MicOS also comes with an optional motorized stage for photoluminescence-mapping, in which the user is not only interested in the measured spectra but also in the spatial distribution of emitting centers on the sample. The map information is generated by a two-dimensional translation of the mapping stage so that PL data is collected at an array of points coincident with the tightly focused excitation beam (Fig. 5).

This spatial distribution information is relevant in diverse applications such as in isolating biological species (Fig. 6), as well as maps of wafer homogeneity in quality control in the semiconductor industry (Fig. 7).
MicOS for photoluminescence research

The system

The MicOS-based system (Fig. 1) described in this Technical Note uses a versatile platform for performing micro- as well as macro-photoluminescence (PL) measurements, and which can take transmittance and absorbance measurements, at an affordable price.

The core of the system is a HORIBA Scientific triplegrating spectrometer with two entrance ports and two exit ports. The MicOS head—the Micro-

PL accessory—is coupled to the front entrance port, while the transmission accessory (sample chamber and tunable light source) is coupled to the side entrance port. Direct coupling of these accessories ensures the highest throughput of light through the spectrometer to the detectors, which are attached the two exit ports. The exit ports can accommodate up to three different detectors (one port can accept two detectors) to cover a wide spectral range (200 nm–40 μm). HORIBA Scientific’s LabSpec software controls all components, and also collects and analyzes the data.

Fig. 1. MicOS multi-spectroscopy system.
Micro-PL measurements

Figs. 2a and b are photoluminescence spectra of InP and GaAs wafers collected using the Micro-PL accessory on the platform. The inserts in the graphs are the samples themselves with the bright circular laser spot at the center. The samples were excited at 532 nm. The InP data were collected using an InGaAs single channel detector on the side exit port, while the GaAs PL spectrum was collected using a CCD array detector on the front exit port. The spectral range spans 200–1600 nm.

Fig. 2. PL spectra of (a) InP and of (b) GaAs wafer.
Macro-PL measurements

Fig. 3 shows the configuration of the system for macro-PL measurements. An optional monochromator (not shown) may be added between the light source and sample chamber for tunability in the excitation wavelength. A 250 W tungsten-halogen lamp was used, but an optional 450 W Xe light source is available as well. For these measurements, a bandpass filter was used inside the sample chamber to select the excitation wavelength. The sample was a cuvette of aqueous coumarin. Fig. 4 shows the fluorescence spectrum following UV excitation of the sample.
Macro-transmission measurements

Fig. 5 shows the system configuration for macro-transmission measurements. A collimated white-light beam was directed through the sample in the sample chamber; lenses collected and imaged the transmitted light onto the input slit of the spectrometer. Fig. 6 is the transmission spectrum of a long-pass glass filter, normalized to a reference measurement without the sample.
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