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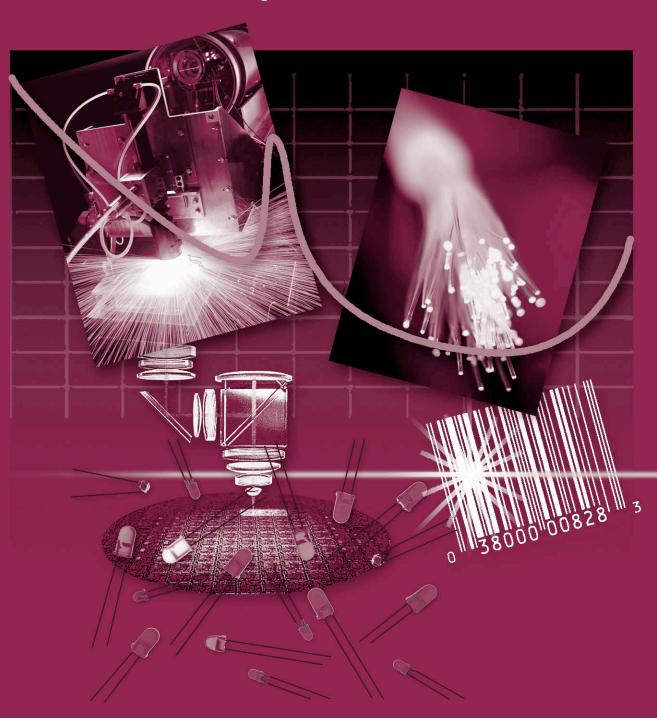
Technology Administration

U.S. Department of Commerce

NISTIR 6615 January 2002 **Electronics and Electrical Engineering Laboratory**

Optoelectronics Division

Programs, Activities, and Accomplishments



The Electronics and Electrical Engineering Laboratory

Through its technical laboratory research programs, the Electronics and Electrical Engineering Laboratory (EEEL) supports the U.S. electronics industry, its suppliers, and its customers by providing measurement technology needed to maintain and improve their competitive position. EEEL also provides support to the Federal government as needed to improve efficiency in technical operations, and cooperates with academia in the development and use of measurement methods and scientific data.

EEEL consists of six programmatic divisions and two matrixmanaged offices:

Electricity Division

Semiconductor Electronics Division

Radio-Frequency Technology Division

Electromagnetic Technology Division

Optoelectronics Division

Magnetic Technology Division

Office of Microelectronics Programs

Office of Law Enforcement Standards

This document describes the technical programs of the **Optoelectronics Division.** Similar documents describing the other Divisions and Offices are available. Contact NIST/EEEL, 100 Bureau Drive, MS 8100, Gaithersburg, MD 20899-8100, Telephone: (301) 975-2220,

On the Web: www.eeel.nist.gov

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Cover: The spectral attenuation curve of an optical fiber overlays photographs of an optical fiber cable, a laser welding system, a barcode, light emitting diodes (LEDs), and a depiction of a laser-based lithography system; all are examples of optoelectronic components and the diverse applications they enable.

Image of Lithographic Projection Optics courtesy of SVGL, Inc.

Electronics and Electrical Engineering Laboratory

Optoelectronics Division

Programs, Activities, and Accomplishments

NISTIR 6615

January 2002

U.S. DEPARTMENT OF COMMERCE

Donald L. Evans, Secretary

Technology Administration

Phillip J. Bond, Under Secretary of Commerce for Technology

National Institute of Standards and Technology

Arden L. Bement, Jr., Director



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Welcome

We appreciate your interest in the work of the NIST Optoelectronics Division. Our job is to provide the optoelectronics industry with the measurement technology, standards, and traceability it needs to flourish.

Part of our work is the development and evaluation of measurement methods that can support product specification. We work closely with American and international standards developing organizations in establishing standard measurement procedures. We also develop measurement methods that can be used in the manufacture of optoelectronic materials and devices—methods that can help lower costs and improve products.

For those who need reliable and accurate methods of calibrating instrumentation, we develop and provide Standard Reference Materials—stable and carefully characterized devices that can provide traceability to national standards. Currently, twelve SRMs are available. Most of them serve as standards for the properties of optical fiber or for other quantities, such as wavelength, related to optical communications.

For those who need traceability in laser radiometry, we provide calibrations of laser power and energy meters, including those intended to be used with optical fiber. These calibrations are traceable to national standards for laser power and energy developed and maintained by the Division. Our standards support calibrations at power levels from nanowatts to kilowatts and wavelengths from the deep ultraviolet through the mid-infrared. We also provide calibrations of the vector frequency response of optical detectors and for laser relative intensity noise.

Our measurement services are described more completely elsewhere in this document, and on our web site; http://www.boulder.nist.gov/div815.

The past year has been a difficult period for the optoelectronics industry, as general economic conditions and distortions in the market for optical communications have resulted in reduced earnings, lower share values, and reductions in employment. Nonetheless, optoelectronic technology has been the enabler of many of the most familiar and successful new products and services of the past couple of decades, and one can foresee a continuation of that trend for several more decades.

We in the Optoelectronics Division are pleased to be able to contribute to this exciting industry. Herein you will find brief descriptions of some of our work. We invite you to contact us to discuss these projects in more detail and to help us understand your needs for metrology. You will find our names and contact information on page vi.

Gordon W. Day, Division Chief



Optoelectronics Division
National Institute of Standards and Technology
325 Broadway
Boulder, CO 80305-3328
(303) 497-5342
Fax: (303) 497-7671
Optoelectronics@boulder.nist.gov

About the Optoelectronics Division

Mission

The mission of the Optoelectronics Division is to provide the optoelectronics industry and its suppliers and customers with comprehensive and technically advanced measurement capabilities, standards, and traceability to those standards.

History

The Division is located in Boulder, Colorado, as a part of the NIST Boulder Laboratories. It was established in 1994, succeeding an earlier NIST organizational unit, the Optical Electronic Metrology Group of the Electromagnetic Technology Division. The Division's roots extend to the first NIST (NBS) work on optoelectronics—research begun in the early 1960s to develop techniques for measuring the output power, or energy, of a laser. Since the late 1960s, NIST research on measurement and standards to support the development and application of lasers has been centered in the Boulder Laboratories. Research related to optical communications was added in the mid 1970s, and expanded substantially in the late 1980s; it now represents more than half of the Division's effort.

Organization

The Division is organized into three Groups. Two Groups—the Sources and Detectors Group and the Optical Fiber and Components Group—focus on the characterization of optoelectronic components. The third, the Optoelectronic Manufacturing Group, focuses on measurements that can lead to more efficient manufacturing of optoelectronic materials and components.

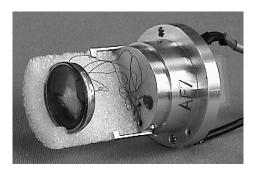
Research and Services

Most of the research performed in the Division is conducted either with NIST-appropriated funds or under contract to other U.S. Government agencies. Results are normally placed in the public domain through publication in the open literature. Some results become the subject of patents, and are available for license. The Division also conducts proprietary research in collaboration with industry and universities through Cooperative Research and Development Agreements (CRADAs).

The Division and its predecessor organizations have been providing calibration services for the characterization of lasers and detectors since 1967, and each year conducts over 200 calibrations for about 50 customers. It also provides the industry with standard reference materials, which are artifact standards that can be used to calibrate a customer's own instrumentation.

The Division maintains close contact with the optoelectronics industry through major industry associations, including the Optoelectronics Industry Development Association (OIDA), and the Lasers and Electro-optics Manufacturer's Association (LEOMA). Division staff members represent NIST to the major domestic and international standards organizations active in optoelectronics—the Telecommunications Industries Association (TIA), the International Electrotechnical Commission (IEC), the International Organization for Standardization (ISO), and the American National Standards Institute (ANSI)—and provide impartial technical expertise in their negotiations.

Primary Standards for Laser Radiometry



Absorbing element from the first primary standard for laser radiometry, a liquid cell calorimeter. Developed in the 1960s and used for pulsed-laser calibrations until the early 1970s.



The Laser-Optimized Cryogenic Radiometer, developed in the late 1990s, and used for the highest accuracy measurements with low-level cw lasers, 100 μ W to 1 mW at wavelengths from 0.4 μ m to 2 μ m.



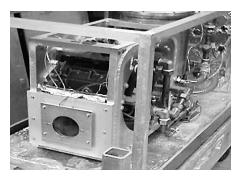
The C-Series Calorimeter, originally developed in the 1970s for use with cw lasers in the range from 50 μ W to 1 W and 0.4 μ m to 2 μ m wavelength. Improved versions in current use for calibrations.



The K-Series Calorimeter, developed in the 1970s, for high power cw lasers in the range from 1 W to 1000 W and wavelengths from 0.4 μm to 20 μm . In current use for calibrations.



Three versions of this standard are used for pulsed-laser calibrations. The Q-Series, developed in the 1980s is used for 1.06 μ m lasers. The QUV and QDUV calorimeters, developed in the 1990s are in current use for calibrations at 248 nm and 193 nm, respectively.



The BB Calorimeter, developed around 1980 for the U.S. Air Force Metrology Laboratory, and in current use for cw lasers with power levels up to 100 kW.

Optoelectronics Division

Division Office 815.00

Name	Extension*
DAY, Gordon W., Chief	5204
SMITH, Annie J., Secretary	5342
BAUER, Beth, Admin.Assist.	5123
McCOLSKEY, Kathy S.,	
Administrative Officer	3288

Sources and Detectors (815.01)

<u></u>	
HALE, Paul D. (Actg. GL) (PL)	5367
SKINNER, Dorothy L., Secretary	3842
CASE, William E. (GS)	3741
CHRISTENSEN, David H.	3354
CLEMENT, Tracy S. (PT)	3052
CROMER, Christopher L. (PL)	5620
DOWELL, Marla L. (PL)	7455
EL-HELBAWY, Mona A. (S)	4304
JONES, Richard D.	3439
KEENAN, Darryl A.	5583
LAABS, Holger J. R. (GS)	7736
LEHMAN, John H.	3654
LEONHARDT, Rodney W.	5162
LI, Xiaoyu	3621
LIVIGNI, David J.	5898
OBARSKI, Gregory E.	5747
PHELAN, Jr., Robert J. (GS)	3696
SIMPSON, Philip A. (GS)	3789
TOBIAS, Iris L.	5253
VAYSHENKER, Igor	3394
YANG, Shao	5409

Legend:

GL	=	Group Leader	
GS	=	Guest Scientist	
PD	=	Postdoctoral Appointment	
PREP PD	=	PREP Postdoctoral	
PL	=	Project Leader	
PT	=	Part Time	
S	=	Student	
*Telephone numbers are: (303) 497-XXXX,			
(the four	digit exte	nsion as indicated)	

Optical Fiber and Components (815.03)

GILBERT, Sarah L. (GL) (PL)	3120
SKINNER, Dorothy, Secretary	3842
CORWIN, Kristan L. (PD)	4217
CRAIG, Rex M.	3359
DENNIS, Tasshi	3507
DiCHIARA, Nicholas R. (S)	5293
DRAPELA, Timothy J.	5858
DYER, Shellee D.	7463
ESPEJO, Robert J. (S)	7630
ETZEL, Shelley M. (PT)	3287
FRANZEN, Douglas L. (GS)	3346
KOFLER, Jonathan D.	4276
NEWBURY, Nathan R.	4227
RABIN, Michael W. (PREP PD)	3021
SWANN, William C.	7381
WILLIAMS, Paul A. (PL)	3805

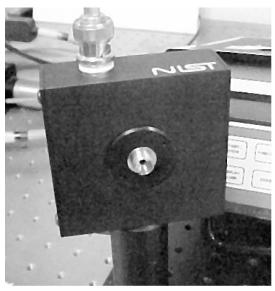
Optoelectronic Manufacturing (815.04)

(
HICKERNELL, Robert K. (GL)	3455
REPETTO, Francesca, Secretary	5187
BERRY, Joseph J. (PD)	5268
BERTNESS, Kristine A. (PL)	5069
CALLICOATT, Bert (PREP PD)	5952
CHEN, Ye (Mike) (GS)	5164
FU, Chih-Chiang (GS)	5572
HARVEY, Todd	3340
KIM, Chulsoo (PD)	3942
LEHMAN, Susan Y. (PREP PD)	7554
MIRIN, Richard P. (PL)	7955
ROSHKO, Alexana	5420
SANFORD, Norman A. (PL)	5239
SCHLAGER, John B.	3542
SILVERMAN, Kevin L.	7948
SONNENBERG-KLEIN, Benjamin	
(PREP PD)	7460
SU, Mark (PREP PD)	7368

CW Laser Radiometry

Project Goals

Develop measurement methods and standards for characterizing laser sources and detectors used with continuous-wave (cw) laser radiation. Develop and maintain measurement services for laser power and energy, optical-fiber power, and related parameters (spectral responsivity, linearity, etc.).



The Optoelectronics Division's new Optical Fiber Power Meter transfer standard. This optical detector has been optimized to provide high-accuracy measurements of optical fiber power traceable to the division's Laser Optimized Cryogenic Radiometer (LOCR).

