InGaN-Based Multi-Quantum-Well-Structured Laser Diodes Fabricated

At present, the main focus of III-V nitride device research is the realization of a current-injected laser diode which is expected to be the shortest-wavelength semiconductor laser diode demonstrated. The bandgap energy of AlInGaN compound varies from 2.0 to 6.2 eV, which is larger than the bandgap energy of other compound materials. Stimulated emission, however, has been observed only by optical pumping, and not by current injection. Using wide bandgap III-V nitride materials, Shuji Nakamura from Nichia Chemical Industries, Ltd. and his collaborators have fabricated laser diodes which emit coherent light at 417 nm from InGaN-based multi-quantum-well (MQW) structures under pulsed current injection at room temperature, as reported in their article published in the January 15 issue of the *Japanese Journal of Applied Physics.*

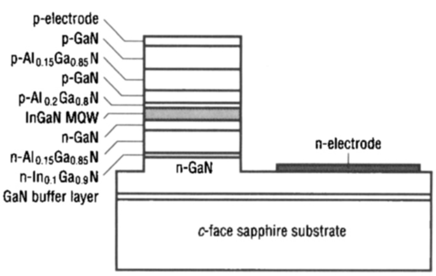
The InGaN MQW laser diode device consisted of a 300 Å GaN buffer layer grown at a low temperature of 550°C, a 3-µm-thick layer of *n*-type GaN:Si, a 0.1-µm-thick layer of *n*-type $In_{0.1}Ga_{0.9}N$:Si, a 0.4-µm-thick layer of *n*-type $Al_{0.15}Ga_{0.85}N$:Si, a 0.1-µm-thick layer of *n*-type GaN:Si, 26 periods of a $In_{0.2}Ga_{0.8}N/In_{0.05}Ga_{0.95}N$ MQW structure consisting of 25-Å-thick $In_{0.2}Ga_{0.8}N$ well layers and 50-Å-thick $In_{0.05}Ga_{0.95}N$ barrier layers, a 200-Å-thick layer of *p*-type $Al_{0.2}Ga_{0.8}N$:Mg, a 0.1-µm-thick layer of *p*-type GaN:Mg, a 0.4-µm-thick layer of *p*-type $Al_{0.15}Ga_{0.85}N$:Mg, and a 0.5-µm-thick layer of *p*-type GaN:Mg (see figure).

The 0.1-µm-thick layer of *n*-type $In_{0.1}Ga_{0.9}N$ served as a buffer layer of the thick AlGaN film growth to prevent cracking of the film. The 200-Å-thick layer of *p*-type $Al_{0.2}Ga_{0.8}N$ was used to prevent dissociation of InGaN layers during the growth of the *p*-type layers. The 0.1-µm-thick *n*-type and *p*-type GaN layers were light-guiding layers. The 0.4-µm-thick *n*-type and *p*-type $Al_{0.15}Ga_{0.85}N$ layers were cladding layers for confinement of light emitted from the active region of the InGaN MQW structure.

It is difficult to cleave the GaN crystal grown on the *c*-face sapphire substrate. Therefore, reactive ion etching (RIE) was employed to form mirror cavity facets. The surface of the *p*-type GaN layer was partially etched with Cl_2 plasma until the *n*-type GaN layer was exposed in order to make a stripe laser diode. The roughness of the facet surface was approximately 500 Å. The area of the stripe laser diode is 30 µm x 1,500 µm. High reflection facet coatings (60–70%) were used to reduce the threshold current. A Ni/Au contact was evaporated onto the entire area of the *p*-type GaN layer, and a Ti/Al contact onto the *n*-type GaN layer. The electrical characteristics of laser diodes were measured under pulsed current-biased conditions (pulse width is 2 µs, pulse period is 2 ms) at room temperature. The output power from one facet was measured by a Si photodetector.

The threshold current was about 1.7 A, which corresponded to a threshold current density of 4 kA/cm². The operating voltage of this device at the threshold current was 34 V. The differential quantum efficiency of 13% per facet and pulsed output power of 215 mW per facet were obtained at a current of 2.3 A. At injection currents below the threshold, the spontaneous emission, which had a full width at half-maximum (FWHM) of 20 nm and a peak wavelength of 410 nm, appeared. Above the threshold current, a strong stimulated emission at 417 nm with a FWHM of 1.6 nm was the dominant emission.

