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ELECTRONICS AND ELECTRICAL ENGINEERING LABORATORY

OPTOELECTRONICS DIVISION

PROGRAMS, **A**CTIVITIES, AND **A**CCOMPLISHMENTS



THE ELECTRONICS AND ELECTRICAL ENGINEERING LABORATORY (EEEL)

One of NIST's seven measurement and standards laboratories, EEEL conducts research, provides measurement services, and helps set standards in support of the fundamental electronic technologies of semiconductors, magnetics, and superconductors; information and communications technologies, such as fiber optics, photonics, microwaves, electronic displays, electronics manufacturing supply chain collaboration; forensics and security measurement instrumentation; fundamental and practical physical standards and measurement services for electrical quantities; maintaining the quality and integrity of electrical power systems; and the development of nanoscale and microelectromechanical devices. EEEL provides support to law enforcement, corrections, and criminal justice agencies, including homeland security.

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 Microelectronic Programs

Office of Law Enforcement Standards

This publication 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, http://www.eeel.nist.gov. These publications are updated biennially.

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Cover caption: A band diagram depicting the operation of a single-photon turnstile is overlaid by a picture of an optical fiber cable, a representation of laser-based surgery, and a Poincaré sphere used to graphically describe optical polarization. All are examples of diverse optoelectronic applications served by the Division.

Image of laser surgery courtesy of Eyesearch.com.

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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



CONTENTS

From the Division Chief	iii
About the Optoelectronics Division	iv
Primary Standards for Laser Radiometry	v
Optoelectronics Division Staff	vi
CW Laser Radiometry	1
Pulsed-Laser Radiometry	4
High-Speed Measurements	7
Interferometry and Polarimetry	10
Spectral and Nonlinear Properties	14
Optical Materials Metrology	18
Nanostructure Fabrication and Metrology	22
Semiconductor Growth and Devices	25
Appendix A: Major Laboratory Facilities	29
Appendix B: NRC Post-Doctoral and Other Research Opportunities	30
Appendix C: Conferences and Workshops	32
Appendix D: Calibration Services	33
Appendix E: Standard Reference Materials	35

FROM THE DIVISION CHIEF

New and better-understood measurement techniques. Instrument calibrations and artifact calibration standards that are traceable to U.S. national standards. Assistance in the development of industry standards for product specification.

We are a group of about 60 scientists, engineers, and support staff, and these are some of the contributions we make to the optoelectronics industry. Herein, you will find examples of our accomplishments through the end of fiscal year 2002 (September 30, 2002) and an indication of our present work and plans. More current information is available on our web site. Please contact us if we can be of assistance.

We take metrology seriously. In optoelectronics, as in many other fields, it is a key part of the industrial infrastructure that establishes competitiveness. Consistently specified products are essential in fair trade, and measurements are a key element in efficient manufacturing. The cost of measurements often ranges between 10 % and 30 % of the cost of producing a product. So while the optoelectronics industry, along with many other high-technology fields, continues to struggle, advancing the field of optoelectronics metrology remains an important task.

We maintain the U.S. national standards for laser power and energy measurements (see page 34) and use them to provide the broadest range of laser calibration capabilities available anywhere. Each year we perform over 200 calibrations of power- or energy-measuring instruments for over 50 customers. We also provide artifact calibration standards, which we call Standard Reference Materials, for over a dozen parameters, most of them related to optical communications (see page 36 for details). These components provide customers with the capability of performing periodic instrument calibrations, traceable to national standards, in their own laboratories.

We develop measurement techniques that can be used, online and in situ, in the manufacture of optoelectronic materials, and provide reference data on the optical properties of important optoelectronic materials. And we work closely with major standards-developing organizations, especially the Telecommunications Industry Association (TIA), the International Electrotechnical Commission (IEC), and the International Organization for Standards (ISO), in producing industry standards.

A personal note: It has been my privilege to work in this area at NIST since 1969 and to lead the Optoelectronics Division since it was formed in 1994. I will retire in early January 2003, at about the time this document is released. Dr. Kent Rochford, who has substantial experience in optoelectronics metrology, both at NIST and in industry, has been appointed my successor. I know that, under his leadership, the Division will continue to play an important role in the industry.

Gordon W. Day, Division Chief

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Gordon W. Day and Kent Rochford

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 opto-electronics — 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



A SiC absorbing cavity for the 157 nm excimer laser calorimeter. Developed over the past few years, we now provide power and energy calibrations at this wavelength to support the semiconductor photolithography industry.



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



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 1,000 W and wavelengths from 0.4 μ m to 20 μ m. In current use for calibrations.



Three versions of this standard are used for pulsedlaser calibrations. The Q-Series, developed in the 1980s is used for 1.06 µm 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 STAFF

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Legend:

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 standards covering the wavelength range 450 nm to 1800 nm. These optical detectors have been optimized to provide highaccuracy 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, laser-based medical 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 also now have measurement capability based on a Laser Optimized Cryogenic Radiometer (LOCR), which provides improvement in accuracy by an order of magnitude for laser power measurements, compared to electrically-calibrated radiometers. 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 2004, reduce by at least a factor of 2 the uncertainties for all calibrations of laser energy and power supplied to customers. In some cases, this will be accomplished through the development of improved laser power transfer standards with greater range in optical power and wavelength.

The continuing 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 optical fiber 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 2003, reduce the uncertainties for optical-fiber power meter 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.

Technical Contact: John H. Lehman

Staff-Years (FY2003): 4.5 professionals 1.0 technicians



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 doubled Nd:YAG laser beam. The new laser system will provide many more wavelengths and higher power capabilities.

Many new systems being developed for the telecommunications industry demand higher power for optical-fiber power meter (OFPM) characterization and calibration. As much as several watts in fiber is being used in systems to pump laser sources for new applications. By developing the capability to provide calibrations for OFPM up to 10 watts, we can meet this need.

DELIVERABLE: By 2004, provide high power capability up to 10 W for optical fiber power meter calibrations.

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 2005, 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.

ACCOMPLISHMENTS

• Completed refurbishment of the calibration facilities for CW laser power and energy calibrations. This included the low power CW, high-accuracy LOCR, optical fiber power, and laser source laboratories.



Shown is the newly refurbished laser laboratory, which supplies laser radiation for CW laser power and optical fiber power meter calibrations. The refurbishment included completely refinishing the room, all new facilities (temperature, lighting, electrical supply, and cooling water), and new laser tables.

• Completed the documentation of the Division's Laser Optimized Cryogenic Radiometer, and have obtained approval to begin a calibration service based on the LOCR. This radiometer serves as the basis for traceability of optical power measurements for several calibration services offered by the Division, including CW laser power and optical fiber power measurements.

• Modeled the errors associated with optical fiber transfer standards using angular uniformity data obtained with the angular response characterization system. The newest designs for optical fiber transfer standards show an error of less than 0.1 % using fiber up to NA of 0.26. When completely implemented into our calibration systems, the new transfer standard will be capable of measurements with a total expanded uncertainty of 0.4 %.

• Delivered both Si and Ge versions of the latest optical fiber transfer standards to Navy and Army sponsors. These detectors will improve the accuracy of their optical fiber power calibration and reduce inter-service variation.

• Built and tested a tunable Ti:Sapphire laser with a tuning range of 700 nm to 900 nm. Also installed a dye laser to extend this range to 550 nm. This enables the calibration of detectors over a wide range of visible wavelengths.



Shown is a new tunable dye laser that will expand the capabilities of the Optoelectronics Division's calibration services in laser power and optical fiber power. The new laser system will provide additional wavelengths for laser power meter calibrations.

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 for a list of calibrations.

EQUIPMENT SUPPLIED TO OTHER AGENCIES AND NATIONAL LABORATORIES

• Delivered both Si and Ge versions of the latest optical fiber transfer standard to Navy and Army sponsors. These detectors will improve the accuracy of their optical fiber power calibration and reduce inter-service variation.

• Various transfer standards for laser and optical-fiber power delivered to DOD sponsors.

STANDARDS COMMITTEE PARTICIPATION

American National Standards Institute: John H. Lehman is a member of committee Z136, subcommittees SSC-01 and SSC-04, which deal with safe use and measurement of lasers.