Customer Needs

Accurate characterization of optoelectronic sources and detectors is important in the development and use of industrial technologies such as lightwave telecommunications, lasermedical instrumentation, materials processing, photolithography, data storage, and laser safety equipment. This Project focuses on selected critical parameters intrinsic to sources and detectors, especially the calibration of optical-fiber power meters and laser power or energy meters at commonly used wavelengths and powers or energies. In addition, special test measurements are available for linearity, spectral responsivity, and spatial and angular uniformity of laser power meters and detectors. Project members participate in national and international standards committees developing standards for laser safety, laser radiation

measurements (such as beam profile and pointing stability), and optical-power-related measurements. They extend and improve source and detector characterizations, including development of low-noise, spectrally flat, highly uniform pyroelectric detectors; high-accuracy transfer standards for optical-fiber and laser power measurements; and advanced laser systems for laser power and energy measurements.

Technical Strategy

Meeting the needs of the laser and optoelectronics industries and anticipating emerging technologies requires investigation and development of improved measurement methods and instrumentation for high-accuracy laser metrology over a wide range of powers, energies, and wavelengths.

NIST has historically used electrically calibrated laser calorimeters to provide traceability to the SI units for laser power and energy. We recently developed a new measurement capability based on a Laser Optimized Cryogenic Radiometer (LOCR), which provides improvement in accuracy by an order of magnitude for laser power measurements. To meet the increasing demands for higher accuracy over a larger range of optical power and wavelength, it is necessary to improve the accuracy of calibration services through the development of better transfer standards, traceable to LOCR.

DELIVERABLE: By 2003, reduce by at least a factor of 2 the uncertainties for all calibrations of laser energy and power supplied to customers. This will be accomplished through the development of improved laser power transfer standards with greater range in optical power and wavelength.

The explosion in the rate of development of technology in the telecommunications industry has led to demands for higher performance and higher accuracy for optical-fiber power meter characterization and calibration. In addition, the Department of Defense (DOD) has begun to accelerate the use of opticalfiber in many of its new weapons systems. By developing improved standards for optical fiber power and by developing instrumentation to accurately quantify the uncertainties of the new standards, we can meet these demanding requirements.

DELIVERABLE: By 2002, reduce by another factor of 2 the uncertainties for optical-fiber power meter

Technical Contact:

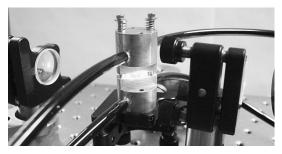
Christopher L. Cromer

Staff-Years:

4.5 professionals
1.0 technicians

Funding Sources: NIST (42 %) Other Government Agencies (53 %) Calibration fees (5 %)

calibrations supplied to customers. This will be accomplished through the development of higher-accuracy transfer standards and instrumentation to characterize these devices and accurately assess uncertainty budgets.



The heart of a new tunable Ti:Sapphire laser that will expand the capabilities of the Optoelectronics Division's calibration services in laser power and optical fiber power. Shown is the laser crystal being pumped by a green laser beam. The new laser system will provide many more wavelengths and higher power capabilities.

Advances in laser technology are continuously producing lasers with new wavelengths and power levels. We are involved in an ongoing effort to expand wavelength and power-range capabilities through implementation of new tunable solid-state laser technology to keep up with customer needs for calibration services at NIST. These new laser systems will be capable of providing a new wavelength requested by a customer with a minimum of development time and cost by having a flexible suite of laser systems available in the laboratory.

DELIVERABLE: By 2003, develop a suite of tunable solid-state lasers to cover the entire spectral range from the deep UV to the thermal IR. This laser system will continue to evolve as new technology becomes available to provide new calibration services as needs arise.

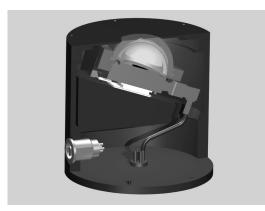
The laboratory systems and facilities that support the laser and optical-fiber power measurement services are aging and in need of updating. In addition, the measurement equipment and software in many cases is outdated and becoming more difficult to maintain. This upgrade to the laser and optical-fiber power measurement services will also involve collocating these measurements systems to take advantage of the new solid-state laser systems also being developed.

DELIVERABLE: By 2002, update the laboratory facilities and services to support the laser-power and optical-fiber power calibration services. This will involve refurbishing the rooms, cooling systems,

electrical, and mechanical services for the laser-power, optical-fiber power, and LOCR measurement systems, and collocating these laboratories near the new solidstate laser system being developed.

Accomplishments

- Improved accuracy of fiber-optic power meter calibrations by a factor of two. Completed a DOD funded project to improve fiber-optic power-meter calibrations. Using a high-accuracy laser-optimized cryogenic radiometer (LOCR), succeeded in reducing the uncertainty of calibration services from 1 % to 0.5 %. Improved accuracy for the NIST calibrations will enable higher accuracy for end users, and will result in tighter tolerances in optical-fiber communication systems. DOD is investing heavily in fiber-optic systems in many applications such as electromagnetic pulse (EMP) resistance, weight reduction, and highspeed communications.
- Developed a new high-accuracy transfer standard for optical-fiber power-meter calibrations. This new transfer standard was evaluated using a new measurement system that characterizes the angular responsivity of the detector responsivity by simulating the diverging output of an optical fiber.
- Completed documentation for optical-fiber power-meter calibration and linearity characterization services, and spectral-response measurement services.
- Developed an improved radiometer for optical measurements using a domainengineered pyroelectric detector element, which has reduced susceptibility to environmental acoustic noise and vibration, resulting in a lower noise floor for optical measurements in most applications. (Collaboration with the NIST Optical Technology Division). Improved the general performance of pyroelectric detectors in optical measurements at NIST and other optical metrology labs.
- Built an automated system for linearity measurements of 1 kW detectors using a custom-built reflective chopper wheel as a high-power laser-beam attenuator.
- Upgraded and refurbished the Air Force's Megajoule laser calorimeters. The calorimeters were designed and built by NIST more than 25 years ago to support the DOD's high-energy laser programs.



A three-dimensional representation of a new pyroelectric-based radiometer head for infrared optical measurements as part of a collaboration with the NIST Optical Technology Division. The critical detector element and gold-black coating are being developed in the Optoelectronics Division.

Calibrations

The Optoelectronics Division provides calibration services for laser and optical-fiber power at many wavelengths and power levels from the ultraviolet, the far-IR spectral regions, and from picowatt to kilowatt power levels. See Appendix D.

Equipment Supplied to Other Agencies and National Laboratories

- Special high-sensitivity pyroelectric detectors with high spatial uniformity supplied to the NPL (National Physical Laboratory of the United Kingdom) and the NMH (Hungarian National Laboratory).
- Domain-engineered pyroelectric detectors supplied to Optical Technology Division in the Physics Laboratory and researchers at Columbia University.
- Various transfer standards for laser and optical-fiber power delivered to DOD sponsors.

Standards Committee Participation

American National Standards Institute/Z136: John H. Lehman is a member of this committee, which deals with Safe Use of Lasers.

Telecommunications Industry Association/FO06/SC.01/WG.10: Igor Vayshenker is a

member of this working group, which deals with Metrology and Calibration.

Recent Publications

- I. Vayshenker, S. Yang, X. Li, T. R. Scott, and C. L. Cromer, "Optical Fiber Power Meter Nonlinearity Calibrations at NIST," NIST SP 250-56, 29 pp (Aug 2000).
- I. Vayshenker, X. Li, D. J. Livigni, T. R. Scott, and C. L. Cromer, "NIST Measurement Services: Optical Fiber Power Meter Calibrations at NIST," NIST SP 250-54, 36 pp (Jun 2000).
- I. Vayshenker, S. Yang, X. Li, T. R. Scott, and C. L. Cromer, "NIST Measurement Services: Optical Fiber Power Meter Nonlinearity Calibrations at NIST," NIST SP 250-56, 29 pp (Aug 2000).
- J. H. Lehman, G. Eppeldauer, J. A. Aust, and M. Racz, "Domain-Engineered Pyroelectric Radiometer," Appl. Opt. **38**(34), 7047-7055 (Dec 1999).
- J. H. Lehman, "Calibration Service for Spectral Responsivity of Laser and Optical-Fiber Power Meters at Wavelengths Between 0.4 μ m and 1.8 μ m," NIST SP 250-53, 39 pp (Dec 1999).
- J. H. Lehman and X. Li, "A Transfer Standard for Optical Fiber Power Metrology," Opt. and Phot. News, Eng. and Lab. Notes, **10**(5), 44f-h (May 1999).
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- S. V. Tikhomirov, A. I. Glazov, M. L. Kozatchenco, V. E. Kravtsov, A. B. Svetlichny, I. Vayshenker, T. R. Scott, D. L. Franzen, "Comparison of Reference Standards for Measurements of Optical-Fibre Power," Metrologia 37: 347-349, (2000)
- I. Vayshenker, H. Haars, J. H. Lehman, D. J. Livigni, "Comparison of Optical-Power Meters Between the NIST and the PTB," Metrologia **37**: 349-350, (2000)
- X. Li, T. R. Scott, C. L. Cromer, D. Keenan, F. Brandt, K. Möstl, "Power Measurement Standards for High-Power Lasers: Comparison Between the NIST and the PTB," Metrologia **37**: 445-447, (2000)
- J. H. Lehman, C.L. Cromer, "Optical Tunnel-Trap Detector for Radiometric Measurements," Metrologia **37**: 477-480, (2000)
- J. H. Lehman, A.M. Radojevic, R.M. Osgood Jr., M. Levy, C.N. Pannell, "Fabrication and Evaluation of a Freestanding Pyroelectric Detector Made from Single-Crystal LiNbO₃," Opt. Lett. **25**(22): 1657-1659, (Nov 2000)
- J. H. Lehman, A. M. Radojevic, R. M. Osgood Jr., "Domain-Engineered Thin-Film LiNbO₃ Pyroelectric-Bicell Optical Detector." IEEE Phot. Tech. Lett. **13**(8): 851-853; (Aug 2001)
- J. M. Houston, D. J. Livigni, "Comparison of Two Cryogenic Radiometers at NIST," NIST JRES 106(4): 641-647, (Jul-Aug 2001)

Pulsed-Laser Radiometry

Technical Contact:

Marla L. Dowell

Staff Years:

4.5 professionals1.0 guest researchers

Funding Sources:

NIST (61 %) Other Government Agencies (35 %) Calibration fees (4 %)

Project Goals

Develop measurement methods and standards for characterizing pulsed-laser sources and detectors. There is ongoing development work in the following areas: standards development, calibration services, and advising customers on in-house measurements.

Customer Needs

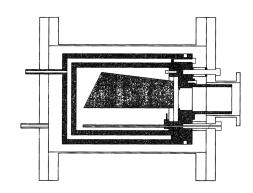
Accurate measurement methods and standards for characterizing pulsed-laser sources and detectors are critical in a number of industrial applications. Project members work closely with industry to develop standards, new technology and appropriate measurement techniques for pulsed-laser measurements. These efforts include development work in standards development, calibration services, and advising customers on in-house measurements. The bulk of our work is concentrated on ultraviolet (UV) laser metrology using excimer lasers.

Excimer lasers are used in a wide range of industrial applications. In addition to optical lithography for semiconductor manufacturing, excimer lasers are used in corneal sculpting procedures for vision correction, for example in photorefractive keratectomy (PRK) and Laser In-situ Keratomileusis (LASIK), as well as in micromachining of small structures such as ink jet printer nozzles. However, the bulk of our efforts are concentrated on UV laser metrology in support of semiconductor photolithography.

Increasing information technology requirements have yielded a strong demand for faster logic circuits and higher-density memory chips. This demand has led to the introduction of deepultraviolet (DUV) laser-based lithographic tools for semiconductor manufacturing. These tools, which employ KrF (248 nm) and ArF (193 nm) excimer lasers, have led to an increased demand for accurate laser measurements at the DUV laser wavelengths. As a result, NIST, with SEMATECH support, has developed primary standard calorimeters for measurements of both 193 nm and 248 nm excimer laser power and energy.

Technical Strategy

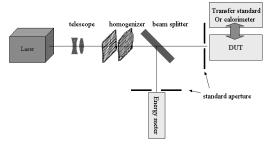
Beginning with the first edition of the National Technology Roadmap for Semiconductors in 1992, the semiconductor industry has made an organized, concerted effort to reduce the feature sizes of integrated circuits. As a result, there has been a continual shift towards shorter exposure wavelengths in the optical lithography process. Because of their inherent characteristics, DUV lasers, specifically KrF (248nm) and ArF (193nm), and more recently F₂ (157nm) excimer lasers, are the preferred sources for high-resolution lithography at this time.



Excimer laser calorimeter for 157 nm measurements.

DELIVERABLE: By 2002, develop a 157 nm excimer laser primary standard and calibration service to provide support for the next generation of optical lithography.

In addition to existing DUV laser measurement services, there is increasing demand for laser dose (energy density) measurements, where the detector samples a fraction of the total laser beam. Accurate laser dose measurements are important because small-area detectors are widely used to monitor laser-pulse energy density at the wafer plane of a lithographic tool. Accurate measurements of laser dose are especially crucial to the development of new mask resist materials, since lower dose requirements lead to greater wafer throughput and also extend the lifetime of an exposure tool's optical components as well.



Dose meter calibration system. The energy meter acts as a monitor to record pulse-to-pulse laser energy fluctuations.

Excimer lasers are seeing increased use in the micromachining of small structures, such as inkjet nozzles and optical waveguides. Laserbeam characterization measurements are critical in some of these processes since excimer laser beams are astigmatic.

DELIVERABLE: By 2002, develop capabilities for characterization and homogenization of excimer laser beam at 248 and 157 nm.