ZnSe-based laser diodes have emitted the shortest wavelength emission under pulsed current at liquid nitrogen temperature. Nakamura and his collaborators used the InGaN MQW structure as an active layer of the laser diodes. The number (26) of quantum wells was much higher than that of the conventional MQW laser diodes.



The structure of the InGaN MQW laser diode.

Laminated Matrix Composites Expand Uses for High-Temperature Parts

By applying alternating thin layers of matrix materials to traditional reinforcement structures, researchers at the Georgia Tech Research Institute (GTRI) have developed laminated matrix composites they believe will be tougher and stronger than conventional fiber-reinforced composites.

Two conventional techniques exist for making mechanically tough composites. The first uses fiber reinforcement within matrix materials such as ceramics or metals, while the other relies on building up multiple bonded layers of different materials such as copper and aluminum.

"We have combined those two approaches," said W. Jack Lackey, a research scientist at GTRI. "We make a fibrous preform by stacking up layers of cloth, then infiltrate a matrix into the preform one layer at a time. We infiltrate for a few moments with one material until we get a layer of it around each fiber, then we infiltrate for a few moments with another material, then we switch back to the first. We keep iterating until we have put down as many as 50 layers."

The researchers used forced-flow thermal-gradient chemical vapor infiltration to apply layers ranging in thickness from 0.02 to 0.5 µm. The layers were formed by alternating the precursor gases flowing into the chemical vapor infiltration reactor.

The work initially focused on infiltrating a carbon fiber preform with alternate layers of carbon and silicon carbide matrix. However, the process should also be applicable to silicon-carbide or aluminum-oxide fibers, as well as to metallic, polymeric, or ceramic matrix materials.

Lackey said that the matrix materials—which bind the reinforcement fibers together and fill in the space between them—must be carefully chosen to be chemically compatible and closely matched in their thermal expansion properties. He said that carbon and silicon carbide are a natural choice. Those materials were easy to infiltrate into a preform, and were compatible chemically. The researchers want to study what kinds of materials work well in this type of composite.

"People are struggling to improve the mechanical properties of ceramics and ceramic-matrix composites to the point that they can replace metals," Lackey said. "If you can replace metals with ceramics, you can operate at higher temperatures and get better efficiency in a heat engine."

Microstructure of Spider Silk Gives Clue to Toughness

Scientists hoping to produce bio-inspired fibers are a step closer with a model for the molecular arrangement of spider silk, proposed by Cornell University researchers in the January 5 issue of *Science.*

Focusing nuclear magnetic resonance (NMR) studies on the dragline silk from the golden orb-weaver spider—and on the crystalline amino acid alanine—the scientists found a blend of highly oriented

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segments, amorphous material and barely oriented segments, all working together to make a fiber that is stronger than steel and much more elastic. Dragline silk is stronger, per cross-sectional area, than steel, yet it can stretch to, and rebound from, 15% of its original length.

Led by Lynn W. Jelinski, physics graduate student Carl A. Michal and postdoctoral researcher Alexandra H. Simmons, now a staff scientist at DuPont Canada, examined alanine, which is known to reside in the crystalline regions of spider silk. They fed the spiders a diet that included deuterated (or "heavy") alanine and collected the deuterium-labeled silk on a spindle with their homemade silking machine. They then probed the deuterated alanine using nuclear magnetic resonance.

The researchers found two types of crystalline alanines. Using computer simulations, Michal found that 40% of the alanines are as highly oriented as molecules in the synthetic fiber Kevlar—an unexpected finding for a biological fiber. The other 60% are far less oriented, but are crystalline nevertheless.

The researchers said, "These poorly oriented crystallites may be important in effectively coupling the highly oriented domains and the amorphous regions, producing a biomaterial with exceptional toughness."