Telecommunications Industry Association: Igor Vayshenker is a member of subcommittee FO-4.9, which deals with Metrology and Calibration.

RECENT PUBLICATIONS

Lehman, J.H.; Radojevic, A.M.; Osgood Jr., R.M., "Domain-Engineered Thin-Film LiNbO₃ Pyroelectric-Bicell Optical Detector," IEEE Phot. Tech. Lett. **13**: 851-853 (Aug 2001).

Houston, J.M.; Livigni, D.J, "Comparison of Two Cryogenic Radiometers at NIST," NIST JRES **106**: 641-647 (Jul-Aug 2001).

PULSED-LASER RADIOMETRY

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 inkjet 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 deep-ultraviolet (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 International 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 (248 nm) and ArF (193 nm), and more recently F_2 (157 nm) excimer lasers, are the preferred sources for high-resolution lithography at this time.

We have recently completed the construction of a



SiC absorbing cavity for the 157 nm excimer laser calorimeter. The cavity is mounted on the thermal reference flange. A temperature-stabilized reference jacket (not shown) is mounted on the thermal reference flange. This jacket maintains a constant temperature reference. The injected energy is a function of the temperature difference between the cavity and the jacket.

system to perform absolute calibrations of 157 nm excimer laser detectors. The primary customers for this new calibration service are semiconductor manufacturers and their suppliers, e.g., detector, laser, and stepper manufacturers. This service, like other NIST excimer laser calibration services, uses an electrical-substitution calorimeter as a primary standard to provide direct traceability to SI units for laser power and energy measurements. Calorimeters, while having the advantage of providing the highest level of accuracy for these measurements, have a slow response time. Therefore, because calorimeter-based calibrations are both timeconsuming and expensive, typically only a few calibration points are measured. However, most laser detectors are typically used over a range larger than that covered by a single calibration point. As a result, additional measurement uncertainty will be introduced if the detector response is not linear and the nonlinearity is not quantified. Range discontinuity, e.g., change in response due to a nonlinear change in detector amplification, can also introduce additional measurement uncertainty.

Technical Contact: Christopher Cromer

Staff-Years (FY2003): 5.0 professionals

Measurements of detector nonlinearity and range discontinuity will give complete information of the detector's response over the entire measurement range, with the added benefit that the measurement technique is much faster, and therefore cost is much less compared to adding more calibration points.

DELIVERABLE: By 2003, develop the capability to perform measurements of detector nonlinearity at 248 and 157 nm (currently available at 193 nm).

In addition to existing DUV laser measurement services, there is increasing demand for measurements of laser dose (energy density), where the detector samples a fraction of the total laser beam. Accurate measurements of laser dose 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.



Schematic diagram of the 157 nm excimer laser energy/power meter calibration system. The system consists of three nitrogen-purged stainless steel enclosures that minimize cross-contamination between the sub-systems. During a calibration, the customer's meter is substituted for either of the two standard calorimeters, labeled "C" in the figure.

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

DELIVERABLE: By 2005, extend the wavelength range of the dose meter service beyond our current 193 nm service to include the capability to perform measurements with 248 and 157 nm excimer lasers.

Recently there has been increasing demand to extend the wavelength range of laser target ranging and designation systems to the eye-safe laser regime. These laser systems provide positional information, such as distance and incident angle, for use in locating targets. There is a need to extend existing pulsed Nd:YAG laser power and energy calibration services to include measurements at $1.55 \mu m$ and to reduce the overall uncertainty associated with these measurements.

DELIVERABLE: By 2004, develop capabilities for measurements of high pulse energy at 1.55 μ m. Design, build, and characterize an improved calibration service for measurements of pulsed Nd:YAG laser power and energy.



Homogenized 193 nm excimer beam profile used for dose calibrations, measured with a pyroelectric array camera. The beam uniformity of the laser beam is 1.3 % (determined using ISO 13694 procedures).

ACCOMPLISHMENTS

• Designed, constructed, and tested a system for characterizing detector nonlinearities at 193 nm. This versatile system can be used to characterize a wide range of UV detectors and has generated interest among detector manufacturers and International SEMATECH. This service may be an inexpensive alternative to customers who are more concerned with the relative rather than the absolute response of their detectors. The primary reason for its lower cost is the reduced turnaround time; a linearity measurement takes approximately one-third the time of a full calibration.

• Characterized the nonlinearity of several commercial 193 nm detectors. Evaluated the performance of several commercial detectors using the nonlinearity system.

• Completed construction of two 157 nm calorimeters. We have also completed a series of electrical calibration measurements on both calorimeters, in air as well as nitrogen-purged environment. From these electrical calibrations, we deduce that the sensitivity of the 157 nm calorimeter is improved by factor of five over that for the NIST 193 nm calorimeters. We also performed an intercomparison of the 157 nm calorimeter and with the NIST 248 nm calorimeters. The optical performance of the working calorimeter, as demonstrated by this intercomparison, exceeds initial design specifications.

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.

STANDARDS COMMITTEE PARTICIPATION

International Standards Organization: Marla L. Dowell is a member of working group TC 172/SC 9/WC 1, which deals with terminology and test methods for lasers.

American National Standards Institute: John H. Lehman is a member of committee Z136, subcommittees SSC-01 and SSC-04, which deal with safe use and measurement of lasers.

U.S. National Committee/International Electrotechnical Commission: John H. Lehman is a member of technical committee TC76, working groups 1 and 3, which deal with laser radiation measurement and safety.

RECENT PUBLICATIONS

Dowell, M.L.; Jones, R.D.; Laabs, H.; Cromer, C.L.; Morton, R., "New Developments in Excimer Laser Metrology at 157 nm," *Proc., SPIE Microlithography Conference*, March 3-8, 2002, Santa Clara, CA, 63-69 (2002).

Dowell, M.L., "Progress Report: 157 nm Excimer Laser Calorimeter Development," *SEMATECH Tech. Report*: 1-13 (Jul 15, 2002).

Chen, D.H.; Dowell, M.L.; Cromer, C.L.; Zhang, Z.M., "Thermal Response and Inequivalence of Pulsed Ultraviolet-Laser Calorimeters," *J. Thermophysics and Heat Transfer* 16(1): 36-42 (Jan 2002).

Laabs, H.; Jones, R.D.; Cromer, C.L.; Dowell, M.L.; Liberman, V., "Damage Testing of Partial Reflectors at 157 and 193 nm," *Proc. Optical Materials for High Power Lasers Symp.*, Oct 1-3, 2001, Boulder, CO (2001).

Dowell, M.L.; Cromer, C.L.; Jones, R.D.; Keenan, D.A.; Scott, T.R., "New Developments in Deep Ultraviolet Laser Metrology for Photolithography," *AIP Conf. Proc*, 2001, Vol. 550, 361-363 (2001). Dowell, M.L., "Pulsed-laser Metrology at NIST," *Optics and Photonics News* 28 (February 2001).

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

Dowell, M.L.; Cromer, C.L.; Jones, R.D.; Keenan, D.A.; Scott, T., "New Developments in Deep Ultraviolet Laser Metrology for Photolithography," *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, pp. 391-394 (2001).

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.



The electro-optic measurement system, shown here being operated by Tracy Clement, measures an electrical pulse on a LiTaO₃ wafer. De-embedding techniques are then used to determine the voltage generated by the photodiode being tested at its coaxial output connector. © Geoffrey Wheeler

CUSTOMER NEEDS

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 gigabits per second and higher 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 low 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 the noise figure of 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, consisting of a photodiode combined with a micro-wave 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, 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. The vector frequency response of a photoreceiver is the Fourier transform of its impulse response. Vector response is required for design of high-speed optoelectronic systems, but at present there are no accepted standard methods for this measurement.

Researchers in the High-Speed Measurements Project have demonstrated time-domain techniques for measuring optoelectronic vector response with verifiable accuracy up to 110 GHz using electrooptic sampling. By developing these measurements our project is pioneering a new paradigm for timedomain measurements with frequency-domain calibrations.

DELIVERABLE: We intend to have a Calibration Service for frequency response phase that uses these new calibration strategies available in 2003. By 2005 we will develop methods for extending frequency response calibrations beyond 110 GHz.