Recently, there has been increasing demand for more accurate laser target ranging and designation systems. These systems provide positional information, such as distance and incident angle, for use in locating targets. There is a need to expand existing pulsed Nd:YAG laser power and energy calibration services to include high pulse energies and to reduce the overall uncertainty associated with these measurements.

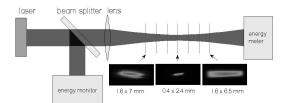
DELIVERABLE: By 2002, develop capabilities for measurements of high pulse energy at 1.06 $\mu m.$ Design, build, and characterize an improved calibration service for measurements of pulsed Nd:YAG laser power and energy.

Accomplishments

■ Established a capability to accurately perform absolute calibrations of responsivity of laser-dose meters at the laser wavelength of 193 nanometers. Additional excimer-laser wavelengths will be added to this service in the near future. The dose measurements are performed using beamsplitter-based a calibration system in which a spatially uniform beam from an argon-fluoride excimer laser is generated using a special beam homogenizer. The beam propagation properties, including homogeneity, uniformity or characterized with a state-of-the-art beam profile measurement system based on a pyroelectric camera array. This uniform beam

then irradiates a NIST-calibrated aperture placed immediately in front of the test detector.

■ Determined the damage thresholds and lifetimes of several materials using 157 nm and 193 nm excimer lasers and a beam profile technique similar to ISO 11254-2. We made these measurements to select an appropriate absorbing material for use in our primary standard laser calorimeter for measurements on 157 nm excimer lasers. The materials we tested were nickel-plated sapphire, chemically-vapor-deposited silicon (CVD SiC), nickel-plated copper, and polished copper. The applied pulse energy densities (or dose) ranged from 80 mJ/cm² to 840 mJ/cm². We determined the applied dose from a series of laser-beam profile measurements. carbide had the highest damage threshold: 730 mJ/cm² per pulse. For this reason, and for its high thermal and electrical conductivities, we have chosen silicon carbide as the absorber material for the 157 nm calorimeter.



Damage threshold measurements. Beam profiles were recorded and characterized at 22 planes along the propagation axis with a CCD camera and quantum converter. Three energy density distributions at inner- and outer-most positions, together with their dimensions, are shown. The dimensions are based on the calculation of the second moments according to ISO 11146.

Calibrations

The Pulsed-Laser Radiometry Project provides pulsed-laser calibration services for laser power and energy using excimer and pulsed-Nd:YAG lasers. See Appendix D for a complete list of available wavelengths and laser power/energy ranges.

Recent Publications

"Thermal Response and Inequivalence of Pulsed Ultraviolet Laser Calorimeters," D. H. Chen, M. L. Dowell, C. L. Cromer, Z. M. Zhang, Journal of Thermophysics and Heat Transfer, in press.

"Damage Testing of Partial Reflectors for 157 nm Laser Calorimeters," H. Laabs, R. Jones, C. Cromer, M. Dowell, V. Liberman, Annual Symposium on Optical Materials for High Powered Lasers, in press. "Pulsed-laser Metrology at NIST," M. Dowell., Optics and Photonics News (February 2001) 28.

"The Power of Light: Choosing the Right Detector is Key to Accurate Beam Power Measurements," M. Dowell, OE Magazine (January 2001) 56.

"New Developments in Deep Ultraviolet Laser Metrology for Photolithography,". M. L. Dowell, C. Cromer, R. Jones, D., Keenan, and T. Scott, Characterization and Metrology for ULSI Technology: 2000, D. Seiler, A. Diebold, T. Shaffner, R. McDonald, W. Bullis, P. Smith, and E. Secula, Eds. (AIP, New York, 2001), pp. 391-394.

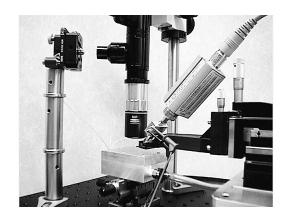
"New Developments in Deep Ultraviolet Laser Metrology for Photolithography": M. Dowell, C. Cromer, D. Jones, D. Keenan, and T. Scott, Society of Manufacturing Engineers Technical Note Paper MS00-236 (2000).

High-Speed Measurements

Project Goals

Provide advanced metrology, standards and measurement services relating to temporal properties of optical sources and detectors used in association with optoelectronic systems.

Customer Needs



Apparatus for measuring the frequency response magnitude of an on-wafer photoreceiver using a heterodyne method. New techniques make de-embedding or removal of the effects of the wafer probe possible.

High-bandwidth measurements are needed to support systems that take advantage of the potential bandwidth of optical fiber. Systems presently being installed operate at 5 to 10 gigabits per second using pure time-division multiplexing (TDM). Research is being done on the next generation of TDM systems at 40 to 80 gigabits per second in laboratories around the world. Methods are needed to accurately characterize the scalar and vector frequency response of high-speed sources, detectors, and instrumentation to three to five times the system modulation rate. Burst-mode operation in asynchronous transfer mode networks requires additional characterization at very frequencies. Increasingly tight tolerances in both digital and analog systems require frequency-response measurements with low uncertainty.

Measurements of source and detector noise are required to predict low bit-error rates in computer interconnects, and high carrier-to-noise ratios in analog systems, and to support measurements of noise figure optical amplifiers.

The intensive use of laser target designators and range finders by the armed forces requires traceable calibration standards for low-level pulse peak power and energy at 1064 nm and 1550 nm.

Technical Strategy

NIST has developed highly accurate heterodyne techniques at 850 nm, 1319 nm, and 1550 nm for measuring frequency response of detectors. A Calibration Service has been established for frequency-response transfer standards operating at 1319 nm and consisting of a photodiode combined with a microwave power sensor. This system is capable of measuring this type of standard from 300 kHz to 110 GHz or more. We have similar capabilities at 850 nm and 1550 nm that will be documented as part of our Calibration Services in FY02, along with a service for calibrating the frequency-response magnitude of bare photodiodes to at least 50 GHz. Calibration of bare photodiodes is more complicated because it requires calibrated microwave power and scattering-parameter measurements.

Optoelectronic phase response, when combined with the magnitude response, is called the vector response and is required for design of high-speed optoelectronic systems. The vector frequency response of a photoreceiver is the Fourier transform of its impulse response. At present there are no accepted standard methods for measuring optoelectronic vector frequency response.

Researchers in the High-Speed Measurements Project have demonstrated time-domain techniques for measuring optoelectronic vector response with verifiable accuracy up to 30 GHz using electro-optic sampling, and we intend to extend this capability to 110 GHz in FY2002. By developing these measurements our project is pioneering a new paradigm for time-domain measurements with frequency-domain calibrations.

DELIVERABLE: We intend to have a Calibration Service for optoelectronic phase that uses these new calibration strategies available in FY2003.

Optical communications analyzers or reference receivers used for measuring digital eyepatterns on optical signals have many similarities to electrical oscilloscopes, but also

Technical Contact:

Paul D. Hale

Staff Years:

4.0 professionals0.5 technician1.0 contractor1.0 student

Funding Sources:

NIST (73 %) Other Government Agencies (25 %) Calibration Fees (2 %) have advantages of their own. In particular, they can be calibrated over a very high bandwidth because they do not require bandlimited microwave calibrations. Use of this calibration on typical measurements, however, possesses some unique problems. We are currently applying our expertise in receiver measurements to these problems in collaboration with the Statistical Engineering Division.

NIST is developing optical-noise measurement systems for calibrating laser relative-intensity noise (RIN) and optical-amplifier noise-figure measurements. We have documented a calibration artifact for RIN and are currently investigating ways of applying this artifact to noise-figure measurements.

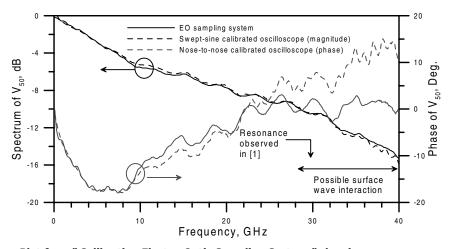
NIST has developed methods for calibrating absolute laser pulse energy and peak power at 1060 nm, available as a Calibration Service in FY2002, and which are traceable to national cw laser power and energy standards maintained by the Sources and Detectors Group.

DELIVERABLE: By 2003 we expect to have available a Calibration Service for low-level peak pulse power and pulse energy at 1550 nm.

Accomplishments

■ Demonstrated a measurement of the vector frequency response of a photoreceiver with rigorous traceablility to fundamental physical principles. The measurement uses electro-optic sampling to measure a voltage in a coplanar waveguide. The voltage measurement is deembedded and calibrated by means of standard frequency-domain microwave procedures. The response of the photoreceiver was also measured with an oscilloscope that had been calibrated using the nose-to-nose method, giving the first independent experimental check of the nose-to-nose method.

- Documented rigorous procedures for deembedding measurements of the response of modulated optical sources and optical receivers from their microwave test fixtures. These procedures can be used with frequency-domain measurements, such as the heterodyne technique or a lightwave component analyzer, or with time-domain techniques, such as optical impulse-response measurements with an oscilloscope or electro-optic sampling. The theory also describes the relationship between the measurements and equivalent circuit models for sources and receivers.
- Completed a detailed uncertainty analysis of a RIN standard. The standard consists of an erbium-doped fiber amplifier, narrow-band optical filter, and polarizer, and provides an amount of noise that can be calculated from first principles. This work provides the foundation of a Measurement Assurance Program for RIN and is also being applied to the calibration of optical-amplifier noise figure-measurements.



Plot from "Calibrating Electro-Optic Sampling Systems" showing preliminary electro-optic sampling measurement of photoreceiver vector frequency response in comparison to state-of-the-art oscilloscope based measurement.

Standards Committee Participation

Telecommunication Industry Association/FO02/SC.01/WG.01: Gregory Obarski is a member of this working group, which deals with Optical Amplifiers.

Telecommunication Industry Association/FO06/SC.01/WG.10: Gregory Obarski and Paul Hale are members of this working group, which deals with Metrology and Calibration.

International Electrotechnical Commission/TC86/WG4/: Paul Hale is a member of this committee, which deals with Fiber Optic Test Equipment Calibration.

International Electrotechnical Commission/ SC86C/WG3: Greg Obarski is a member of this committee, which deals with Optical Amplifiers.

Measurement Services

The Optoelectronics Division provides measurement services relating the temporal properties of optical sources and detectors used with optoelectronic systems including: optical receiver frequency response (scalar or vector), impulse response, low-level pulse power and energy, and relative intensity noise. For more information see Appendix D or contact Paul Hale at (303) 497-5367.

Recent Publications

"Measuring the Frequency Response of Gigabit Chip Photodiodes"; P. D. Hale; T. S. Clement; D. F. Williams; E. Balta; N. D. Taneja, IEEE J. Lightwave Technol. **19**(9): 1333-1339; Sept 2001.

"Transfer Standard for the Spectral Density of Relative Intensity Noise of Optical Fiber Sources Near 1550 nm"; G. E. Obarski; J. D. Splett, J. Opt. Soc. Am. B **18**(6): 750-761, Jun 2001.

"Calibrating Electro-Optic Sampling Systems"; D. F. Williams; P. D. Hale; T. S. Clement; J. M. Morgan, Proc., Intl. Microwave Symposium, May 20-25, 2001, Phoenix, AZ, 1527-1530, May 2001.

"Frequency Response Metrology for High-Speed Optical Receivers"; P. D. Hale; T. S. Clement; D. F. Williams, Tech. Dig., Optical Fiber Communication Conf. (OFC'01), Mar 17-22, 2001, Anaheim, CA, WQ1-1-3, Mar 2001.

"Alignment of Noisy Signals"; Coakley, K.J.; Hale, P.D. IEEE Trans. Instrum. Meas. **50**(1): 141-149; Feb 2001.

Interferometry and Polarimetry

Technical Contact:

Paul A. Williams

Staff-Years (FY 2001):

5 professional 0.4 technician 1.5 student

Funding Sources:

NIST Base (68 %) NIST Non-Base (12 %) Other (20 %)

Project Goals

Use interferometric and polarimetric techniques to provide calibrating measurements, standards, and expertise in support of the optoelectronics industry (primarily telecommunications).



Measurement Assurance Program artifact exhibits stable polarization-dependent loss, certified as a function of wavelength.

Customer Needs

telecommunication industry requires characterization of its optical fiber-based communication systems and components with improved temporal and spectral resolution. Well-characterized measurements and artifact standards are needed to ensure accuracy of measurements. This applies to areas such as polarizationand wavelength-dependent propagation delay, polarization-dependent loss polarization-dependent and spectral transmission.

Much of the metrology in telecommunications relates to well known properties. However, difficulties arise due to the high level at which these quantities must be resolved to enable current and future systems with high data-rate and high spectral efficiency. Increasing the overall data rate tends to reduce a system's tolerance to such parameters as chromatic dispersion, relative group delay, polarizationmode dispersion, polarization-dependent loss, spectral polarization-dependent transmission. We are developing new measurement techniques for these parameters and are working to increase the spectral and temporal resolution of existing methods.