Polymer Concrete Shows Promise for Civil Engineering Applications

Researchers at the University of Delaware Center for Composite Materials (CCM) are demonstrating that polymer concrete—a composite comprising of thermosetting resin (generally, vinyl-ester, polyester, or epoxy) and a mineral aggregate (stone)—shows promise for use in a variety of building applications. "Compared to Portland cement, polymer concrete shows significantly improved flexural, compressive, and tensile properties," said Giuseppe R. Palmese, research associate at CCM. "At the same time, because of the polymer content, it offers improved damping characteristics and good protection against corrosive environments."

The work underway at CCM under the Bridge Infrastructure Renewal (BIR) Program with the University of California at San Diego focuses on the use of polymer concrete as a wear surface for all-composite bridge decks. The researchers expect the bond between the composite (such as glass/vinyl-ester) used in the bridge deck and the polymer concrete (such as a vinyl-ester-based polymer concrete) wear surface to offer improved mechanical properties over bridge materials currently used. Palmese said that the durability of these materials is affected by the weakness of the interphase between the aggregate and the polymer. The researchers are studying various issues involved with the use of polymer concrete as a wear surface, including control of the thermal expansion coefficient to ensure proper bonding and long-term environmental durability.

Edited from *Composites Update: The Newsletter of the University of Delaware Center for Composite Materials*, Issue No. 3, 1995.

Low-Residue Soldering for Electronics Reduces Pollution, Saves Time

Sandia National Laboratories coordinated a task force to study a low-residue soldering process, concluding that this process saves time and money and reduces pollution, in comparison with conventional processes.

Conventional processes for soldering printed wiring boards use an activated rosin-based flux prior to soldering to remove oxidation. Oxidation also is prevented by doing the soldering in a sealed nitrogen chamber. The rosin-based flux leaves a tacky residue that previously was removed with ozone-depleting chlorofluorocarbon (CFC) solvents.

Low-residue processes eliminate the need for these CFC cleaning solvents by using a mild organic acid as the fluxing agent. These organic agents, such as the common food additive adipic acid, leave little residue and require no cleaning.

Jeff Koon, a Texas Instruments process engineer who served on the task force, said the low-residue process is friendlier to the environment in several ways. It eliminates the need for CFCs and other noxious cleaning materials, and it significantly reduces the amount of lead dross produced during the manufacturing process.

Electrochemical Process Treats Variety of Toxic Waste

Researchers at Los Alamos National Laboratory and Faraday Technology Inc. have tested an electrochemical process to treat hazardous wastes. Running an electric current through a waste solution splits salts into acids and bases or extract heavy metals; for example mercury will stick to graphite cathodes. Cyanides and toxic organic compounds are broken into harmless carbon dioxide and nitrogen.

"The problem here is the diversity of waste types," said Jacek Dziewinski, from Los Alamos, who leads the project. "We have thousands of compositions, but not a large volume of any. So it makes sense to address many kinds of chemicals with one technique."

The system uses several electrochemical cells, each designed to break down a separate group of wastes. It recovers and recycles materials rather than merely immobilizing and disposing of them. Once treated for other toxic substances, the radioactive component of mixed waste can be handled by other processes. The electrochemical process works at room temperature and low pressure, runs on familiar direct-current electricity, and does not produce toxic fumes or secondary waste.

Many electrochemical processes are standard technologies in industry. However, breaking down chemicals with an electric current, or electrolysis, has not been used at an industrial scale for waste treatment. The main challenge, said Dziewinski, lies in applying the method to waste management operations that require treatment to very low concentrations—down to micrograms per liter.

Process Functionalizes Polymers and Enables Oxidation

Research Corporation Technologies has received a U.S. patent for a process that functionalizes alkylstyrene-based polymers and copolymers. The process enables oxidation of nonpolar methyl and alkyl functional groups to polar acid, aldehyde, or alcohol groups by using a combination of solvents and catalysts. This allows the selective placement of polar functionalities on homopolymers and block copolymers. The process permits control of block copolymer molecular weight and degree of polarity within each polymer block.

The process combines appropriate solvents and catalysts with a range of polymers. Oxidation type and extent of oxidation are controlled by the selection of solvents and reaction times. One of the inventors, H.D.H. Stöver, has obtained polymer conversions up to 98% on homopolymers, up to 40% on short block copolymers, and up to 15% on long-chain block copolymers.