Optical communications analyzers or reference receivers used for measuring digital eye-patterns on optical signals have many similarities to electrical oscilloscopes, but also have advantages of their own. In particular, they can be calibrated over a very high bandwidth because they do not require band-limited microwave calibrations. Use of this Technical Contact: Paul D. Hale

Staff-Years (FY2003): 4.0 professionals 0.5 technician 1.0 contractor calibration on typical measurements, however, possesses some unique problems. For example, we must develop methods for removing time-base distortions and deconvolving the oscilloscope response. In collaboration with Divisions in EEEL and ITL, we are currently applying our expertise in receiver measurements to these problems.

DELIVERABLE: During 2003 NIST will work with the IEC to document procedures developed at NIST for calibrating the frequency response of optical waveform measurement equipment.

DELIVERABLE: We will develop methods that improve the accuracy of eye-diagram and other time-domain measurements by calibrating and removing instrument errors in optical oscilloscopes in 2004.

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.

DELIVERABLE: In 2003 we will document a method for applying RIN standards to calibrating optical amplifier noise figure measurements.

ACCOMPLISHMENTS

Developed an electro-optic sampling system to sample high-speed electrical waveforms on a coplanar waveguide with ultrashort laser pulses via the electro-optic effect. Standard microwave techniques are used to calibrate the response of a photoreceiver at its 1 mm electrical port, which is physically removed from the sampling plane. The frequency range of the calibration is limited only by the 1 mm coaxial connectors. The characterized photoreceiver has a bandwidth of about 80 GHz; the signal-to-noise ratio of the measurement is greater than 150:1 at 110 GHz.

• Preliminary measurements of a commercially available photodiode demonstrated an (uncorrected) impulse response FWHM of 5.4 ps, a response spectrum that has rolled off by only 10 to 13 dB at 110 GHz, and a phase response flat to within \pm 10 degrees up to 110 GHz. The photodiode also displayed response linear in power with outputs as high as 0.1 V into 50 Ohms, making it suitable for comparison with heterodyne measurements.



Electro-optic sampling is capable of determining the vector frequency response of a photodiode, that is, it can determine both the magnitude and phase response of the photodiode.

• Accurately characterized the modulation response magnitude and phase of a commercially available photoreceiver to 110 GHz, nearly three times the bit rate of 40 Gb/s optoelectronic systems. High-speed photo-receivers are commonly used in fiber optic communications systems and test equipment. Measurement of the modulation response of the photoreceiver over a frequency range much larger than its bandwidth is necessary for accurately modeling its response in the time domain, providing critical information for digital communications systems.

STANDARDS COMMITTEE Participation

Telecommunication Industry Association: Gregory Obarski is a member of subcommittee FO-4.5, which deals with optically amplified devices, sub-systems, and systems. Gregory Obarski and Paul Hale are members of subcommittee FO-4.9, which deals with fiber optic test and measurement instrumentation and related calibration issues. International Electrotechnical Commission: Paul Hale is a member of technical committee TC86, working group 4, which deals with Fiber Optic Test Equipment Calibration. Gregory Obarski is a member of subcommittee 86C, working group 3, 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, lowlevel pulse power and energy, and relative intensity noise. For more information see Appendix D or contact Paul Hale at (303) 497-5367.

RECENT PUBLICATIONS

Clement, T.S.; Hale, P.D.; Williams, P.A., "Tutorial: Fiber and Component Metrology for High-Speed Communication," Proc., Optical Fiber Communication Conf., Mar 17-22, 2002, Anaheim, CA, WZ1 (March 2002).

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Clement, T.S.; Diddams, S.A.; Jones, D.J., "Lasers, Ultrafast Pulse Technology," Encycl. Phys. Sci. Tech. Third Edition, Vol. 8, 499-510 (Jan 2001).

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

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

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

INTERFEROMETRY AND POLARIMETRY

Technical Contact: Paul A. Williams

Staff-Years (FY2003): 5.75 professionals 0.4 technicians 1.0 student

PROJECT GOALS

This project uses interferometric and polarimetric techniques to provide calibration measurements, standards, and expertise in support of the optoelectronics industry. Our current program primarily supports the optical fiber communications industry, but also has applications to optical fiber sensors.



Graphical technique demonstrating the effects of various amounts of polarization-dependent loss on the transmitted polarization state.

CUSTOMER NEEDS

The 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 polarization- and wavelengthdependent propagation delay, polarizationdependent loss and polarization-dependent 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, polarization-mode dispersion, polarizationdependent loss, and polarization-dependent spectral 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 wavelength). This variation in propagation velocity results in broadening of the optical pulses used in communication systems. Broadened pulses interfere with each other and are difficult to distinguish, causing communication errors. System chromatic dispersion must be dealt with by managing its spectral profile through dispersion-tailored fibers, choice of zerodispersion wavelength, and dispersioncompensating fibers. This requires accurate measurement of the wavelength dependence of chromatic dispersion. Similarly, wavelength-dependent relative group delay in components degrades system performance. Often, RGD must be measured in components that have sub-nanometer 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 two 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 polarization mode dispersion (PMD) metrology where propagation velocity depends on the polarization state of the light. Communication systems require PMD measurement with a few tens of femtoseconds of resolution with a spectral resolution of a few tens of picometers. In addition, PMD is a statistical quantity, making stability of a reference artifact difficult.

Non-temporal polarization effects such as polarization-dependent loss (PDL) and polarizationdependent wavelength shift (PDW) of optical filters cause variations in intensity 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. The interaction of PMD and PDL in a communication system can lead to complex and unexpected signal degradation. This interaction needs to be characterized so that system behavior can be predicted from known fiber and component properties.

Fiber Bragg gratings (FBGs) have applications in both optical fiber communications and optical fiber sensor systems. Strain sensor networks consisting of many FBGs can provide information on the integrity of structures such as buildings and bridges. To reliably extract strain information, sensor stability and demodulator accuracy must be assessed.

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 calibration artifact distribution, measurement comparison, and special tests.

Chromatic Dispersion: Our chromatic dispersion measurement capabilities enable us to perform Special Test calibrations of customer fibers. We are working to improve the accuracy of these measurements.

DELIVERABLE: In 2003, study and publish characterization of amplified spontaneous emission effects on measured chromatic dispersion.

Relative Group Delay (RGD): Our RGD measurement work is directed toward assessing the achievable temporal resolution for our 200 MHz RF phase shift system, and establishing fundamental standards with theoretically predictable RGD profiles.

DELIVERABLE: Complete uncertainty characterization of RF phase shift and low-coherence interferometry RGD systems in 2003.

Polarization-Mode Dispersion (PMD): Our work is directed at absolute calibration of RF phase shift measurements of PMD in narrow (5 GHz) bandwidths and characterization of measurement pitfalls in measurement of second-order PMD. We will also consider possible artifacts for stable emulation of second-order PMD.

DELIVERABLES: In 2003, complete evaluation and documentation of Modulation Phase Shift (MPS) method for narrowband PMD measurement. In 2003 and 2004, assess calibration needs for second-order PMD.

Polarization-Dependent Loss (PDL): We are validating a new PDL measurement technique based on 6 launched polarization states (rather than 4). This is a promising method for reducing the PDL measurement uncertainty due to system birefringence. We are also studying the interaction between PDL and PMD and its effect on measurements.

DELIVERABLE: In 2003, complete evaluation of the 6-state PDL measurement technique and its experimental comparisons with the 4-state technique.

Component Spectral Transmission/Reflection: As we co-develop techniques for spectral reflection characterization of fiber Bragg gratings, we will be comparing the results for different techniques and evaluating the accuracy of each method.

DELIVERABLES: In 2003, complete uncertainty characterization of tunable laser and low-coherence interferometric spectral characterization techniques.



Fiber Bragg grating in temperature-controlled chamber for spectral reflection measurement.

Optical Fiber Bragg Grating Characterization: We are beginning a project to use low-coherence interferometry for real-time monitoring of longitudinal index variations in fiber Bragg gratings during the grating writing process.

DELIVERABLES: In 2003, develop low-coherence interferometric system capable of measuring spectral characteristics of a remotely located fiber Bragg grating; in 2003 and 2004, use this system to conduct studies during fiber Bragg grating inscription.