Chromatic dispersion in optical fiber arises from the variation of the light's propagation velocity as a function of wavelength (it is the derivative of the relative group delay (RGD) versus This variation in propagation wavelength). velocity results in broadening of the optical pulses used in communication systems. Broadened pulses interfere with each other and difficult to distinguish, causing communication errors. System chromatic dispersion must be dealt with by managing its spectral profile (through dispersion-tailored fibers, choice of zero-dispersion wavelength, and dispersion-compensating fibers). This requires accurate measurement of dependence chromatic wavelength of dispersion. Similarly, wavelength-dependent relative group delay in components degrades system performance. Often, RGD must be measured in components that have subnanometer optical bandpass regions (requiring <100 pm spectral resolution). At the same time, high data rates require that RGD be measured with sub-picosecond temporal resolution. These requirements are in opposition fundamentally, spectral and temporal resolution are inversely related. A challenge is to optimize the mutual resolution of these quantities. This same dilemma is experienced in polarizationmode dispersion (PMD) metrology where propagation velocity depends on polarization state of the light. Communication systems require PMD measurement with a few tens of femtoseconds of resolution in a bandwidth of a few tens of picometers. In addition, PMD is a statistical quantity, making reference artifact stability difficult.

Non-temporal polarization effects such as polarization-dependent loss (PDL) polarization-dependent wavelength shift (PDW) of optical filters cause intensity variations as a function of polarization and wavelength. These effects generally weaken the transmission signal in a way that can vary with time and system conditions—again causing communication errors. PDW of a channel filter can also cause crosstalk between wavelength channels. PDL and PDW present unique metrology needs; PDL can arise in almost every component in an optical system, making assessment of PDL measurement accuracy difficult. PDW of filter

spectra is tedious to measure and rapid measurement techniques are needed.

Technical Strategy

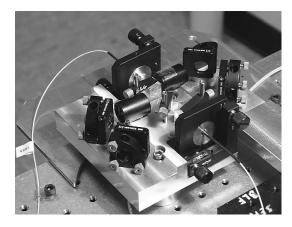
In general, we approach these needs through the development of high-resolution measurement techniques and rigorous uncertainty analysis. Transfer of this metrology comes through artifact distribution, measurement comparison, and special tests.

Chromatic Dispersion: With our RF phase shift system's recently enhanced stability in chromatic dispersion and zero-dispersion wavelength measurements, we are able to perform certifying measurements of chromatic dispersion and zero-dispersion wavelength of customer artifacts.

DELIVERABLE: By 2002, extend chromatic dispersion measurement capabilities to L-band wavelengths.

Relative Group Delay (RGD): Our RGD measurement work is directed toward improving temporal resolution, reducing the necessary measurement bandwidth and establishing fundamental standards with theoretically predictable RGD profiles.

DELIVERABLE: Uncertainty characterization of RF phase shift and low-coherence interferometry RGD systems by 2002.



Polarizing Mach-Zender interferometer for fundamental generation of large differential group delays for PMD validation during customer certification.

Polarization-Mode Dispersion (PMD): Our efforts are toward improved understanding of uncertainties in PMD measurement, improving temporal resolution of measurements, decreasing bandwidth needed for and developing measurement, new measurement techniques.

DELIVERABLE: By 2002, release non-mode-coupled PMD artifact as Standard Reference Material (SRM) 2538

Polarization-Dependent Loss (PDL): We are developing new PDL measurement techniques for accurate wavelength-dependent characterization of PDL and measurements with reduced detrimental effects due to system birefringence.

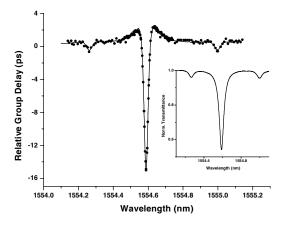
DELIVERABLE: Certified PDL artifact available for wavelength-dependent calibration through the Measurement Assurance Program (MAP) by 2002.

Component Spectral Transmission/Reflection: Accurate characterization of the bandpass spectrum of narrow optical filters is being developed in both the frequency domain (tunable-laser based system) and in the Fourier domain.

DELIVERABLE: Implement second generation Fourier-transform spectroscopy system for stable measurement of fiber-Bragg-grating center wavelength by 2002.

Accomplishments

- Expanded and improved NIST's chromatic dispersion metrology capabilities. Through the addition of active wavelength referencing, the existing RF phase shift system has been certified for chromatic dispersion measurement with an uncertainty of better than 0.2 %. Stability of the system was improved to yield < 40 pm uncertainty on zero-dispersion wavelength.
- Constructed a 200 MHz RF phase-shift system for measuring relative group delay (RGD) in optical components. The system's operating frequency allows ~ 3 pm wavelength resolution with ~ 1 ps temporal resolution.
- Measured the RGD across an absorption line of a hydrogen cyanide gas cell using the 200 MHz RF phase-shift system. The RGD of the absorption line was theoretically predicted from measured spectra and agreed with measurements to within the uncertainty. This device was also measured using a low-coherence Fourier-transform spectroscopy measurement system.



Theoretical and experimental relative group delay (RGD) for hydrogen cyanide absorption line - predictable from transmission spectra (inset).

- Demonstrated polarization-mode dispersion measurement using an improved modulation phase shift (MPS) technique at 2.5 GHz. The technique, based on a comb-generator and high phase-resolution lock-in amplifier, shows preliminary temporal resolution of a few tens of femtoseconds in a bandwidth of 40 pm.
- Completed testing and certification of a measurement system for Polarization Dependent Loss (PDL) as a function of wavelength.
- Revised our calculations for the NIST standard quarter-wave retarder for use in the 1550 nm band. This quarter waveplate offers an optical retardance, stable against temperature, entrance angle and wavelength, and is now characterized for the 1200 to 1600 nm wavelength region.

Standard Reference Materials (SRM) See appendix D

SRM 2524, Optical Fiber Chromatic Dispersion Standard, no longer available – Contact Interferometry and Polarimetry Project for alternative options.

SRM 2525, Linear Retardance Standard, no longer available as an SRM – Contact Interferometry and Polarimetry Project for artifact availability.

SRM 2518, Polarization-Mode Dispersion (Mode-Coupled); sold out at time of printing.

SRM 2538, Polarization-Mode Dispersion (Non-Mode-Coupled); about to be released at time of printing.

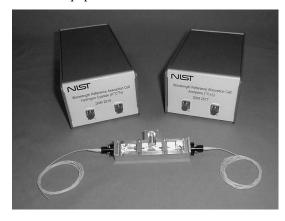
Recent Publications

- T. Dennis; E. M. Gill; and S. L. Gilbert, "Interferometric Measurement of Refractive-Index Change in Photosensitive Glass", *Appl. Opt.*, **40**, 1663-1667, (April 2001).
- S. D. Dyer; A. H. Rose; and K. B. Rochford, "Fast and Accurate Measurement of the Dispersion of Cascaded Components", *Tech. Dig., Optical Fiber Communication Conf. (OFC'01)*, ThB5 1-3, (March 2001).
- S. D. Dyer; and K. B. Rochford., "Low-Coherence Interferometric Measurements of the Dispersion of Multiple Fiber Bragg Gratings", *IEEE Photonics Tech. Lett.*, **13**, 230-232 (March 2001).
- J. B. Schlager; and A. H. Rose, "Annealed Optical Fiber Mode Scrambler", *Elect. Lett.*, **37**, 9-10, (January 2001).
- Lim, H.-J., DeMattei, R.C., Feigelson, R.S., and Rochford, K.B., "Striations in YIG Fibers Grown by the Laser-Heated Pedestal Method", *J. Crystal Growth*, **212**, 191-203, (2000).
- A. H. Rose; C. -M. Wang; and S. D. Dyer, "Round Robin for Optical Fiber Bragg Grating Metrology", *NIST JRES*, **105**, 839-866; (November 2000).
- S. M. Etzel, A. H. Rose, and C. –M. Wang, "Dispersion of the Temperature Dependence of the Retardance in SiO₂ and MgF₂", *Appl. Opt.*, **39**, 5796-5800, (November 2000).
- Rochford, K.B., Espejo, R.J., Rose, A.H., and Dyer, S.D., "Improved Fiber-Optic Magnetometer Based on Iron Garnet Crystals", *Proc.*, 14th Optical Fiber Sensors Conf., Venice, Italy, 332-335, (October 2000).
- A. H. Rose; P. G. Polynkin; J. Blake, "Electro-Optic Kerr Effects in Spun High-Birefringent Fiber Current Sensors", *Proc.*, 14th Optical Fiber Sensors Conf., Venice, Italy, 348-351, (October 2000).

Spectral and Nonlinear Properties

Project Goals

Develop techniques for the measurement of spectral and nonlinear properties of optical fiber and components. Develop wavelength-calibration transfer standards to help industry calibrate equipment.



Wavelength calibration Standard Reference Materials.

Customer Needs

This project concentrates on the needs of the optical-fiber communications industry; our focus is primarily on metrology for the characterization of optical fiber and components used in high-bandwidth systems. The rapid high-bandwidth optical-fiber growth communication systems has greatly increased the need for accurate measurement of spectral, dispersion, polarization, and nonlinear properties of optical fiber and components. This project concentrates on the spectral and nonlinear properties, and the Interferometry and Polarimetry Project concentrates on polarization and dispersion properties.

In an optical fiber communication system, wavelength division multiplexing (WDM) increases bandwidth by using many wavelength channels. Most systems employ 50 GHz or 100 GHz channel spacing (0.4 nm or 0.8 nm, respectively) in the 1540 nm to 1560 nm region, but narrower channel spacing may be implemented in the future. It appears likely that systems will be implemented in other wavelength regions as well, possibly covering the entire range from about 1280 nm to 1630 nm. WDM systems use a variety of optical components, and new components are under

development for next-generation systems. For effective system operation, the wavelength dependence of WDM optical components must be characterized and controlled. We are developing spectral characterization techniques and wavelength-calibration transfer standards to help industry evaluate optical components and calibrate wavelength-measuring instruments.

Future communication systems will increase the total bandwidth by increasing both the number of WDM channels and the bit rate per channel. Both increases will lead to higher optical power in the fiber, and will in turn increase the importance of nonlinear effects. These nonlinear effects, such as cross-phase modulation, selfphase modulation, and 4-wave mixing can cause pulse broadening, pulse distortion, and crosstalk, and ultimately limit system performance. On the other hand, beneficial nonlinear effects, such as Raman amplification, can be used to improve overall system performance through lower noise and the ability to amplify over the full WDM region. Because of the increasing importance of nonlinear effects, we are developing measurement techniques to characterize these effects in optical fiber.

Technical Strategy

Supporting WDM fiber and component metrology needs requires development and evaluation of new measurement techniques, dissemination of this knowledge, and, when appropriate, development of Standard Reference Materials or other calibration aids to help industry calibrate instrumentation. The project currently focuses on two areas: wavelength-calibration standards for WDM and nonlinear properties of optical fiber.

Wavelength calibration transfer standards: Wavelength standards are needed to calibrate instruments that measure the wavelengths of sources and characterize the wavelength dependence of WDM components. Fundamental references based on atomic and molecular absorption or emission lines provide the highest accuracy, but they are not available in all wavelength regions. The project currently produces two wavelength reference Standard Reference Materials (SRMs) based on fundamental molecular absorption lines: SRM

Technical Contact:

Sarah L. Gilbert

Staff-Years (FY 2001):

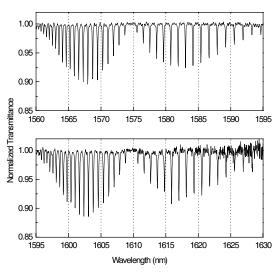
3 professionals 0.4 technicians 1.2 post docs

Funding Sources:

NIST Base (43 %) NIST Non-base (31 %) Other Government Agencies (26 %) 2517a (acetylene, high resolution) and SRM 2519 (hydrogen cyanide). Together these SRMs can be used to calibrate the wavelength scale of instruments between 1510 nm and 1565 nm. WDM will soon expand into other wavelength regions, such as the 1565 nm to 1625 nm L-band and the 1300 nm to 1500 nm region. Absorption lines of carbon monoxide can provide single-point and scan linearity calibration references in the L-band region. Artifacts such as etalons or fiber Bragg gratings can provide references at arbitrary wavelengths, but they can suffer from large sensitivity to temperature, strain, and pressure. We are developing a hybrid reference incorporating superimposed fiber Bragg gratings and a molecular absorption cell.

DELIVERABLE: Provide wavelength calibration Standard Reference Materials for the new WDM Lband (1565 to 1625 nm) by 2002.

DELIVERABLE: By 2002, develop hybrid artifact-molecular wavelength reference for other wavelength regions.



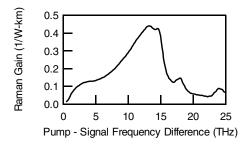
Spectrum of carbon monoxide wavelength calibration references for the WDM L-band (upper trace: ¹²C¹⁶O, lower trace: ¹³C¹⁶O).

High-accuracy wavelength standards: NIST needs higher accuracy internal wavelength references to calibrate its equipment. We have developed references for this purpose at 1560 nm and 1314 nm with uncertainties of a few megahertz. In collaboration with the NIST Time and Frequency Division, we have begun to develop an accurate frequency comb in the telecommunication wavelength region. This frequency comb can then be used to measure references throughout the 1280 nm to 1630 nm region.

DELIVERABLE: Demonstrate accurate frequency comb, based on femtosecond pulsed lasers, in the WDM telecommunications region and establish high-accuracy references for NIST internal calibration by 2003.

Nonlinear Properties: In 2001, we began a program on nonlinear properties of optical fiber and components. We are currently concentrating two areas: Raman amplification and supercontinuum generation in optical fiber. Raman amplifiers operate by sending a strong pump beam down the optical fiber at a wavelength ~100 nm below the weaker signal channels. Through stimulated Raman scattering, the strong pump beam then amplifies the weaker signals. Since the Raman gain depends strongly on the difference between the pump and signal wavelengths, often multiple pumps at different wavelengths are used to flatten the gain across the WDM region In addition to this beneficial, of interest. intentional Raman amplification, there will also be Raman interactions between the various WDM channels that can lead to a Raman gain tilt at the end of a long transmission fiber. In order to accurately predict the performance of both Raman amplifiers and the Raman gain tilt, a solid understanding of the Raman gain dependence on both signal and pump wavelengths is needed. We are developing measurement techniques accurately characterize Raman gain, including wavelength and polarization dependence.