A methyl-styrenic polymer, for example, is oxidized by first adding the polymer to acetic acid solvent and an organic cosolvent. A cobalt oxidation catalyst and a sodium cocatalyst are then added to oxidize the methyl functional groups, first to aldehyde groups and then to carboxylic acid groups after longer exposure of the polymer to the reaction mixture. When the mixture is added to water, the new modified polymers are precipitated out.

Phillips Manages Surface/Molecular Spectroscopy Dept. at Sandia

Julia M. Phillips has moved to Sandia National Laboratories in September 1995 as manager of the Surface/Molecular Spectroscopy Department. She was formerly technical manager of the Thin Film Research Group at AT&T Bell Laboratories where research centered on the growth and properties of thin films, including epitaxial insulators and metals, high-temperature superconductors, transparent conductors, and electronic-oxide materials. Prior

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to that, from 1981 to 1988, she was a member of the technical staff at AT&T.

Phillips received her PhD degree in applied physics from Yale University. She has served on the editorial board of *Applied Physics Letters* and *Journal of Applied Physics*, and is currently principal editor of the *Journal of Materials Research*. Phillips's professional affiliations include the American Physical Society, Sigma Xi, and the Materials Research Society where she is currently serving as immediate past president.

Sunlight Used to Power Laser

Researchers at the National Renewable Energy Laboratory (NREL) and the University of Chicago powered a laser with concentrated sunlight instead of electricity. A series of mirrors concentrated the sunlight into an intense, focused beam that reached concentrations of up to 50,000 suns.

To create the solar laser, a neodymium-doped yttrium aluminum garnet (Nd:YAG) laser crystal was mounted at the target of a specially designed secondary concentrator. The crystal and secondary concentrator were then placed at the focal point of the primary concentrator. When sunlight was added, this concave-convex mirror system created a solar laser.

The laser had a peak power output of 57 W. Lasers are very energy intensive, consuming much more energy than they produce. Those powered by electricity operate at about 1–2% efficiency, meaning they require 10 kW of energy to produce 100 to 200 W lasers. The solar laser has an efficiency of almost 1%. The researchers achieved the highest reported efficiency for a solar-pumped laser operating at this power level. They expect the efficiency to improve as researchers refine and optimize the system. They believe that solar lasers may prove more efficient than traditional lasers.

SBIR Update

Lightning Optical Corporation (Tarpon Springs, Florida) has been awarded two SBIR contracts. One is a Phase II SBIR contract from NASA Langley Research Center for approximately $600,000 for the crystal growth of highly doped Cr:$LiSrAlF_6$ for diode-pumping applications. The other contract signed together with the **University of Central Florida** (Orlando, Florida) is a Phase I award of $71,500 from the Department of Energy for the crystal growth and characterization of Cr-doped $LiSrGaF_6$ for diode-pumped, solid-state laser applications.

James E. West Awarded Silver Engineering Medal

James E. West of AT&T Bell Laboratories was awarded the Acoustical Society of America's Silver Medal for co-inventing with Gerhard Sessler the foil electret microphone. Invented in 1962, the foil-electret microphone is made with a metalized thin foil of material that is given a permanent electrical charge. Transducers based on this device convert sound into an electrical signal, and are used in hearing aids, lapel microphones, and tape recorders.

West is now applying electret principles to devices used in the medical field. He helped develop blood pressure transducers, and collaborates with Brown University on the regeneration of nerve cells from damaged tissues by using the small electrical fields created by electrets.

A Bell Labs Fellow and distinguished member of technical staff in the Acoustics Research Department, West has received numerous honors. He has 36 U.S. patents and more than 100 patents issued outside the United States on various microphones and techniques for permanently charging polymers. He also has authored or coauthored more than 100 technical papers and has contributed to several books on acoustics, solid state physics, and materials science.

Recently Announced CRADAs

Structured Materials Industries, Inc. (SMI) (Piscataway, New Jersey) and the **Army Research Laboratory** (Fort Monmouth, New Jersey) signed an agreement to develop nanocrystal-based phosphors for luminescent-based displays.