Accomplishments

New SRM for Polarization-Mode Dispersion (PMD) — We have completed the development and delivered certified units of a new Standard Reference Material for non-mode-coupled polarization mode dispersion (SRM 2538). This is the second SRM for PMD. The first, SRM 2518, emulates the differential group delay in a long length of optical fiber. The new artifact, SRM 2538, is a fiber-pigtailed quartz plate, designed to emulate the PMD typically found in telecommunications components. This new SRM is certified for mean Differential Group Delay (DGD) over a wavelength range of approximately 1275–1625 nm for measurements by all PMD techniques.

 New Calibration Reference for Polarization-Dependent Loss — The first NIST calibration reference for Polarization Dependent Loss is now available to the public as a part of the Measurement Assurance Program (MAP). The MAP artifact consists of a short section of polarizing fiber spliced to a single-mode input fiber and a stepindex multimode output, and its PDL is certified over the wavelength range of 1535–1560 nm. The artifact is implemented as a MAP (rather than an SRM) to mitigate the degradation to uncertainty that is caused by exposure of the artifact to unpredictable temperatures. The MAP implementation includes a recording thermometer to indicate the temperature exposure of the device during the customer measurement process.

Relative Group Delay Measurements Verified — Along with a commercial collaborator, we have independently measured the relative group delay of a line in the same hydrogen cyanide gas absorption cell using low- coherence interferometry, RF phase shift, and swept-wavelength optical interferometry, respectively. The agreement among all measurements and our theoretical prediction is within 0.3 ps when measured with a 6 pm wavelength resolution. This serves as a good test of the accuracy of these measurement techniques and may lead to a relative group delay transfer standard based on hydrogen cyanide.

• Low-Coherence Measurement System Modeled — We have modeled and experimentally verified the effect of signal-to-noise ratio on lowcoherence interferometric measurements of relative group delay (RGD) in optical components. The model describes the often misunderstood tradeoff between temporal and spectral resolution. We also improved the wavelength resolution of the lowcoherence interferometer by installing a Nd:YAG reference laser that provides a measured wavelength stability of 1 pm.

• Improved PDL Measurement Technique Developed — We have demonstrated an improved "6-states" technique for measuring polarization dependent loss (PDL), enabling the measurement of PDL in the presence of birefringence. This new method also reduces the measurement uncertainty by a factor of 2 over the current "4-states" method. • PMD Emulator Developed — We have assembled and tested a polarization-mode dispersion (PMD) emulator capable of generating arbitrary values of non-mode-coupled PMD with an uncertainty of 1.5 % (limited by the measurement system).

• Polarization-Dependent Wavelength Shift Measurement Technique — In collaboration with the Spectral and Nonlinear Properties Project, we have developed and demonstrated a fast and accurate technique to measure the polarizationdependent shift of the center wavelength of fiber Bragg gratings.

STANDARD REFERENCE MATERIALS (SRM) AND MEASUREMENT ASSURANCE PROGRAM (MAP)

(See Appendix D)

http://www.boulder.nist.gov/div815/NEWsrms.htm

SRM 2518, Polarization-Mode Dispersion (Mode-Coupled); available

SRM 2538, Polarization-Mode Dispersion (Non-Mode-Coupled); available

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 availability of artifact under the MAP program.

Polarization Dependent Loss MAP, Simulates wavelength-dependent PDL in optical components and fiber. Certified for optical sources between 1535– 1560 nm, with singlemode input and multimode output fiber. Available for loan.

STANDARDS COMMITTEE PARTICIPATION

Telecommunication Industry Association: Rex M. Craig, Timothy J. Drapela, and Paul A. Williams are members of committee FO-4, which deals with standards issues relating to optical fibers, optical fiber components, and optical fiber systems.

International Electrotechnical Commission: Timothy J. Drapela is a member of technical committee TC86, which deals with active and passive fiber optic metrology.

RECENT PUBLICATIONS

http://www.boulder.nist.gov/div815/IPP.htm

Dennis, T.; Williams, P.A., "Relative Group Delay Measurements with 0.3 ps Resolution: Toward 40 Gbit/s Component Metrology," *Proc., Optical Fiber Communication Conf. (OFC'02)*, paper WK3, pp. 254-256 (March 2002).

Dyer, S.D.; Espejo, R.J.; Williams, P.A., "High-Resolution Group Delay Measurements of Hydrogen Cyanide Gas Cell Using Low-Coherence Interferometry," *Tech. Digest, Symposium on Optical Fiber Measurements (SOFM 2002)*, NIST Special Publication 988, pp. 45-48 (September 2002).

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Swann, W.C.; Dyer, S.D.; Craig, R.M., "Four-state measurement method for polarization dependent wavelength shift," in *Tech. Digest, Symposium on Optical Fiber Measurements (SOFM 2002)*, NIST Special Publication 988, pp. 125-128 (2002).

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Rochford, K.B., "Polarization and Polarimetry," in the Encycl. of Phys. Sci. Tech., Third Edition, Vol. 12, 521-538 (January 2002).

SPECTRAL AND NONLINEAR PROPERTIES

Technical Contact: Sarah L. Gilbert

Staff-Years (FY 2003): 4.25 professionals 0.4 technicians 2 post-doctorals

GOALS

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



Kristan Corwin studying supercontinuum generation in microstructure fiber. © Geoffrey Wheeler

CUSTOMER NEEDS

This project concentrates on the needs of the optical fiber communications industry; we focus primarily on metrology for the characterization of optical fiber and components used in communication systems. High-bandwidth optical fiber communication systems require accurate measurement of spectral, polarization, dispersion, 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 or 100 GHz channel spacing (0.4 or 0.8 nm, respectively) in the 1540 to 1560 nm region, but narrower channel spacing may be used in the future. It appears likely that systems will be implemented in other wavelength regions as well, particularly in the 1565–1625 nm WDM L-band. 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, self-phase 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 (SRM) 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 four wavelength reference Standard Reference Materials (SRMs) based on fundamental molecular absorption lines: SRM 2514 (carbon monoxide 12C16O), SRM 2515 (carbon monoxide ¹³C¹⁶O), SRM 2517a (acetylene, high resolution) and SRM 2519 (hydrogen cyanide). Together these SRMs can be used to calibrate the wavelength scale of instruments between 1510 and 1630 nm.

WDM may expand into other wavelength regions, such as the 1300 to 1500 nm region. We have developed a hybrid multiple-wavelength reference that incorporates the wavelength flexibility of artifact references and the stability of fundamental molecular absorption references. Customized multiple wavelength reflectors can be generated by writing multiple superimposed fiber Bragg gratings (FBG) into optical fiber. Each grating is a reflector for a specific wavelength of light; the wavelengths are selected during the grating fabrication process. Strain and temperature changes can cause the center wavelengths of these reflectors to change. On the other hand, atomic and molecular absorption lines are very stable under changing environmental conditions. If one of the FBG reflectors is located near an atomic or molecular absorption line, it can be actively stabilized to that line. This stability is then transferred to the other gratings, because they are superimposed at the same location in the fiber. We have demonstrated a hybrid reference based on this principle that provides multiple calibration references in the 1300 and 1550 nm regions, which are stable to better than 1 pm.

DELIVERABLE: Transfer hybrid wavelength reference technology to industry in 2003 and 2004.

DELIVERABLE: Develop high-resolution version of SRM 2519 (hydrogen cyanide) by 2004.



Measured spectra of two sampled fiber Bragg gratings interleaved on a single fiber for use in a hybrid wavelength calibration reference.

High-accuracy wavelength standards: To calibrate its equipment, NIST needs higher accuracy internal wavelength references. 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. To generate an optical frequency comb in the near infrared, pulses from a Cr:Forsterite femtosecond laser are launched into highly nonlinear optical fiber, which broadens the laser spectrum to a supercontinuum with a width of > 1000 nm. Since the laser is pulsed, this broad supercontinuum will actually be composed of discrete frequency lines – a frequency comb – spaced by the laser repetition rate. This optical frequency comb can be stabilized and used to measure references throughout the 1280 to 1630 nm region.