DELIVERABLE: Complete calibrated measurements of the pump-wavelength dependence of Raman gain; investigate metrology for polarized Raman gain at low frequency offset by 2002.



Raman gain in standard telecommunication fiber at a pump wavelength of 1465 nm.

Optical fiber can host a complex combination of nonlinear effects such as cross-phase modulation, self-phase modulation, and 4-wave mixing. We have begun measurements in a system that exhibits these effects very strongly: supercontinuum generation in microstructure

and/or tapered optical fiber. This supercontinuum generation is important for the generation of broad optical frequency combs, which, in turn, can be used for accurate frequency measurements and wavelength standards. A better understanding of these complex processes in this strongly nonlinear regime should improve understanding of the behavior of future fiber-optic systems.

DELIVERABLE: By 2003, conduct experimental and theoretical study of noise processes in frequency comb and supercontinuum generation in microstructure fiber.

Accomplishments

- New Wavelength Calibration Standard Reference Material (SRM) Α wavelength reference that is now available as SRM 2517a, High Resolution Wavelength Calibration Reference for 1510-1540 nm. The SRM is a single-mode optical-fiber-coupled absorption cell containing acetylene (¹²C₂H₂) gas at a pressure of 6.7 kPa (50 Torr). The main difference between SRM 2517a and its predecessor, SRM 2517, is the use of lower pressure in the acetylene cell to produce narrower absorption lines. We made accurate measurements of the pressure-shift of the acetylene lines in order to certify the center wavelengths of 56 lines. Fifteen of the lines are certified with an uncertainty of 0.1 pm (about 12 MHz), two lines are certified with an uncertainty of 0.6 pm, and the remainder of the lines are certified with an uncertainty of 0.3 pm. This can be contrasted with SRM 2517, whose lines were certified with 0.6 pm uncertainty. Thus SRM 2517a extends the use of this SRM to higher resolution and higher accuracy applications.
- WDM L-Band Wavelength Calibration Reference Development We have completed pressure shift and broadening measurements for two isotopes of carbon monoxide (¹²C¹⁶O and ¹³C¹⁶O). These measurements will be used to determine certified wavelength values of wavelength calibration Standard Reference Materials for the new WDM L-band (1565 nm to 1625 nm). Based on the measurements, we expect to certify line centers with an uncertainty of about 0.5 pm.
- NIST Traceable Reference Material Plan Developed — Researchers in the Spectral and Nonlinear Properties Project and the Interferometry and Polarimetry Project have

developed a plan for the implementation of NIST Traceable Reference Materials (NTRMs) for fiber cladding diameter, polarization-mode dispersion, polarization dependent loss, and wavelength calibration. Under the plan, industry could sell calibration references that have a well-defined traceability linkage to NIST. This would reduce NIST's burden in meeting the demand for high-volume Standard Reference Materials (such as SRMs 2517a, 2518, 2519, and 2520) and would allow NIST staff to spend more time developing new metrology.

■ Raman Gain Measurement System Developed — We have developed a system to study the pump-wavelength dependence of Raman gain and have made preliminary measurements using this system.

Standard Reference Materials (SRM) See appendix D

SRM 2513, Mode Field Diameter Standard for Single-Mode Fiber; available.

SRM 2517a, High Resolution Wavelength Calibration Reference for 1510-1540 nm – Acetylene ¹²C₂H₂; available.

SRM 2519, Wavelength Reference Absorption Cell – Hydrogen Cyanide (H¹³C¹⁴N); available.

SRM 2520, Optical Fiber Diameter Standard; available.

Additional SRMs for optical fiber communications produced by NIST: SRMs 2522 & 2523 for optical fiber ferrule geometry and SRMs 2553-2555 for optical fiber coating diameter.

Recent Publications

- W. C. Swann and S. L. Gilbert, "Wavelength Calibration Standards for the WDM L-Band," in *Proc., Optical Fibre Measurement Conference*, Sept. 26-28 2001, Cambridge, UK
- S. L. Gilbert; W. C. Swann; and T. Dennis, "Wavelength Control and Calibration for Wavelength Division Multiplexed Optical Communication," in *Proc.*, 2001 IEEE/EIA International Frequency Control Symposium, (IEEE, Piscataway, NJ, 2001) pp. 122-126.
- S. L. Gilbert; S. D. Dyer; P. A Williams; and A. H. Rose, "Optical Metrology for Wavelength Division Multiplexed Fiber Communications," Optics and Photonics News, Vol. 12 No. 3, March 2001.
- T. Dennis; W. C. Swann; and S. L. Gilbert, "Wavelength References for 1300 nm and L-Band WDM," in *Optical Fiber Communication Conference*, OSA Technical Digest

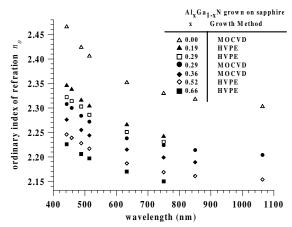
(Optical Society of America, Washington DC, 2001), paper WDD83.

- S. L. Gilbert and W. C. Swann, "Standard Reference Materials: Acetylene $^{12}C_2H_2$ Absorption Reference for 1510 nm to 1540 nm Wavelength Calibration SRM 2517a," NIST Special Publication 260-133 2001 Edition.
- S. L. Gilbert; W. C. Swann; and T. Dennis, "Wavelength Standards for Optical Communications," in *Proc., SPIE Laser Frequency Stabilization, Standards, Measurement, and Applications Conference*, Jan. 25-26, 2001, pp. 184-191.
- T. Drapela, "Effective Area and Nonlinear Coefficient Measurements of Single-Mode Fibers: Recent Interlaboratory Comparisons," in *Proc., Intl. Conf. On Applications of Photonics Tech. (ICAPT 2000)*, June 2000, 293-297.

Optical Materials Metrology

Project Goals

Develop complementary metrology methods of nonlinear optical analysis, X-ray diffraction imaging, and cathodoluminescence for studies of optical, electronic, and structural features of bulk and thin-film III-nitrides and nitride device structures. Accumulate a database of refractive index and birefringence for AlGaN and InGaN alloy semiconductors. Maintain established nonlinear optical analysis techniques for the uniformity analysis of LiNbO3 in support of device manufacturers of high-speed modulator telecommunications. devices for optical Develop prototype ultrafast and narrow-line cw solid-state waveguide lasers.



Ordinary refractive index of Al_xGa_{1-x}N measured by prism coupling.

Customer Needs

Customer needs fall into two main categories: (1) development of metrology suites for rapid non-destructive uniformity characterization of photonic materials in terms of optical, electronic, and structural characteristics for bulk crystals and thin film, (2) specialized ultrafast and continuous-wave solid-state waveguide lasers for applications in analog-to-digital conversion, characterization of high-speed optical detectors, and RF synthesis.

The success of our previous work in the nonlinear optical characterization of LiNbO₃ has led in a very natural way to extending this work into other important photonic materials such as the III-nitrides. GaN and related materials have made enormous recent economic impact with the realization of semiconductor

lasers and LEDs emitting in the blue. Other important applications of this material system include high-power, high-temperature and solar-blind UV detectors. transistors Problems with bulk and thin film growth of these materials remain, however, hindering the ability to meet the application demands. There persists a fundamental lack of understanding of the role of defects on the electrical, optical, and structural properties of these materials. These issues compel the development of new correlated metrology methods, with resolution on the scale of 20 to 100 nm. Furthermore, the lack of a database for the linear optical properties of the III-nitrides is hampering development of engineering design tools for blue-emitting GaN-based laser diodes.

Compact, solid-state waveguide lasers and amplifiers are emerging as a technology with impact ranging from telecommunication to high-speed signal processing to high-speed detector metrology. With assistance from industrial and DARPA collaborators, NIST has been on the cutting edge of developing technology for these application areas. Customer needs range from the optical characterization of laser glasses, to the testing of the impact of fabrication methods on laser performance, to the development of specialty lasers for detector frequency response measurements or all-optical, analog-to-digital conversion applications.

Technical Strategy

Nonlinear optics (NLO) offers rapid and versatile measurement capabilities that may be used to examine bulk and thin film materials at various stages of crystal growth and device processing. Furthermore, NLO methods may be directly correlated to other conventional measurement methods. Thus. material uniformity evaluated using NLO methods may, in many cases, be directly related to the material information derived from analytical methods that are not conveniently adapted to the manufacturing environment. For example, our strategy in applying nonlinear optical analysis methods to the study of bulk GaN single crystals relies on correlating the NLO results with X-ray diffraction imaging performed at Brookhaven National Laboratory collaboration with NIST Materials Science and Engineering Laboratory (MSEL). NLO is

Technical Contact: Norman A. Sanford

Staff Years:

2.7 professionals1 post-doc

Funding sources

NIST base (62 %) NIST non-base (15 %) Other government agency (23 %) extremely sensitive to such undesirable structural features as stacking faults, domain reversals, and mixed cubic and hexagonal phases. X-ray diffraction imaging reveals fullcrystal images of stacking faults and domain reversals, but is less sensitive to the presence of mixed phases. Thus, a combination of NLO analysis and X-ray imaging methods results in a reduction in ambiguity compared to the case where only one method is employed. Depthdependent artifacts are also problematic in GaN. During our year 2001 collaborations with MSEL, we found correlation between polishinginduced structural damage in bulk GaN with cathodoluminescence (CL) studies. We also examined the degree by which the damage could be removed using chemically-assisted ion-beam etching. We will continue this work in 2002 seeking further correlation with NLO studies.

The NLO work grew out of our earlier work with the characterization of LiNbO₃ whereby we have developed methods for fully non-destructive evaluation of composition and strain in finished wafers of the material. Studying LiNbO₃ wafers cut sequentially from full boules has allowed us to track these quantities and relate them to growth conditions of large crystals. We anticipate similar evolution of the NLO work aimed at the III-nitrides.

Analysis of thin-film GaN structures offers additional challenges due to the high density of structural defects and the impact of these defects on optical, electronic, and transport properties of these materials. Our strategy is to begin development of collection-mode near-field optical methods and examine the group III-nitrides using NLO and photoluminescence (PL) spectroscopies on a resolution scale of 20 to 100 nm.

DELIVERABLE: In 2002 and 2003, working closely with collaborators in the NIST MSEL and Chemical Science and Technology Laboratory (CSTL), we will correlate these near-field optical measurements with CL studies and other high-resolution electron beam and X-ray metrologies.

DELIVERABLE: In 2002, we will apply NLO analysis to examine the uniformity of transparent electrode structures on GaN, which is important for many configurations of light-emitting diode structures.

Establishing a database for linear optical properties of the AlGaN and InGaN compounds is also important, as we are receiving requests from industry and universities for these data. In 2001 we compiled refractive-index and birefringence data for a number of AlGaN

samples ranging in composition from pure GaN to pure AlN, using prism coupling to waveguide modes. The wavelength span of the measurements was from 442 nm to 1064 nm.

DELIVERABLE: In 2002 we will add to this refractive index database using additional samples of different AlGaN composition, extend the wavelength range further to the blue, and begin work on InGaN samples.

We are developing cw and mode-locked waveguide lasers using ion-exchange in rareearth-doped phosphate glasses. The optical, thermal, and chemical properties of the glass are measured and optimized in collaboration with a glass manufacturer. Unlike semiconductor laser sources, the solid-state sources benefit from a long upper-state, laser-transition lifetime (~10 ms) that allows low-noise production of modelocked pulses independent of repetition rate and low-noise generation of narrow-linewidth light. Unlike fiber laser sources, waveguide sources have sufficient rare-earth doping concentrations (~10²⁰ cm⁻³) to provide sufficient gain and power in short (~2 cm) lengths. This permits high repetition rates without the complications associated with harmonic-mode locking. We have demonstrated cw mode-locked operation using a semiconductor saturable absorber mirror We will explore hybrid mode-(SESAM). locking schemes to synchronize the pulse output to an external clock and improve SESAM performance by reducing the mirror's nonsaturable loss. Narrow-linewidth lasers are fabricated using a distributed Bragg reflector (DBR) etched directly onto the waveguide structure.

DELIVERABLE: In 2002 we will optimize performance of mode-locked lasers by reducing measured laser jitter, shortening the laser cavity, and optimizing waveguide fabrication techniques.

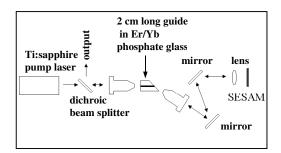
DELIVERABLE: In 2002 we will reduce DBR laser linewidth to 10 kHz or less via active and passive stabilization techniques. Noise reduction will also be pursued through the use of hybrid, bonded-glass technology.