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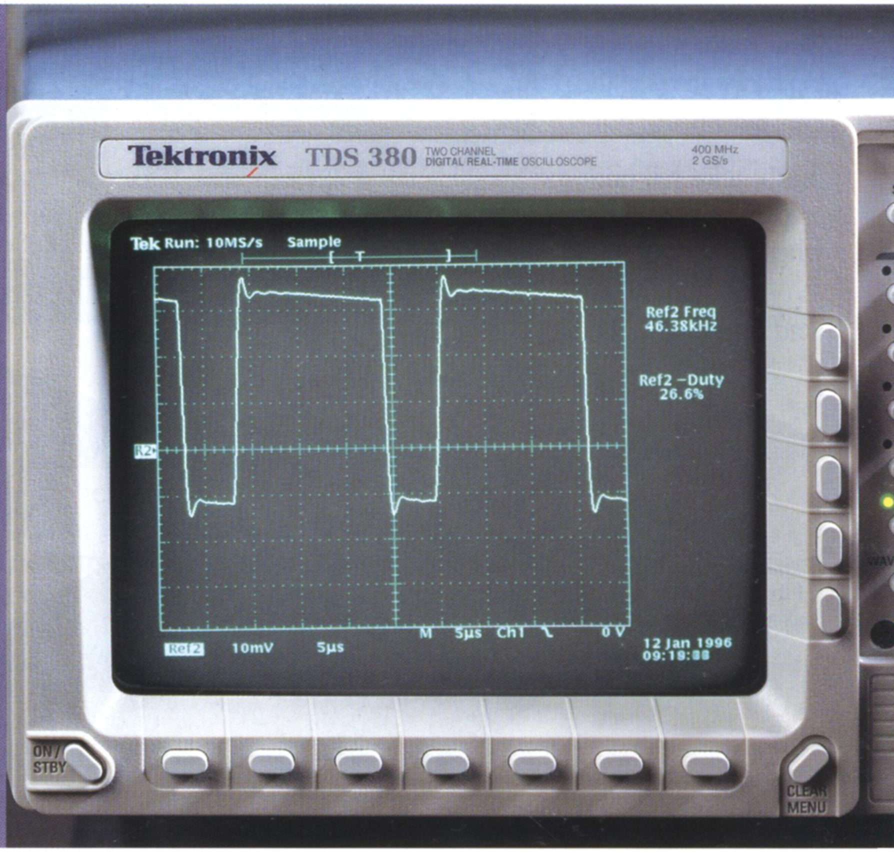

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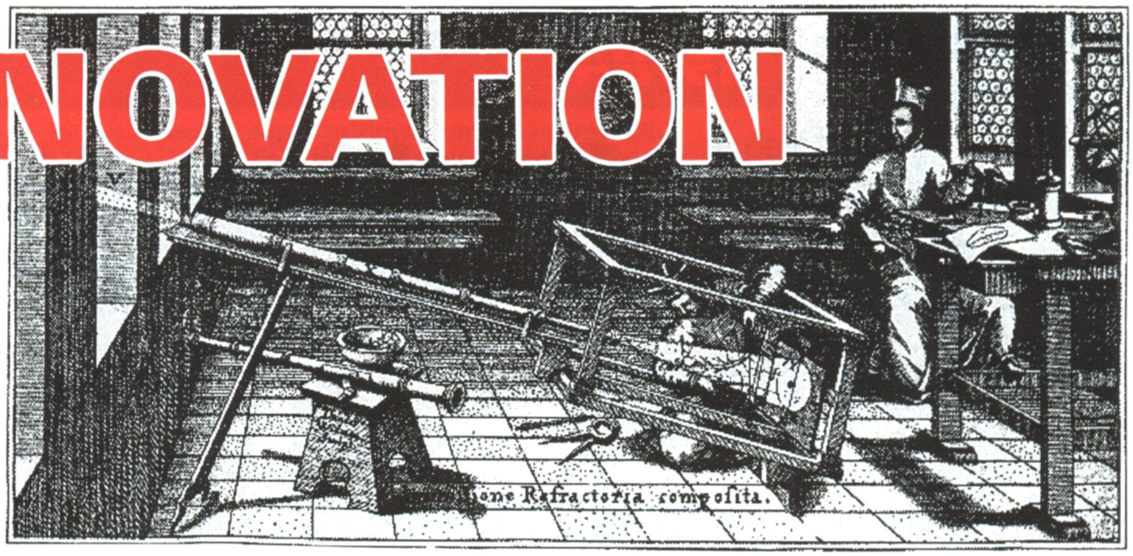