We have developed two Cr:Forsterite femtosecond lasers for optical frequency comb generation, one with a 100 MHz repetition rate and one with a 400 MHz–1 GHz repetition rate. The output spectrum of the 400 MHz laser is shown below. This spectrum can support a pulse width of ~30 fs; the average output power of the laser is 480 mW and it can operate stably for many hours.

DELIVERABLES: Demonstrate accurate frequency comb in the WDM telecommunications region and establish a series of high-accuracy references for NIST internal calibration in 2003 and 2004.



Output spectrum from the 400 MHz Cr: Forsterite laser and the resulting supercontinuum spectrum generated when this laser is transmitted through 6 meters of highly nonlinear fiber.

Nonlinear Properties: We are concentrating on two areas: Raman amplification and supercontinuum generation in optical fiber. In Raman amplification, a strong pump beam amplifies a weaker signal beam through stimulated Raman scattering. The Raman gain depends strongly on the pumpsignal wavelength difference and weakly on the absolute pump wavelength. There are well-known techniques for measuring the strong dependence on the pump-signal wavelength difference, but not the weaker dependence on the absolute pump wavelength. In 2002, using the asymmetry of the Raman gain curve, we developed a simple technique for determining this weak, but potentially important, pump-wavelength dependence. **DELIVERABLES:** Fully document new technique for measuring the pump-wavelength dependence of the Raman gain; participate in round robin on Raman gain measurements in 2003.

A striking example of the nonlinear effects in optical fiber is provided by supercontinuum generation in highly nonlinear fiber. Supercontinua can be generated in the near infrared using a Cr:Forsterite laser, or in the visible using a Ti:Sapphire laser. The supercontinua have a number of possible applications in telecommunications, including as a wavelength reference or as a WDM source after spectral slicing. In addition, these supercontinua will find uses in optical coherence tomography and spectroscopy. For many applications, the amplitude noise on the supercontinuum can be a limiting factor. Unfortunately, the same nonlinear processes that give rise to the supercontinuum also amplify any input noise. In 2002, we conducted a systematic study of this amplitude noise for a range of input parameters and have identified both a lowfrequency noise component that arises from the technical noise on the laser, and a broadband frequency noise component that arises from the initial shot noise on the input laser and spontaneous Raman scattering.

DELIVERABLE: Conduct additional experimental and theoretical studies of noise processes in frequency comb and supercontinuum generation in microstructure fiber in 2003.

ACCOMPLISHMENTS

WDM L-Band Wavelength Calibration Standard Reference Materials - Two wavelength calibration transfer standards for the new L-band of WDM optical fiber communications have been developed. These standards, based on the absorption spectrum of carbon monoxide, are now available as SRMs 2514 and 2515. SRM 2514 can be used to calibrate the wavelength scale of measurement equipment in the 1560 to 1595 nm region. The unit is a single-mode optical-fiber-coupled absorption cell containing carbon monoxide ¹²C¹⁶O gas, which has many absorption lines in this region. SRM 2515 is nearly identical to SRM 2514, except that it contains the ¹³C¹⁶O isotopic species of carbon monoxide. This isotopic species has numerous absorption lines at longer wavelengths, ranging from 1595 to 1630 nm. Forty-one line center wavelengths of each SRM are certified with uncertainties ranging from 0.4 to 0.7 pm.

• NIST Traceable Reference Material (NTRM) Program Established — In response to the large demand for our wavelength calibration SRMs, we have established an NTRM alternative: *The NIST Traceable Reference Material Program for Wavelength Reference Absorption Cells*. An NTRM is a commercially produced reference material with a well-defined traceability linkage to existing NIST standards. Although the NTRM gas absorption cells are produced and distributed by commercial vendors, the pressure and wavelength value assignments, and respective uncertainties, are made by NIST.

Polarization Dependent Wavelength Shift (PDW) Measurement Technique Developed — In collaboration with the Interferometry and Polarimetry Project, we have developed and demonstrated a fast and accurate technique to measure the polarization-dependent wavelength shift of the center wavelength of fiber Bragg gratings. This new technique uses only four polarization states to derive the PDW. It is a rapid alternative compared to the conventional measurement of the fiber Bragg grating spectrum for many (30 or more) different polarization states to approximate coverage of all polarization states.

Hybrid Wavelength Calibration Reference De-veloped — We have completed our investigation of interleaved, sampled fiber Bragg gratings (FBG) for the 1300 and 1550 nm regions, which produce multiple reflection peaks in each region. We have evaluated the polarization-dependent wavelength shift of sampled gratings and found that it is very small (1-2 pm). We have also evaluated the stability of these gratings in the actively stabilized hybrid reference configuration. With one of the 1550 nm FBG peaks stabilized to a hydrogen cyanide absorption line, we strained the fiber and measured the center wavelength of a FBG peak in the 1300 nm region. The active stabilization of the 1550 nm grating forced the 1300 nm peak to remain stable to about 0.3 pm. If the gratings were not stabilized, the 1300 nm peak would have shifted by about 200 pm under these strain conditions.

• Near-Infrared Frequency Comb — In collaboration with the NIST Time and Frequency Division, a broad frequency comb (>1000 nm wide) in the near infrared region was produced using a Cr:Forsterite laser and nonlinear optical fiber. Once stabilized, this comb will be used to measure highaccuracy wavelength references for the WDM region.

• Supercontinuum Noise Study — We have completed an extensive study of the noise in a supercontinuum produced by pulsed light in microstructure fiber. Amplitude noise has been measured as a function of wavelength, pulse duration, pulse chirp, and Fourier frequency, and collaboration with a university researcher has produced numerical simulations. The combination of these data and simulations is the first definitive work on the fundamental noise processes that occur in supercontinuum generation using microstructure fiber. This work clearly shows how to minimize the noise, which can degrade the frequency comb produced in the fiber and limit its usefulness for accurate metrology.

Measurement of Raman Gain — Measurements of the pump-wavelength dependence of Raman gain using two techniques have been completed. The first technique involves measuring the integrated gain. The second, more elegant, technique uses the asymmetry of the gain curve for the Stokes and anti-Stokes regions. This new technique (developed at NIST) has the advantage that it is independent of the pump and signal powers and therefore has lower uncertainty. The dependence of Raman gain on pump wavelength is important for understanding the details of Raman amplifier performance, particularly gain tilt.

STANDARD REFERENCE MATERIALS (SRM)

See Appendix E

http://www.boulder.nist.gov/div815/NEWsrms.htm

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

SRM 2514, Wavelength Calibration Reference for 1560–1595 nm – Carbon Monoxide ¹²C¹⁶O; available.

SRM 2515, Wavelength Calibration Reference for 1595–1630 nm – Carbon Monoxide ¹³C¹⁶O; available.

SRM 2517a, High Resolution Wavelength Calibration Reference for 1510-1540 nm – Acetylene ${}^{12}C_2H_2$; available.

SRM 2519, Wavelength Calibration Reference for 1530–1560 nm – 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.

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OPTICAL MATERIALS METROLOGY

PROJECT GOALS

Develop complementary metrology methods of nonlinear optical analysis, near-field optical spectroscopy, 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. Develop prototype ultrafast and narrow-line cw solidstate waveguide lasers.



Ordinary refractive index of $Al_xGa_{l-x}N$ 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; and (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 re-

cent 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 transistors and solar-blind UV detectors. 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 nm 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 GaNbased 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), including secondharmonic generation (SHG) and two-photon luminescence (2PL) spectroscopy, 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 has relied on correlating the SHG results with X-ray diffraction imaging performed at Brookhaven National Laboratory in collaboration with NIST Materials Science and Engineering Laboratory (MSEL). SHG is extremely sensitive to such undesirable

Technical Contact: Norman A. Sanford

Staff-Years (FY2003): 2.7 professionals 2.0 post-doctorals

structural features as stacking faults, domain reversals, mixed cubic and hexagonal phases, and subsurface polishing-induced damage. X-ray diffraction imaging reveals full-crystal images of stacking faults and domain reversals, but is less sensitive to the presence of mixed phases. Thus, a combination of SHG analysis and X-ray imaging methods results in a reduction in ambiguity compared to the case where only one method is employed. Depth-dependent artifacts are also problematic in GaN. Polishing-induced structural damage is revealed with cathodoluminescence (CL) studies. Further, SHG and CL analysis will examine the degree to which the damage can be removed using chemically assisted ion-beam etching.