Accomplishments

■ Nonlinear optical analysis was used to examine the optical and structural properties of GaN bulk crystals grown by high-pressure processing, and thin films of the material grown by HVPE, MOCVD, and MBE. NLO reveals evidence for stacking faults and mixed cubic/hexagonal phases in the bulk high-pressure grown GaN platelets. The existence of stacking faults and domain-inverted regions was

corroborated in the bulk GaN samples, using high-resolution x-ray diffraction imaging in collaboration with NIST MSEL researchers. Additionally, a number of AlGaN samples ranging in composition from pure GaN to pure AlN were examined. These were grown by methods of HVPE and MOCVD. An unexpected trend in the NLO coefficients for alloys of AlGaN was also observed. Symmetry considerations require that $\chi_{33} = -2\chi_{31}$ in these materials. We found that this is indeed obeyed for Al concentrations up to roughly 20 %. At higher concentrations of Al, χ_{33} rapidly decreases approaches zero, while χ_{31} monotonically but remains nonzero.

- our year-2000 studies that used X-ray diffraction imaging to examine CAIBE removal of polishing-induced damage in bulk GaN were correlated with CL studies in 2001. CL revealed that band edge-luminescence, that duplicated the result from as-grown faces of bulk GaN crystals, could be nearly recovered on the polished/CAIBE processed surfaces of the crystals after using CAIBE to remove 200 nm of the polished and damaged layer. However, X-ray diffraction imaging revealed that sufficient structural damage still remained that prevented imaging the interior microstructure of the crystals.
- Refractive index and birefringence measurements were performed on a number of film AlGaN samples ranging composition from pure GaN to pure AlN. The wavelength range spanned 442 nm to 1064 nm. The full birefringence of the films substrates were accounted for in the analysis. Measurement uncertainty typically ± 0.004 . This work will contribute to a database on linear optical properties that is needed by the industry.



Schematic of cw mode-locked waveguide laser operating at 1534 nm.

- A mode-locked waveguide laser with record performance was demonstrated. Nearly transform-limited pulses of 5.8 ps duration at fundamental repetition rates up to 511 MHz were generated from a passively mode-locked erbium/ytterbium co-doped planar waveguide laser in an extended-cavity configuration. Averaged output power was as high as 20 mW. This represents a five-fold increase in fundamental mode-locked repetition frequency and a 14-fold increase in averaged output power over previously reported results for such lasers. Diode-pumped, mode-locked operation, which is critical for compact devices, was also demonstrated.
- A landmark 17 kHz linewidth was demonstrated in a rare-earth-doped DBR waveguide laser operating near 1550 nm. The result was obtained using a laser diode pump but required no external locking, feedback or stabilization schemes of any kind. The linewidth is competitive with the best reported results for stabilized ring fiber lasers, but the waveguide lasers are far simpler than those devices. Furthermore, the measured linewidth is much narrower than the 1 MHz or greater linewidth typical for semiconductor DFB laser diodes.

Recent Publications/Presentations

"Optical, Nonlinear Optical, and X-ray Analysis of GaN and AlGaN Grown by HVPE and MOCVD on Sapphire," N. A. Sanford, A. V. Davydov, D. V. Tsvetkov, V. A. Dmitriev, S. Keller, U. K. Mishra, and S. P. Den Baars, *Fourth International Conference on Nitride Semiconductors (ICNS-4)*, July 16-20, 2001, Denver, CO, paper P17.7.

"Nonlinear Optical Analysis of LiNbO₃ and GaN", N. A. Sanford, 13th International Symposium on Integrated Ferroelectrics, March 11-14, 2001, Colorado Springs, CO, Paper 7.1.1I. (invited)

"Examination of Bulk GaN Crystals by Nonlinear Optical Analysis and High-Resolution X-ray Diffraction Imaging," N. A. Sanford, B. Steiner, *Materials Research Society Spring Meeting*, April 16-20, 2001, San Francisco, CA. Paper E2.5.

"Mode Locked Erbium/Ytterbium Co-doped Waveguide Laser," J. B. Schlager, B. E. Callicoatt, K. J. Silverman, R. P. Mirin, N. A. Sanford, D. L. Veasey, *Conference on Lasers and Electro-optics, CLEO-2001*, May 2001, Baltimore, MD. Paper CMS1.

"Crystal Growth, Characterization, and Domain Studies in Lithium Niobate and Lithium Tantalate Ferroelectrics," V. Gopalan (Penn State University), N. A. Sanford and J. A. Aust (NIST, USA), K. Kitamura (NIRIM, Japan), in Handbook of Advanced Electronic and Photonic Materials and Devices, edited by H. S. Nalwa, Vol. 4: Ferroelectrics and Dielectrics, Academic Press (2001). (invited)

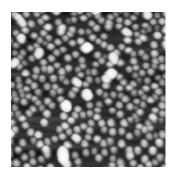
"Hybrid Glass Substrates for Waveguide Device Manufacture," S. D. Conzone, J. S. Hayden, D. S. Funk, A. Roshko, D. L. Veasey, Opt. Lett. 26(8): 509-511, Apr 15, 2001

"Design and Optimization of a Diode-Pumped Fiber-Coupled Yb:Er Glass Waveguide Laser," W. Liu, S. N. Houde-Walter, D. L. Veasey, A. P. Peskin, Appl. Opt. 39(33): 6165-6173; Nov 20, 2000.

Nanostructure Fabrication and Metrology

Project Goals

Develop modeling, fabrication, and measurement methods for semiconductor nanostructures, including quantum dots and photonic crystals. Develop fundamentally new devices for single-photon sources and detectors and entangled-photon sources.



AFM image of self-assembled InGaAs quantum dots grown by molecular beam epitaxy (image width is 1 μ m).

Customer Needs

Semiconductor nanostructures, especially selfassembled quantum dots and photonic crystals, are emerging as an important future direction for optoelectronics. Photonic crystals offer a wide range of possibilities, ranging from improved light-emitting diodes to photonic integrated circuits to spontaneous emission control. Semiconductor quantum dots have an atomic-like density of states that offers opportunities for substantial unique improved devices. These include ultra-lowthreshold laser diodes and semiconductor optical amplifiers with reduced cross-gain modulation. Several fundamental properties of quantum dots have yet to be measured, including resonant absorption cross-sections and homogeneous linewidth. These set limitations on device operation.

The ability to generate and control single photons offers new opportunities to meet customer needs in quantum-based radiometric measurements where optical power is measured by counting photons. Quantum cryptography is an emerging form of ultrasecure communications that requires an on-demand source of single photons. The single-photon turnstile will allow us to meet the requirements

of this new technology. The single photon turnstile can be extended to generate pairs of entangled photons. These entangled photon pairs are useful for a wide range of applications, from fundamental tests of quantum mechanics to reduced dimensions with optical lithography.

Technical Strategy

We are using molecular beam epitaxy (MBE) to grow self-assembled quantum dots. Reflected high-energy electron diffraction (RHEED), atomic force microscopy (AFM), transmission electron microscopy (TEM) techniques are applied to characterize quantumdot morphology as a function of growth parameters. Photon counting and cavity ringdown methods are required to measure the properties of optical semiconductor nanostructures, particularly single quantum dots or small ensembles of dots.

DELIVERABLE: In 2002 we will develop multiple reflection and cavity ring-down spectroscopy systems for the high-precision absorption measurement of semiconductor quantum dots and the reflectance measurement of distributed Bragg reflectors.

We have developed ultrafast pump-probe methods to characterize carrier lifetimes and are measuring the homogeneous linewidth via transient four-wave mixing. Collaborators in JILA and the NIST Physics Laboratory are contributing to various aspects of nanostructure measurement and modeling.

DELIVERABLE: We will complete measurements of polarization-dependent absorption of ensembles of self-assembled quantum dots in a waveguide structure in 2002.

The fabrication of photonic crystals requires the precision growth and anisotropic etching of semiconductor epilayers. We are using a chemically assisted ion-beam (CAIBE) system to etch semiconductor nanocavities in layers grown by MBE.

DELIVERABLE: In 2002-2003, we will apply finite-difference time-domain (FDTD) and Green's function methods to model these nanocavities and interpret the results of optical and structural measurements.

Single-photon-turnstile (SPT) operation relies on a large Coulomb blockade effect, which we have shown to exist for self-assembled quantum dots. We therefore expect the Coulomb **Technical contact**: Richard Mirin

Staff-Years:

2.6 professional4.0 postdoctoral

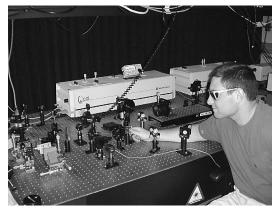
Funding:

NIST base (41 %) NIST nonbase (54 %) Other government agency (5 %) blockade to be observable near room temperature. The photon repetition rate of the SPT is controlled by the frequency of an applied AC voltage, allowing precisely defined emission of single photons. The ultimate goal is the embedding of a SPT inside a photonic crystal nanocavity, which will enable enhanced emission efficiency and directionality. Work has also begun on the development of a single-photon detector.

DELIVERABLE: We will demonstrate an optically driven photon turnstile in 2002 and measure SPT operation with a single-electron pump in 2003.

Accomplishments

■ This past year a co-linear, co-polarized pump-probe spectroscopy system assembled and applied to the measurement of carrier lifetime in quantum dots in semiconductor optical amplifiers and in quantum wells in semiconductor saturable absorbers. The long excited-state lifetimes (>100 ps) observed in InGaAs quantum-dot optical amplifiers are evidence of a phonon bottleneck. Initial measurements of the coefficient of self-assembled absorption semiconductor quantum dots were performed; they showed surprisingly strong polarization dependence.



Ultrafast measurement system with tunable wavelength for pump-probe characterization of compound semiconductor structures.

■ In collaboration with researchers in the Electromagnetic Technology Division of the Electronics and Electrical Engineering Laboratory (EEEL), we measured the capacitance from ensembles of self-assembled InGaAs quantum dots. The Coulomb blockade observed was about 30 meV, which is larger than previous reports by almost 20 %. Models

of the tunneling rates and capacitance of quantum dots were developed. Setup of the laboratory for single photon measurements was completed to support characterization of the single photon turnstile and the optical properties of individual quantum dots.

- We demonstrated multi-mode lasing of InAs/GaAs quantum dot lasers at room temperature immediately above threshold. The lasing modes were separated by about ten times the Fabry-Perot mode spacing, with several dark modes between the lasing modes. simulations indicate equation that multimode behavior can be explained by a homogeneous broadening that is on the order of Narrow homogeneous the mode spacing. broadening leads additionally to a reduction of cross-gain modulation in a quantum-dot semiconductor optical amplifier.
- Semiconductor quantum-dot formation by the dewetting of an InAs epilayer on GaAs was observed for the first time. As evidenced by RHEED an initial measurements, dimensional epilayer of InAs evolves as the sample cools to form three-dimensional islands. We also showed that the critical thickness for the 2D-3D transition of an InAs/GaAs film is not a simple function of substrate temperature, but depends also on other parameters such as deposition temperature. RHEED patterns measured during reheating and recooling of the samples demonstrate the reversibility of the 2D-3D transition. The fact that InAs epilayers can evolve during quenching from growth temperature may have implications on the accuracy of measurements of the shapes and sizes of quantum dots formed at growth temperature.
- Construction of a chlorine-based chemically-assisted ion beam etcher (CAIBE) was completed, and operations have commenced. High surface-quality etching has been demonstrated. The CAIBE will enable the fabrication of two-dimensional and three-dimensional photonic bandgap structures.
- Laser-diode packaging technology, including indium-solder die-attach and electron-beam deposition of antireflection coatings, was developed. This supports a project on the development of tunable lasers for the measurement of the spectral responsivity of photodetectors.

Recent Publications/Presentations

- R.P. Mirin, K.L. Silverman, and B. Sonnenberg-Klein, "Quantum Dot Semiconductor Optical Amplifiers," Materials Research Society Fall Meeting 2000, paper J4.2, November 27-December 1, 2000, Boston, MA. (Invited)
- K.L. Silverman, B. Sonnenberg-Klein, and R.P. Mirin, "InAs/GaAs quantum dot semiconductor optical amplifers for WDM systems," American Physical Society March Meeting 2001, paper Q33.007, March 12-16, 2001, Seattle, WA.
- S.A. Anders, C. Kim, M. Keller, and R.P. Mirin, "Evidence for two coexisting sets of self-assembled InGaAs quantum dots", poster R40.192, American Physical Society March Meeting 2001, March 12-16, 2001, Seattle, WA.
- B. Sonnenberg-Klein, K.L. Silverman, and R.P. Mirin, "Multimode Lasing from Room Temperature InAs/GaAs Quantum Dot Lasers" SPIE ITCOM 2001 Symposium on Semiconductor Lasers for Lightwave Communications Systems, August 20-24, 2001, Denver, CO.
- R.P. Mirin and A.C. Gossard, "Growth, Characterization, and Applications of Self-Assembled InGaAs Quantum Dots", in *Quantum Semiconductor Devices and Technologies*, Chapter 5, 184-231, Kluwer Academic Publishing Group, Norwell, MA, 2000.

Semiconductor Growth and Devices

Technical Contact: Kris Bertness

Staff Years:

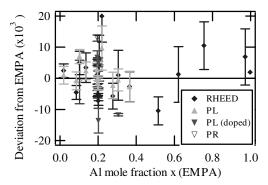
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Funding Sources:

NIST base (60 %) NIST non-base (23 %) Other Agency/CRADAs (17 %)

Project Goals

Develop measurement methods and provide data to support the efficient manufacture of semiconductor optoelectronic devices. Provide advanced materials and devices to support research in industry, other parts of NIST, universities and government laboratories.



Deviations of Al mole fraction measurement from EMPA mole fraction value for various composition measurement techniques.