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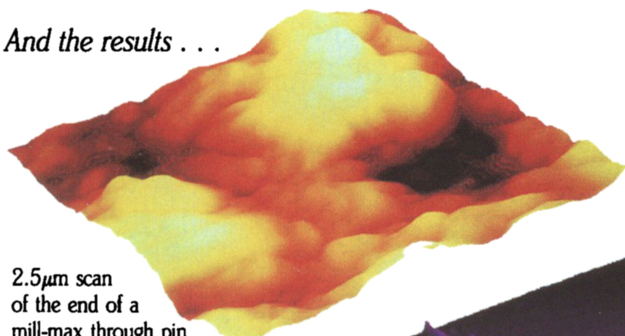
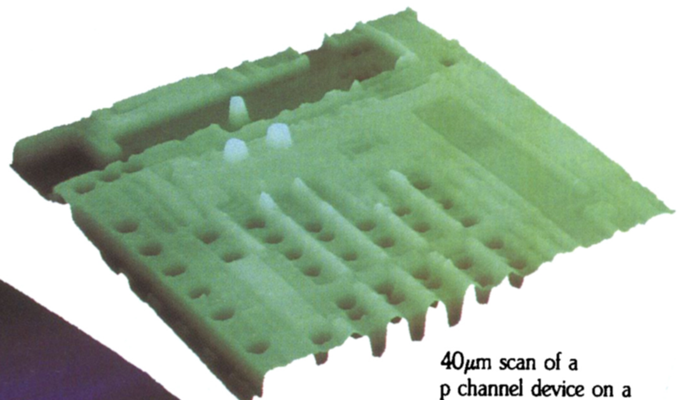
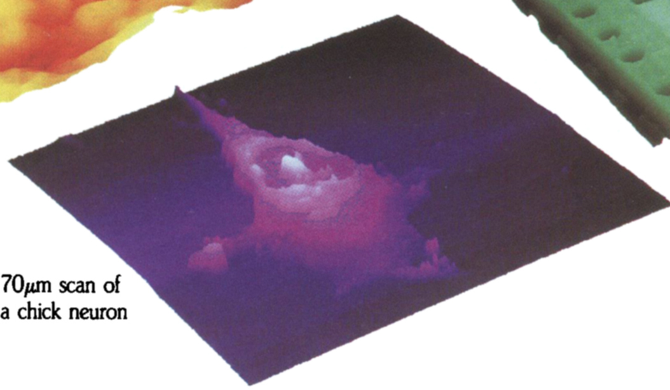


2.5μm scan of the end of a mill-max through pin



40μm scan of a p channel device on a semiconductor wafer



70μm scan of a chick neuron

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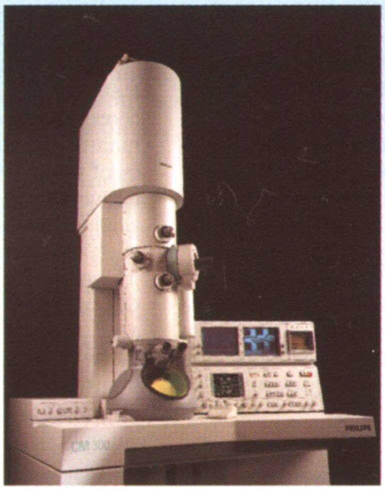
Points of view for advanced materials analysis



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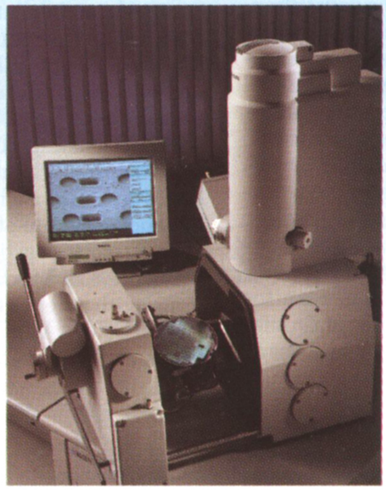

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