2PL spectroscopy offers a means by which the uniformity of an active quantum well layer in a IIInitride laser or LED structure can be probed regardless of the thickness of the surrounding layers. Standard direct above-bandgap photoluminescence (PL) spectroscopy is problematic since the pump beam could be absorbed elsewhere in the structure before exciting the regions of interest. We will correlate 2PL measurements with CL results, obtained when the capping layer is thin enough (~20 nm) to allow CL examination of the active region through the capping layer, with those of a more realistic device structure where the active layer is buried hundreds of nanometers within the structure.

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 SHG and photoluminescence (PL) spectroscopies on a resolution scale of 20 to 100 nm.

DELIVERABLES: In 2003 and 2004, working closely with collaborators in the NIST MSEL and Chemical Science and Technology Laboratory (CSTL), we will correlate near-field optical measurements and 2PL studies with CL studies and other high-resolution electron beam and X-ray metrologies. We will establish the inclusion of time-resolved spectroscopy into our suite of correlated measurements by 2004. In 2004 and 2005 we will apply nanoscale spatially resolved spectroscopy to the study of low In-content material.

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. We have 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 is currently from 442 nm to 1064 nm. Extension of the measurements into the ultraviolet region requires grating coupling and/or nonlinear optical techniques. Calibration of composition of epilayers using techniques such as EDS is critical. Using x-ray diffraction, we are also measuring the bowing of lattice constants as a function of composition .

DELIVERABLES: In 2003 we will add to the refractive index database for AlGaN in the ultraviolet and deep blue regions. In 2004-2005 we will establish methods to measure the refractive index of layers in active GaN devices.

We are developing cw and mode-locked waveguide lasers using ion-exchange in rare-earth-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 solidstate sources benefit from a long upper-state, laser-transition lifetime (~10 ms) that allows low-noise production of mode-locked pulses independent of repetition rate and low-noise generation of narrowlinewidth light. Unlike fiber laser sources, waveguide sources have sufficient rare-earth doping concentrations ($\sim 10^{20}$ 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 (SESAM). We will improve SESAM performance by reducing the mirror's nonsaturable loss and will extend the frequency of our phase noise measurement capability. We will also lock the output of the mode-locked laser to an optical frequency standard to measure absolute timing jitter. Narrowlinewidth lasers have been fabricated using a distributed Bragg reflector (DBR) etched directly onto the waveguide structure.

DELIVERABLE: In 2003, working with the NIST Physics Laboratory, we will measure the absolute timing jitter of mode-locked waveguide lasers.

ACCOMPLISHMENTS

• Correlated the wavelength dependence of the refractive index and birefringence of $Al_xGa_{1-x}N$ with Al fraction using energy dispersive X-ray spectroscopy (EDS). The calibration of the EDS permits a resolution of $\pm 2\%$ in the Al mole fraction. Prism coupling methods have been extended to include the wavelengths 442 nm, 458 nm, 488 nm, 514 nm, 532nm, 632 nm, 690 nm, 750 nm, 850 nm, and 1064 nm. Extraordinary and ordinary refractive indices were measured in the c-plane oriented Al_vGa_{1.v}N films grown on sapphire substrates. The uncertainty in the refractive index measurements is ± 0.005 , and all data is fit to firstorder Sellmeier equations over the wavelength span indicated. Ordinary refractive index data were compared with those measured using reflectance/transmittance spectroscopy. Samples were grown both by MOVCD and HVPE techniques, and the Al fractions x ranged from 0 to 0.71. Index measurements are critical to the design of nitride-based, short wavelength lasers for optical data storage and biological agent detection.

• Using x-ray diffraction analysis, compiled a complete set of "a" and "c" lattice constant data for the series of AlGaN samples. Correlated these data with the results illustrating the dependence of NLO coefficients on Al mole fraction. Showed that for samples with the Al fraction exceeding roughly 30 %, the expected symmetry relationship between second-order nonlinear coefficients $\mathfrak{X}_{33} = -2\mathfrak{X}_{31}$ fails. The AlGaN films are in a state of in-plane compressive strain with *c* axis to *a* axis strain ratios comparable to those in recently published work by other authors. This work will complete the SHG study of AlGaN and assist in correlating the results with lattice constants and strain.

Achieved a reduction in residual jitter by more than two orders of magnitude in a passively modelocked waveguide laser operating at 1534 nm. The round-trip frequency of the laser cavity was actively referenced to the frequency (472 MHz) of a stable electronic oscillator using PZT control of the cavity length and a phase-error detection circuit. In combination with temperature stabilization and pump isolation, this lowered the residual rootmean-square timing jitter from greater than 10 ps to as low as 83 fs over the range 100 Hz - 10 MHz. Compact, low-jitter optical pulse sources are critical for high-data-rate communication systems and optical sampling systems such as those used for high-speed, analog-to-digital conversion or optical eye-pattern evaluation.

Reduced the linewidth of an Er/Yb-doped, DBR waveguide laser operating near 1550 nm to less than 10 kHz through active and passive temperature stabilization. Demonstrated heterodyne beatnote production from two independent DBR solid state waveguide lasers. The linewidth is comparable with the best reported results for stabilized fiber ring lasers, but the waveguide lasers are simpler and more compact than those devices. Furthermore, the measured linewidth is much narrower than the 1 MHz or greater linewidth typical for semiconductor DFB laser diodes. The waveguide lasers are important to RF synthesis and sensing applications.

STANDARDS COMMITTEE PARTICIPATION

Telecommunications Industry Association: John B. Schlager is a member of working group FO-4.2.1, which deals with the Modal Dependence of Multimode Fiber Bandwidth.

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

NANOSTRUCTURE FABRICATION AND METROLOGY

Technical contact: Richard Mirin

Staff-Years (FY2003): 2.6 professionals 3.0 post-doctorals

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.



Emission spectrum of optically pumped, single InGaAs quantum dot.

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 lightemitting diodes to photonic integrated circuits to spontaneous emission control. Semiconductor quantum dots have an atomic-like density of states that offers substantial opportunities for unique and improved devices. These include ultra-low-threshold 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 properties 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 ondemand source of single photons. The singlephoton 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. Reflection highenergy electron diffraction (RHEED), atomic force microscopy (AFM), and transmission electron microscopy (TEM) techniques are applied to characterize quantum-dot morphology as a function of growth parameters. Photon counting and cavity ringdown methods are required to measure the optical properties of semiconductor nanostructures, particularly single quantum dots or small ensembles of dots.

DELIVERABLE: In 2003 we will measure the temperature-dependent absorption of single quantum dots using cavity ring-down spectroscopy.

We have developed ultrafast pump-probe methods to characterize carrier lifetimes and are measuring the homogeneous linewidth of quantum dots 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 measure the homogeneous linewidth of self-assembled InGaAs quantum dots in 2003.

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. Comprehensive modeling is key to the design and understanding of these nanostructures. Photonic crystal cavities and waveguides are expected to play important roles in advanced optoelectronic devices such as photonic integrated circuits.

DELIVERABLES: In 2003-4, we will investigate the dependence of optical properties on the fabrication parameters for 2D photonic crystal cavities embedded in a ridge waveguide and compare with calculations.

DELIVERABLE: In 2003, we will apply finite-difference time-domain (FDTD), full vector Maxwell, 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 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. We have implemented a Hanbury-Brown-Twiss apparatus to measure the photon statistics from our SPT. A true single-photon device will exhibit photon emission rates that are non-classical. Specifically, photon anti-bunching is expected to be observed.

DELIVERABLES: We will demonstrate an optically driven photon turnstile in 2003 and measure SPT operation with a single-electron pump in 2004. We will incorporate the SPT in a photonic crystal cavity and characterize its operation in 2004. We will develop a single-photon emitter based on rare-earth-dopants in semiconductors in 2005.