Customer Needs

The rapid growth of the U.S. optoelectronics industry is dependent on the high-yield manufacture of devices with increasingly tight specifications. Compound semiconductor materials form the basis for LEDs, lasers, photodetectors, and modulators critical to optical communication, display, data storage, and many other applications. Issues of materials purity and uniformity are at the foundation of device yield and performance. Measurements of starting materials and epitaxial layers must be supported by standard procedures and reference materials. Increasingly, the needs are for accurate in-process measurements. semiconductor devices now structures with a high degree of strain, and characterization of this strain relationship to device failure is an important area of research. In addition, specialty devices are needed for use in metrology systems inside and outside of this project.

Technical Strategy

Inaccuracy of semiconductor composition measurement has been an impediment to achieving consistency of device performance across production lines. It has also inhibited the collection of sufficiently accurate materials parameters for use in the simulation of devices, which is critical to fast product cycle times. The problems have been exacerbated by the increase in outsourcing of epitaxial growth. A goal of this project is to develop certification techniques for standard reference materials having composition uncertainty specified to a level over ten times lower than that of techniques currently in use by industry. Our approach is to combine conventional methods of composition determination (photoluminescence (PL), photo-reflectance (PR), and x-ray diffraction (XRD)) with less common methods (in situ monitoring, electron microprobe analysis (EMPA), and quantitative chemical analysis) to enable certification of alloy composition. This program will pave the way for future production of standard reference materials (SRMs) in the AlGaAs alloy system. As part of this research, we are quantifying error sources and accuracy limits of the indirect composition measurement techniques currently in use by industry, specifically PL and XRD. We are also demonstrating the composition accuracy that can be achieved with direct microanalytical techniques such as EMPA in combination with accurate reference artifacts and carefully chosen correction procedures.

DELIVERABLE: By 2002, we will develop AlGaAs composition standards with relative mole fraction uncertainty of \pm 2 % or less.

In a related project, the accuracy of InGaAsP composition analysis is being assessed through an interlaboratory comparison of conventional ex-situ characterization techniques: PL and XRD. This program seeks to develop reliable methods for measurement and data analysis that can be used throughout the industry. A set of six samples with three different compositions is being analyzed by industry, government and university laboratories. By examining the different operating and analytical conditions used in the various laboratories and how they affect the data, the importance of the measurement variables for the techniques will be assessed. A necessary aspect of the study will be to distinguish between variations intrinsic to the materials and those caused by the measurement systems or techniques. Through repeated measurements and cross-correlation, this work will help clarify appropriate measurement and calculation methods. Finally, we are collaborating with the NIST Materials Science and Engineering Laboratory to evaluate Raman spectroscopy as a possible method to quantify strain in InGaAsP.

DELIVERABLE: By 2002, we will report the conclusions of the study including analysis of important variables to control ex-situ characterization of InGaAsP. By 2003, we will publish recommended procedures for the evaluation of composition and strain in InGaAsP.

Contamination is a serious problem in phosphine, arsine, silane, ammonia, and similar gases used in the epitaxial growth of high-purity semiconductor layers. Semiconductor device manufacturers have expressed frustration with the irreproducibility of source material purity from vendor lot to vendor lot and as the vendors change process or packaging. The issue is described by industry as primarily a measurement problem in that manufacturers frequently report impurity levels as "not detected" for a series of lots that produce very different device results. The critical concentrations of the impurities are not well known; however, it is believed that >10 nmol/mol oxygen or water in phosphine is undesirable. In collaboration with researchers in the NIST Chemical Science and Technology Laboratory, this project is developing a cavityring-down spectroscopic technique to measure impurities with very low concentrations in semiconductor source gases. In this technique a high-finesse optical cavity is filled with the sample gas and then pumped with light that is strongly absorbed by the impurity molecule. The light is abruptly shuttered, and the decay time can be simply related to the impurity concentration. The advantages of this technique are that its accuracy relies primarily on accurate time measurement and detector linearity, and it is insensitive to absorption outside the cavity. The initial focus is on measuring water content in phosphine gas. Progress to date has been challenged by the frequency instabilities in commercially available tunable lasers.

DELIVERABLE: By 2002, we will develop and prove cavity ring-down spectroscopy as a technique for measuring water in phosphine to 10 nmol/mole, calibrated to humidity standards. By 2003, we will measure water concentrations in ammonia to 10 nmol/mole.

Native-oxide layers have a significant impact on photonic devices due to their ability to provide both electrical and optical confinement, similar to native SiO₂ in integrated Si technology. They are used as confinement apertures and/or broadband antireflection (AR) coatings in cavity vertical surface emitting lasers (VCSELs), photodetectors, light-emitting diodes, and saturable Bragg mirrors. In particular, the use of AlGaAs oxide apertures in VCSELs has resulted in record low threshold currents and high efficiencies. However, AlGaAs layers undergo a relatively large contraction during oxidation, resulting in strain in the adjacent GaAs layers. This frequently causes structures containing AlGaAs oxide layers to delaminate and/or fail. To address this issue we are collaborating with the NIST Materials Science and Engineering Laboratory on imaging strain in these materials using highresolution convergent-beam electron diffraction (CBED). This technique allows the identification of strains as small as 0.02 %, with lateral resolution on a nanometer scale. We will study how the strain in these structures varies as a function of the oxidation and post-oxidation processing conditions.

DELIVERABLE: By 2002, we will characterize the strain in GaAs layers adjacent oxidized AlGaAs layers.



Atomic force microscopy of semiconductor nanostructures.

Quantum dots are semiconductor structures with quantized energy levels that result in improved efficiency and tuning range for semiconductor lasers and less sensitivity to environmental changes for lasers and photodetectors. Dot formation is driven by strain during epitaxial crystal growth, but measuring the strain in structures less than 100 nm in lateral dimension presents new challenges. We are contributing to this field by making careful studies correlating growth parameters with dot density and size as measured by atomic force microscopy (AFM).

We are also evaluating strain in the region of the dots with transmission electron microscopy (TEM).

DELIVERABLE: By 2002, we will evaluate the structure and density of buried dots as compared to dots on a bare surface, and examine the correlation between dot density and dot height for different growth conditions.

Collaborations with industrial, university and government laboratories leverage division resources and provide valuable feedback to our programs. Collaborations can range from growth of a few device structures to multiyear Cooperative Research and Development Agreements (CRADAs).

Accomplishments

- AlGaAs SRMs—In this past year the sources of uncertainty in two techniques for measuring $Al_xGa_{1-x}As$ composition accurately quantified and in many cases Using specimens provided by the Semiconductor Growth and Devices Project, collaborators in two other NIST divisions measured the uncertainties in PL and EMPA composition determination. PL data were used to refine the conversion of band-gap energy E as a function of mole fraction x to the value $dE/dx = 1.402 \pm 0.010$ eV. The main sources of uncertainty in the PL measurements arise from temperature drift of the specimens (apparent shift in x of up to 4×10^{-4} /K, depending on composition and doping), and band-edge energy shifts when carrier concentrations exceed 1 x 10¹⁷ cm⁻³. The shifts in PL peak energy are nonlinear and could be misinterpreted as shifts in x of up to 0.011 for specimens with carrier concentrations typical for a heavily doped device layer. The EMPA analysis was expanded to make use of cross-correlation between data taken at different accelerating potentials in order to reduce the reliance on calibration standards.
- AlGaAs SRMs—The accuracy of composition measurements using situ metrology has also been improved. RHEED analysis of data taken under different conditions has shown that the largest uncertainty in the growth rate measurement comes from variation of the flux across the specimen. The interference between oscillations in growth rate from different parts of the sample also introduces a systematic error of about 2 % in the growth rate measurements. Flux transients were

found to contribute errors from 0 % to 3 % depending on growth conditions. Specimen crystal orientation was found to have no measurable effect on the apparent growth rate. By controlling the relevant experimental parameters, it was possible to reduce the uncertainty in x as measured by RHEED from ± 3 % to ± 1 % (relative) for most specimens. With assistance from the NIST Information Technology Laboratory, Statistical Engineering Division, the optical reflectance spectroscopy (ORS) reflectance model was found to include inherent nonlinearities that resulted in a high sensitivity of curve-fitting parameters to noise in the data even at the 0.1 % level. Hardware modifications to the optical reflectance spectroscopy system lowered the noise and drift in the system from 0.5 % and 3 % to 0.1 % and respectively. However, 1.5 %, there intermittent electrical noise in the laboratory environment that still occasionally raises the noise floor, and it is clear that even a noise level of 0.1 % is insufficient to determine film composition from the index of refraction to better than $\pm 10\%$ relative.

- InGaAsP composition—The initial results of the interlaboratory comparison data on InGaAsP have shown that there is good agreement among the x-ray diffraction peak separation measurements, but more variation in the photoluminescence peak positions. result is particularly relevant because companies tend to rely on photoluminescence as their primary characterization technique for production material. NIST also constructed a PL measurement system optimized for this project, and added mapping capabilities to its x-ray data software analysis.
- Semiconductor source gas impurities—The ring-down cavity system was converted to 935 nm in order to use a more stable external-cavity diode laser source. Attempts to improve the ring-down signals generated with the 1336 nm laser were abandoned when it was shown that even a transfer-cavity locking system could not react quickly enough to stabilize the laser fluctuations. New collaborations were initiated within NIST and with two industrial gas companies to explore the potential of this technique. The ring-down cavity safety review for phosphine use was completed this year, and the cavity was used to confirm that phosphine gas would not damage the high-reflectivity mirrors used in this experiment.

- Native oxides of AlGaAs—The location of the specimens within the oxidation tube furnace was found to have a substantial effect on reproducibility of the oxidation rate of AlGaAs native oxide layers. Suspending the specimens in the center of tube, rather than allowing them to rest near the bottom, improved run-to-run reproducibility by a factor of five. improvements allowed a detailed study of the oxidation kinetics using the Deal-Grove model, which was developed for the thermal-oxidation kinetics of Si. The model takes into account the reactions at the two oxide layer boundaries as well as diffusion between the boundaries. Under the annealing conditions used so far, various times at 460 °C, the oxidation was found to be surface-reaction-limited for compositions up to Al_{0.94}Ga_{0.06}As, diffusionlimited for AlAs, and mixed for compositions in between.
- Native oxides of AlGaAs—SIMS analysis of AlGaAs native oxide specimens resolved an ongoing mystery as to why oxidation rates varied substantially among MOCVD specimens grown by two collaborators and MBE specimens grown at NIST. The SIMS data showed that the Al concentration within the layers varied substantially from the nominal values for certain specimens. After correcting for the concentration variations, we were able to demonstrate that neither the epitaxial-growth method (MBE and MOCVD), the V/III ratio, nor the impurity concentration affect the oxidation rate of the AlGaAs layers.
- Strain in semiconductor structures—Crosssectional specimens of partially oxidized AlGaAs layers buried in a GaAs matrix were prepared for TEM/CBED analysis using conventional methods. Specimens yielded a relatively large area of material sufficiently thin for TEM and CBED imaging, enabling assessment of diffraction-pattern quality as well as spatial resolution. Preliminary TEM examination and CBED patterns demonstrated that the preparation procedure does not damage the samples. Simulations of the CBED patterns showed that the level of strain in these specimens should be large enough to distinguish from unstrained GaAs.
- Quantum dots—Growth of quantum dot InGaAs on GaAs was examined with AFM and TEM. The heights and densities of quantum dots as determined by AFM show that the density of dots decreases with increasing

- temperature, while the height of the dots increases. For some of the samples a bimodal distribution of dot heights was observed. This distribution typically occurs at high temperatures and is probably due to Ostwald ripening, by which a few larger dots grow at the expense of smaller dots in order to reduce the overall interfacial energy of the system.
- Quantum dots—Because quantum dots are typically sharper than the tips used for AFM imaging, each dot actually produces an image of the AFM tip in the AFM images. To measure the size and shape of dots, high-resolution TEM was used. These micrographs revealed that there is a fairly large distribution of dot sizes in InAs quantum dots deposited at 540 °C. The dots are slightly elongated along the [110] direction with an aspect ratio of ~1.75, and no apparent dependence on the dot size. At a higher magnification, moiré fringes are imaged. These fringes can be used to measure both the dot dimensions and the lattice mismatch between the dots and the substrate.
- NIST and Systine, Inc (formerly Materials Research Source, LLC) modified an existing CRADA to include more work on the development of nanopatterned substrates for growth of ordered arrays of semiconductor quantum dots.
- Materials were provided to two other groups at NIST to evaluate magnetic spin effects in field-emission transistors and to evaluate the suitability of using Raman spectroscopy as a method for certifying doping level in compound semiconductor thin films.

Recent Publications

K. A. Bertness, J. T. Armstrong, R. B. Marinenko, L. H. Robbins, A. J. Paul, J. G. Pellegrino, P. M. Amirtharaj, and D. Chandler-Horowitz; "AlGaAs Composition Measurements from *In Situ* Optical Reflectance," Newsletter of the Lasers and Electro-Optics Society (LEOS, an affiliate of IEEE), Volume 14, No.5, pp 15-16.

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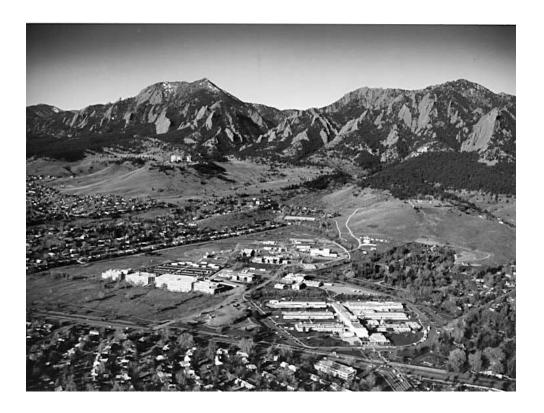
Appendix A: Major Laboratory Facilities

Semiconductor Growth and Optoelectronic Device Fabrication

The Division makes use of a gas-source molecular-beam epitaxy system and associated in situ and ex situ measurement equipment for III-V semiconductor growth and characterization. It also maintains a cleanroom facility for thin-film deposition, photolithography, and wet and dry etching. The facilities support the activities described above, particularly for the Optical Materials Metrology, Nanostructure Fabrication and Metrology, and Semiconductor Growth and Devices Projects.

Laser Power/Energy Detector Calibration Systems

The Optoelectronics Division has established and maintains several state-of-the-art measurement systems for calibrating most types of laser power and energy detectors. These measurement systems incorporate unique, specially designed, electrically calibrated, laser calorimeters that are used as primary standards. The calorimeters are used in conjunction with beamsplitter-based optical systems to provide measurement services for laser power and energy that cover a wide range of powers, energies, and wavelengths for detectors used with both cw and pulsed lasers. This assembly of laser power and energy detector calibration systems represents the best overall capability of this kind in the world. In many cases (e.g., excimer laser measurements at 248 nm and 193 nm), the Division has the only measurement capability in the world.



Appendix B: NRC Postdoc and Other Research Opportunities

National Research Council Associateship Opportunities

The National Institute of Standards and Technology (NIST), in cooperation with the National Research Council (NRC), offers awards for postdoctoral research in many fields. These awards provide a select group of scientists and engineers an opportunity for research in many of the areas that are of deep concern to the scientific and technological community of the nation. NIST, with direct responsibilities for the nation's measurement network, involves its laboratories in the most modern developments in the physical, engineering, and mathematical sciences and the technological development that proceed from them. The Research Council, through its Associateship Programs office, conducts an annual national competition to recommend and make awards to outstanding scientists and engineers at the postdoctoral level for tenure as guest researchers at participating laboratories. The deadline for applications is January 15, 2002 for appointments beginning between July 2002 and the following January 2003.

The objectives of the Programs are:

- To provide postdoctoral scientists and engineers of unusual promise and ability opportunities for research on problems, largely of their own choosing, that are compatible with the interest of the sponsoring laboratories.
- To contribute thereby to the overall efforts of the federal laboratories. Eligibility requirements include U.S. citizenship and receipt of Ph.D. within 5 years of application. NRC positions involve a two-year tenure at NIST, and the annual basic salary for the 2002 program year is \$53,200.

For more detailed information, including instructions for applicants, please contact the Optoelectronics Division Office and request a copy of the NRC Postdoctoral Opportunities booklet. You may also visit the NRC Research Associateship Program web page (http://national-academies.org/rap) to see a list of opportunities within our division.

Opportunities for the year 2002 with the Optoelectronics Division through the NRC Research Associateship program:

- Deep Ultraviolet Laser Metrology
- High-Speed Optoelectronics Measurements
- High-Speed Optical Receivers and Optoelectronic Integrated Circuits
- Ultrashort Optical Pulse Characterization for High-Speed Measurements
- Modeling and Simulation of High-Speed Optoelectronic Devices
- Intensity Noise in Optical Fiber Amplifiers and Transmitters
- Chromatic Dispersion Metrology
- Polarization-Mode Dispersion Metrology
- Polarization Metrology for Optical Telecommunications
- Low-Coherence Interferometry for Fiber Optic and Component Metrology
- Nonlinear Properties of Optical Fiber and Components
- Wavelength Standards for Optical Communications
- Fiber Bragg Gratings and UV Photosensitivity
- Study of Compound Semiconductor Native Oxides
- Quantum-Dot Morphology
- Photonic Crystals and Optical MEMS
- Semiconductor Quantum Optics
- Ultrafast and CW Optical Frequency Synthesis
- Nanoscopic Wide-Bandgap Materials Characterization by CW and Ultrafast Nonlinear Optics
- In-Situ Metrology of Epitaxial Crystal Growth for Semiconductor Optoelectronics

Professional Research Experience Program (PREP)

The Professional Research Experience Program (PREP) is designed to provide valuable laboratory experience to undergraduate and graduate students from the University of Colorado at Boulder and from the Colorado School of Mines at Golden, and to recent Ph.D. recipients from these and other universities. Students and postdocs are employed by the University of Colorado or the Colorado School of Mines and normally carry out research at the NIST Boulder Laboratories.

Students are usually hired just before the spring, fall, or summer terms, and may be employed for one or more terms. Postdocs may begin any time during the year. Applications are accepted throughout the year.

NIST pays in-state tuition for PREP undergraduate students during the fall and spring semesters and an hourly wage. Graduate students receive in-state tuition and a stipend. Postdocs receive a stipend.

Eligibility requirements include U.S. citizenship or permanent residency and, for students, a minimum 3.0 GPA (grade point average).

An application form and further information are available from:

Phyllis Wright, Student Outreach Coordinator NIST, MC 360 325 Broadway Boulder, CO 80305

Phone: (303) 497-3244

Appendix C: Conferences and Workshops

Symposium on Optical Fiber Measurements

The Optoelectronics Division, in cooperation with the Optical Society of America and the IEEE Lasers and Electro-Optics Society, organizes the biennial Symposium on Optical Fiber Measurements held in Boulder in the fall of even-numbered years. Check the Division web page or call the Division Office for the date of the next Symposium. This Symposium is a 2 ½-day meeting devoted entirely to the topic of measurements on fiber, related components, and systems. It provides a forum for reporting the results of recent measurement research and an opportunity for discussions that can lead to further progress. It consists entirely of contributed and invited papers.

Experimental and analytical papers on any measurement aspect of guiding lightwave technology are solicited for the Symposium. Subjects and measurements include:

Optical Fibers

Telecom, sensors, fiber lasers/amplifiers

Integrated Optics

Planar waveguides, photonic crystals, MEMs

Components

Amplifiers, lasers, detectors, modulators, switches, couplers

Systems

Long haul, LANs/subscriber loops, WDM, TDM

Standards

Field and laboratory instrumentation

Examples of typical measurements include:

Attenuation/loss Four wave mixing efficiency
Chromatic dispersion Index of refraction profile

Crosstalk Mode-field diameter
Cutoff wavelength Nonlinear coefficients

Effective area Polarization dependent loss
Effective index Polarization-mode dispersion

A limited number of Digests from previous Symposia are available, as well as a CD-ROM with collected papers from 1980-2000. For information on obtaining either, or for information on upcoming Symposia, please contact the Optoelectronics Division Office or visit our web page, http://www.boulder.nist.gov/div815/.

Laser Measurements Short Course

The Optoelectronics Division, in cooperation with the University of Colorado at Boulder, offers an annual Short Course on Laser Measurements. Check the Division web page or call the Division Office for the date of the next short course. The 3-1/2 day course emphasizes the concepts, techniques, and apparatus used in measuring laser parameters. A tour of the NIST laser measurement laboratories is included. The faculty consists of laser experts from NIST, industry, and other government agencies. A degree in physics or electrical engineering, or equivalent experience is assumed, and some experience in the use of lasers is desired for attendees.

Appendix D: Calibration Services and Standard Reference Materials

	Laser Power and Energy Calibrations			
	Laser	Wavelength	Range	
CW	He Cd	325 nm	100 μW to 50 mW	
	Argon	488,514 nm	1 μW to 1 W	
	HeNe	633 nm	1 μW to 10 mW	
	Diode	830 nm	1 μW to 20 mW	
	Nd:YAG	1064 nm	1 μW to 450 W (6 kW off-site)	
		1319 nm	1 μW to 50 mW	
	Erbium	1550 nm	1 μW to 30 mW	
	CO ₂	10.6 μm	100 mW to 1kW (6 kW off-site)	
Pulsed	KrF	248 nm	5 μJ/pulse to 250 mJ/pulse	
			50 μW to 7 W average	
	ArF	193 nm	5 μJ/pulse to 5 mJ/pulse	
			10 μW to 3 W average	
			Dose Measurements	
	F_2	157 nm	[Fall 2002]	
	Nd:YAG	1064 nm	1 mJ to 50 mJ/pulse (1 to 20 Hz prf)	
			[300 mJ/P Fall 2001]	
			10 ⁻⁴ to 10 ⁻⁸ W(peak)	
			10 ⁻¹¹ to 10 ⁻¹⁵ J/pulse	

Optical Fiber Power Meters and Detectors				
Parameter Wavelength		Range	Status	
Absolute Power Calibration	670, 780, 850, 980, 1310, 1550 nm	10 to 100 μW	Calibration Service	
	980 nm [1480 nm Fall 2001]	1 to 200 mW	Special Test	
Optical Receiver	1319 nm	300 kHz to	Calibration Service	
Frequency Response		50 GHz		
	850 and 1550 nm	1 MHz to	Special Test	
		50 GHz		
Optical-Fiber Power- Meter Linearity	850, 1310, 1550 nm	60 to 90 dB	Calibration Service	
Reference Receiver Impulse Response	800 and 1550 nm	100 fs impulse source	Special Test	
Optical-Detector Spatial Uniformity	635, 850, 1300, 1550 nm		Special Test	
Optical-Fiber Power- Meter Spectral Responsivity	400 to 1700 nm		Calibration Service	
Relative Intensity Noise (RIN)	1550 nm	0.1 to 4.1 GHz	Calibration Service	

For more information on calibration services for optoelectronics, contact: Marla L. Dowell, Acting Group Leader at (303) 497-7455, mdowell@boulder.nist.gov.

Standard Reference Materials						
SRM Number	Name Brief Description		Availability			
	Composition Standards					
XXXX	Composition Standards for Compound Semiconductors	None Yet Available	2002			
	Wavelength	Calibration Standards	l			
2514	Wavelength Calibration Reference for 1560-1595 nm — Carbon Monoxide (¹² C ¹⁶ O)	Fiber-coupled molecular gas absorption cell with absorption lines in the WDM L-band between 1560 and 1595 nm	Spring 2002			
2515	Wavelength Calibration Reference for 1595-1630 nm — Carbon Monoxide (¹³ C ¹⁶ O)	Fiber-coupled molecular gas absorption cell with absorption lines in the WDM L-band between 1595 and 1630 nm	Spring 2002			
2517a	High Resolution Wavelength Calibration Reference for 1510-1540 nm — Acetylene (¹² C ₂ H ₂)	Fiber-coupled molecular gas absorption cell with narrow absorption lines between 1510 and 1540 nm	Available			
2519	Wavelength Reference Absorption Cell— Hydrogen Cyanide (H ¹³ C ¹⁴ N)	Fiber-coupled molecular gas absorption cell with absorption lines between 1530 and 1560 nm	Available			
	Polarization M	ode Dispersion Standards	l			
2518	Polarization Mode Dispersion Standard	Device with stable and known value of polarization mode dispersion which simulates optical fiber	Available			
2538	Deterministic Polarization Mode Dispersion Standard	To simulate PMD in discrete components — wavelengths 1250 to 1650 nm	Spring 2002			
Fiber and Fiber-Connector Geometry Standards						
2513	Mode Field Diameter Standard for Single-Mode Fiber	Optical fiber specimen with cleaved end and calibrated mode field diameter	Available			
2520	Optical Fiber Diameter Standard	Optical fiber specimen with cladding diameter values known to approximately ± 40 nm	Available			

continued next page

	The following fiber and fiber connector geometry SRM's are produced by NIST's			
	Precisio	n Engineering Division (821.00)		
2522	Pin Gauge Standard for Optical Fiber Ferrules	Wire used to size bores of connector ferrules; diameter known to approximately ± 40 nm	Available	
2523	Optical Fiber Ferrule Geometry Standard	Ceramic connector ferrule with specified outside diameter and roundness	Available	
2553	Optical Fiber Coating Diameter	Glass rod with index of refraction n =1.504 and diameter of approximately 250 μ m known within ±0.1 μ m.	Available	
2554	Optical Fiber Coating Diameter	Glass rod with index of refraction n =1.515 and diameter of approximately 250 μ m known within ±0.1 μ m.	Available	
2555	Optical Fiber Coating Diameter	Glass rod with index of refraction n =1.535 and diameter of approximately 250 μm known within ±0.1 μm.	Available	

Technical information on SRMs 2522, and 2523, 2553, 2554, and 2555 can be obtained from:

Precision Engineering Division (821.00)

National Institute of Standards and Technology 100 Bureau Drive, Stop 8210, Met A109 Gaithersburg, MD 20899

Phone: (301) 975-3463 Fax: (301) 869-0822

Discontinued Standards				
2524	Optical Fiber Chromatic Dispersion Standard	Approximately 10 km of optical fiber with zero-dispersion wavelength known to approximately ± 0.08 nm	Contact Division	
2525	Optical Retardance Standard	Nominally 90 degree retarder with retardance known to approximately \pm 0.1 $^{\circ}$	Contact Division	

For additional technical information contact:

Optoelectronics Division (815.00)

National Institute of Standards and Technology 325 Broadway Boulder, CO 80305

Phone: (303) 497-5342 Fax: (303) 497-7671 Email: optoelectronics@boulder.nist.gov

For detailed ordering and shipping information contact:

SRM Program Office

Building 202, Room 204 National Institute of Standards and Technology Gaithersburg, MD 20899-0001

Phone: (301) 975-6776 Fax: (301) 948-3730 email: <u>SRMINFO@nist.gov</u>