ACCOMPLISHMENTS

Measured the optical properties of a single self-assembled quantum dot (QD). The quantum dot was fabricated from InGaAs on a GaAs substrate using molecular beam epitaxy (MBE). The areal density of QDs was 1 per μ m² as measured using atomic force microscopy. This low density requires careful control of several MBE growth parameters, including total thickness and substrate temperature. Mesas of varying size were etched into the specimen to isolate small numbers of QDs as well as individual ODs. A mesa containing a single QD was optically excited using a 532 nm laser to create excitons in the dot, and these excitons then collapsed to emit single photons that were spectrally resolved using a grating and a CCD camera. Several emission peaks around 950 nm were observed from the single QD, including those due to excitons, charged excitons, and biexcitons. Understanding the optical emission from a single QD is essential for the development of an ondemand, single-photon source, for application to quantum-based radiometry and quantum cryptography.

 Measured polarization-dependent absorption on various ensembles of self-assembled InGaAs quantum dots in a waveguide. We devised a novel method of measuring absorption in a waveguide, in which a short laser pulse reflects back and forth in a long, low-Q cavity, and the output pulse energies are measured using sum-frequency generation with a gating pulse scanned in delay length. Absorption of both ground state and excited states was measured. In the ground state the quantum dots strongly absorb TE polarized light and do not absorb TM polarized light. Depending on the areal density of the quantum dots, the TE absorption coefficient is 4-6 cm⁻¹.

• Completed fabrication of the first generation of quantum-dot optically-gated field effect transistors (QDOGFETs), being developed for singlephoton detection. The design includes an on-chip resistance bridge that should allow measurements of resistance change of 1 part in 10⁶. Preliminary tests of the QDOGFET as a photodetector demonstrate sensitivity at the sub-picowatt level.

• Fabricated several photonic crystal nanocavities in GaAs thin films. The smallest nanocavity has a modal volume that approaches the fundamental physical size limit for supporting an optical mode. We used electron- beam lithography to write a pattern, followed by chemically assisted ion-beam etching (CAIBE) to transfer the pattern into the GaAs. These nanocavities are modeled using a full vector Maxwell solver.

• Developed a multi-bounce reflection spectroscopy setup to measure semiconductor distributed Bragg reflectors (DBRs). Results show that the reflectance can be measured with an uncertainty of 0.05 %.

RECENT PUBLICATIONS

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SEMICONDUCTOR GROWTH AND DEVICES

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.



Gas-source molecular beam epitaxy growth system used to study III-V crystal growth by Kris Bertness (in picture) and others at NIST. © Geoffrey Wheeler

CUSTOMER NEEDS

The continued growth of the U.S. optoelectronics industry depends 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 inprocess measurements. Many semiconductor devices now incorporate structures with a high degree of strain, and characterization of this strain and its relationship to device failure is an important area of research. In addition, specialty devices are needed for use in metrology systems inside and outside this project.

TECHNICAL STRATEGY

Inaccuracy of semiconductor composition measurement has impeded the achieving of 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 one tenth of 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 is paving the way for production of standard reference materials (SRMs) in the AlGaAs alloy system. As part of this research, we have quantified error sources and accuracy limits of the indirect composition measurement techniques, specifically PL and XRD, currently in use by industry. We have also demonstrated the compositional accuracy that can be achieved with direct microanalytical techniques such as EMPA in combination with accurate reference artifacts and carefully chosen correction procedures.

DELIVERABLE: In 2003, we will offer 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 samples with different compositions was analyzed by industrial 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 are being assessed. A necessary aspect of the study has been to distinguish between variations intrinsic to the materials and those caused by the measurement systems or techniques. Systematic measurements in a controlled environment at NIST will help clarify this and the appropriate measurement and calculation methods. Finally, we are collaborating with the NIST Materials Science and Engineering Laboratory to evaluate Raman

Technical Contact: Kris Bertness

Staff-Years (FY2003): 2.6 professionals 1.0 post-doctoral 2.0 graduate students spectroscopy as a possible method to quantify strain in InGaAsP.

DELIVERABLE: In 2003 we will report the analysis of a controlled study of important variables in PL measurements of InGaAsP at NIST. By 2004 we will collaboratively determine the stress dependence of the Raman and PL line positions. By 2005, 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. 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 has developed a cavity-ringdown 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, rather than measurement of absolute light intensities, and it is insensitive to absorption outside the cavity. Water content has been measured in phosphine and nitrogen gas with and without in-line purifiers. Further studies are correlating the impurity concentrations in phosphine with the quality of semiconductor material and devices grown using the gas. Of particular interest is the effect of impurities on nanostructures. Our system will also be used to measure the line shape, absorption coefficients, and frequency of optical transitions for water, phosphine and ammonia in the vicinity of 935 nm. This information is critical to facilitate the use of high-sensitivity spectroscopy techniques in these gases.

DELIVERABLE: By 2003, we will measure water concentrations in ammonia to 1 μ mol/mol. By 2003, we will measure pressure-broadening coefficients of water in phosphine for the stronger water absorption lines. By 2005 we will increase the sensitivity of impurity concentration measurement toward a goal of 5 nmol/mol.

Native-oxide layers have a significant impact on compound semiconductor 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 vertical cavity 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 electron backscatter diffraction (EBSD) and high-resolution convergent-beam electron diffraction (CBED). These techniques allow 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. We are also pursuing means to minimize the strain through appropriate choice of materials and oxidation conditions.

DELIVERABLE: By 2003, we will characterize the strain in GaAs layers adjacent oxidized AlGaAs layers. By 2004, we will publish the results of the study to minimize strain in devices employing native oxides.

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 substrate preparation and growth parameters with dot density and size as measured by atomic force microscopy (AFM). We are also evaluating the shape of the dots and strain in the region of the dots with transmission electron microscopy (TEM). **DELIVERABLE**: By 2003, we will evaluate the uniformity of quantum dots as a function of substrate preparation and growth conditions.

ACCOMPLISHMENTS

• Demonstrated the first measurement of water concentrations in phosphine using ringdown cavity spectroscopy. The ringdown results showed that the phosphine contained water at the several micromoles/mole level, much larger than the specifications provided by the manufacturers. There was indication that the phosphine cylinders can degrade substantially over time. With the same ringdown system, we measured a water vapor background of 150 ± 15 nanomoles/mole in purified nitrogen, and from these data we set our current detectivity limit at about 50 nanomoles/mole. This technique was developed in collaboration with NIST CSTL.



Cavity ring-down spectroscopy data of phosphine gas with and without in-line purification. The unpurified phosphine gas contains $18.0 \pm 1.7 \mu mol/mol$ of water, while there is no detectable water in the purified phosphine. The peaks in the purified phosphine gas spectrum arise from weak phosphine absorption lines.

Demonstrated that the oxidation kinetics of AlGaAs follows a Deal-Grove model and determined the reaction rates and diffusion coefficients. We found that the oxidation of layers with Al compositions from 90 % to 94 % are surface-reaction limited, leading to oxidation depths that vary linearly with time and oxidation rates that are exponentially dependent on temperature. For layers with 96 % Al and higher, the kinetics were in a mixed regime, where both the surface reaction and diffusion control the oxidation. The diffusion coefficients in the mixed regime are unusually high for a solid diffusion process, suggesting that diffusion takes place either through nanoscale pores in the oxide or a thin, dense layer near the semiconductor-oxide interface.

Developed methods to grow GaAs and AlGaAs films of acceptable quality on Ge substrates rather than the conventional growth on GaAs substrates. The new methods will be used to calibrate composition of standard thin films materials using analytical chemistry techniques executed in NIST's Chemical Science and Technology Laboratory. The analytical technique, inductively-coupled plasma optical emission spectroscopy (ICP-OES), was used this year to anchor the absolute calibration of the composition of AlGaAs thin films. Our initial results using ICP-OES agreed with EMPA, PL, and RHEED results within the uncertainty limits of all three techniques. ICP-OES analysis is traceable to the mole and has absolute calibration uncertainty of less than 0.1 %. The Ge substrates were necessary, however, because any contamination from the GaAs substrate would add larger uncertainty to the measurement.

• Determined that the absolute calibration of the reflectance in an *in situ* optical reflectance spectroscopy (ORS) system must be accurate to 0.05 % in order to determine A1 mole fraction x in $A1_xGa_{1,x}$ As to an uncertainty of 0.003. Improving the noise in the reflectance below a standard deviation of 0.3 % does not relax this requirement until the noise is below 0.02 %. Neither the calibration goal nor the noise requirements are readily achieved in practice. We plan to look at ways to use growth rate information determined from other methods to constrain the ORS data fits and thereby obtain more accurate values for index of refraction at growth temperature.

• Determined that the uncertainty in RHEED growth rate measurements varies from 1 % to 7 % depending on specimen size, control of RHEED beam position, and data analysis method. We upgraded our RHEED equipment and refined our procedures to reach the lower end of this range for further work on AlGaAs composition standards. We also collaborated with NIST's Information Technology Laboratory to identify the optimal use of the redundant data collected in order to minimize the error propagated to the Al mole fraction as determined from RHEED growth rate data.

Measured strain in semiconductor nanostructures. In collaboration with the NIST Materials Science and Engineering Laboratory (MSEL) we obtained qualitative measurements of strain in the vicinity of an AlGaAs oxidation front using electron backscatter diffraction (EBSD). The EBSD map indicated that the highest strain was found near but behind the oxidation front. We have also provided AlGaAs specimens to another group in MSEL for micro-Raman studies. This past year the group has made the first determination of the shifts in Raman and photoluminescence peaks for AlGaAs under biaxial stress.

Analyzed the results of measurements of x-ray and photoluminescence taken by five different companies, using eight different instrument sets in the InGaAsP interlaboratory comparison. The specimens were grown by industrial collaborators. We found that x-ray rocking-curve accuracy appeared to be limited primarily by nonuniformity in the specimen strain and/or composition. Future study will be carried out with specimens having greater uniformity. We have also undertaken a major equipment purchase to enhance our ability to determine strain with x-ray. In contrast with the x-ray results, the study revealed significant variations in photoluminescence peak wavelength measurements from laboratory to laboratory. These variations were large compared with wavelength grid spacing in dense wavelength-division-multiplexed systems. The variations were also large compared with specimen uniformity. They appeared uncorrelated with the instrument settings and environmental conditions reported by participants. Because the variables were measured in different labs with different tools and differing degrees of precision, this does not necessarily mean that real correlations do not exist. Further systematic measurements at NIST will clarify this issue.

RECENT PUBLICATIONS

Roshko, A.; Bertness, K.; Armstrong, J.; Marinenko, R.; Salit, M.; Robins, L.; Paul, A.; Matyi, R., "X-ray, Photoluminescence and Composition Standards of Compound Semiconductors," accepted for publication in *Phys. Stat. Sol.*

Roshko, A.; Bertness, K.A., "Interlaboratory Comparison of InGaAsP *Ex-situ* Characterization," accepted for publication in *J. Crystal Growth*.

Fu, C.; Bertness, K.A.; Wang, C., "Effects of Noise Level in Fitting In-Situ Optical Reflectance Spectroscopy Data," submitted to *J. Crystal Growth*.

Harvey, T.E.; Bertness, K.A.; Wang, C.; Splett, J.D., "Accuracy of AlGaAs Rates and Composition Determination Using RHEED Oscillations," submitted to *J. Crystal Growth.*

Chen, Y.; Roshko, A.; Bertness, K.A.; Readey, D.W.; Allerman, A.A.; Tan, M.; Tandon, A., "Comparison of AlGaAs Oxidation in MBE and MOCVD Grown Samples," *Proc., Mat. Res. Soc. Symposium.*, Vol. 692, Nov. 26-29, 2001, Boston, MA, H6.11.1-6 (2001).

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 beamsplitterbased 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, 193 nm, and 157 nm), the Division has the only measurement capability in the world.



APPENDIX B: NRC POST-DOCTORAL 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 post-doctoral 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 post-doctoral level for tenure as guest researchers at participating laboratories. The deadline for applications is February 1 for appointments beginning between July and the following January.

THE OBJECTIVES OF THE PROGRAMS ARE:

• To provide post-doctoral 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 base salary for the 2003 program year is \$55,700.

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

Opportunities for the year 2003 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. Post-doctorals 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 Email: pkwright@boulder.nist.gov http://www.boulder.nist.gov/exec/bdprepo.htm

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 2½-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 3½-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.

Laser Power and Energy Calibrations			
	Laser	Wavelength	Range
CW	He Cd	325 nm	100 nW to 10 mW
	Argon	488,514 nm	1 nW to 2 W
	Doubled Nd:YAG	532 nm	1 nW to 10 W
	HeNe	633 nm	1 nW to 20 mW
	Diode	830 nm	1 nW to 20 mW
	Ti:Sapphire	700 to 1,000 nm	Up to 1 W
	Dye	550 to 700 nm	Variable
	Nd:YAG	1,064 nm	1 μW to 450 W (6 kW off-site)
		1,319 nm	1 µW to 10 mW
	Erbium	1,550 nm	1 µW to 10 mW
	CO ₂	10.6 µm	35 mW to 900 W (6 kW off-site)
Pulsed	KrF	248 nm	50 µJ/pulse to 150 mJ/pulse
			3 mW to 7 W average
	ArF	193 nm	20 µJ/pulse to 150 mJ/pulse
			3 mW to 3 W average
			Dose (Irradiance) Measurements
	F ₂	157 nm	20 µJ/pulse to 20 mJ/pulse
			3 mW to 4 W average
	Nd:YAG	1,064 nm	10 µJ to 300 mJ/pulse (1 to 20 Hz prf)
			10 ⁻⁴ to 10 ⁻⁸ W(peak)
			10^{-11} to 10^{-15} J/pulse

APPENDIX D: CALIBRATION SERVICES

Optical Fiber Power Meters and Detectors			
Parameter	Wavelength	Range	Status
	670, 780, 850, 980, 1310, 1550 nm	10 to 100 µW	Calibration Service
Absolute Power Calibration	830 to 860 nm, 1270 to 1330 nm, 1510 to 1560 nm, 1580 to 1625 nm	10 to 100 μW	Calibration Service
	980, 1290, 1550 nm	1 to 250 mW	Special Test
Optical Receiver	1319 nm	300 kHz to 50 GHz	Calibration Service
Frequency Response	850 and 1550 nm	1 MHz to 50 GHz	Special Test
Optical-Fiber Power 850, 1310, 1550 nm Meter Linearity		60 to 90 dB	Calibration Service
Reference Receiver 800 and 1550 nm Impulse Response		100 fs impulse source	Special Test
Optical-Detector Spatial 635, 850, 1300, 1550 nm Uniformity			Special Test
Optical-Fiber Power 400 to 1700 nm Meter Spectral Responsivity			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, Group Leader at (303) 497-7455, mdowell@boulder.nist.gov.

APPENDIX E: STANDARD REFERENCE MATERIALS

Standard Reference Materials					
SRM Number	Name	Brief Description	Availability		
	Composition Standards				
XXXX	Composition Standards for Compound Semiconductors	None Yet Available	2003		
	Wavelength	h Calibration Standards			
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	Available		
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	Available		
2517a	High-Resolution Wavelength Calibration Reference for 1510-1540 nm – Acetylene $({}^{12}C_{2}H_{2})$	Fiber-coupled molecular gas adsorp- tion 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			
2518	Polarization Mode Dispersion Standard	Device with stable and known value of polarization mode dispersion that simulates optical fiber	Available		
2538	Deterministic Polarization Mode Dispersion Standard	To simulate PMD in discrete components — wavelengths 1250 to 1650 nm	Available		
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		
	Continu	ed on next page			

The following fiber and fiber connector geometry SRMs are produced by NIST's				
Precision 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 mm known within ± 0.1 mm.	Available	
2554	Optical Fiber Coating Diameter	Glass rod with index of refraction $n = 1.515$ and diameter of approximately 250 mm known within ± 0.1 mm.	Available	
2555	Optical Fiber Coating Diameter	Glass rod with index of refraction $n = 1.535$ and diameter of approximately 250 mm known within ± 0.1 mm.	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 20899-8210				
	Phone: (301) 975-3463 Fax: (301) 869-0822			
Measurement Assurance Programs				
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 °	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

National Institute of Standards and Technology 100 Bureau Drive, Stop 2322 Gaithersburg, MD 20899-2322 Phone: (301) 975-6776 Fax: (301) 948-3730 email: SRMINFO@nist.gov

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