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14. ABSTRACT This Phase II STTR project developed and demonstrated a compact, turn-key, cryogen-free, p-type germanium laser, tunable over the range 1.5-4.2 THz (70-200 micron wavelength), with 1 Watt peak power. A new type of intracavity gain modulator with potential application to active modelocking was discovered. Improvement in the gain of the active p-Ge crystal by homogeneous neutron transmutation doping was experimentally shown. An alternative means of gain improvement, which allows operation at 77 K, via periodic delta-doping in chemical-vapor-deposited planar Ge structures was realized through Monte Carlo simulations. Potential applications to satellite communications, bio/chem sensing, non-destructive testing, and mine detection were considered.					
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Objectives

- Compact, turn-key, cryogen-free operation of THz p-Ge laser
- Frequency selection in range 1.5 – 4.2 THz (50-140 cm^{-1}).
- Intracavity modulator for mode-locking
- Gain improvements
- Applications development

Work done and results

Nearly all work done and results obtained relevant to each of the project objectives is described in 5 refereed journal publications, 16 conference publications, a PhD dissertation, a MS thesis, and a patent disclosure. Summaries are presented next along with some recent unpublished results.

Compact, turn-key, cryogen-free operation of THz p-Ge laser

Operation of a turn-key, cryogen-free bulk p-Ge laser was achieved in a 4 K closed cycle refrigerator. A SmCo permanent magnet assembly provided the necessary magnetic field for the laser. A customized high voltage (HV) power supply and thyatron pulser were developed to replace the stack of general electronics previously used to operate the laser, and a fivefold decrease in weight and volume was achieved. These developments are described in

"High field p-Ge laser operation in permanent magnet assembly," C. J. Fredricksen, E. W. Nelson, A. V. Muravjov, and R. E. Peale, *Infrared Physics and Technology* **44**, 79-84 (2003), and

"Operation of THz p-Ge laser in a closed cycle refrigerator," C. J. Fredricksen, A. V. Muravjov, and R. E. Peale, in Terahertz for Military and Security Applications, edited by R. J. Hwu and D. L. Woolard, Proc. SPIE v. 5411 (2004).

Wavelength selection in the range 1.5-4.2 THz (50-140 cm^{-1}).

Operation of a p-Ge laser in an open quasi-optical resonator was demonstrated. This contrasts with previous designs where mirrors were fixed to surfaces of the active crystal. Enhanced stability and tuning of the laser cavity length were demonstrated, which are steps toward continuous tunability without mode-hops. This work is described in

"Far-infrared p-Ge laser with variable length cavity," A. V. Muravjov, E. W. Nelson, R. E. Peale, V. N. Shastin, and C. J. Fredricksen, *Infrared Physics & Technology* **44**, 75-78 (2003).

A robust metal-free intracavity fixed-wavelength selector for the p-Ge laser was demonstrated. The device is a back mirror consisting of a thin silicon etalon and dielectric SrTiO_3 flat. A laser line width of 0.2 cm^{-1} was achieved, which corresponds to an active cavity finesse of ~ 0.15 . The wavelength position and spectral purity are maintained over a wide range of laser operating fields. Use of SrTiO_3 lowers the laser resonance line frequencies by $\sim 1 \text{ cm}^{-1}$ compared with expectations for metal mirrors.

The effect is due to phase shift, which was determined from far-infrared reflectivity measurements of SrTiO₃. A p-Ge laser with such selector is free from danger of electrical breakdown and mirror oxidation during repeatable thermal cycling, which makes it more reliable than previous selection schemes for practical applications. This work is summarized in

"Dielectric selective mirror for intracavity wavelength selection in far-infrared p-Ge lasers," T. W. Du Bosq, R. E. Peale, E. W. Nelson, A. V. Muravjov, C. J. Fredricksen, N. Tache, and D. B. Tanner, *J. Appl. Phys.* 94, 5474 (2003), and

"Fixed wavelength selection for the far-infrared p-Ge laser using thin silicon intracavity etalon," T. W. Du Bosq, R. E. Peale, E. W. Nelson, A. V. Muravjov, C. J. Fredricksen, in *Solid State Lasers XII*, R. Scheps, Ed., Proc. SPIE Vol. 4968, pp. 18-23 (2003).

A lamellar mirror made from Si wafer by anisotropic chemical etching and coated with gold was demonstrated as an intracavity wavelength selector for the p-Ge laser. The etching process produces rectangular grooves with precisely predetermined depth and 100 nm surface smoothness. This lamellar-grating structure defines the resonant laser wavelength within the 200 to 70 μm wavelength range of the p-Ge laser. Single wavelength laser operation with this mirror was demonstrated on the third-order resonance with an active cavity finesse of at least 0.09. A p-Ge laser equipped with such a selector easily can be prepared to operate in regions of relatively high atmospheric transmission for applications in chemical sensing, THz imaging, and non-destructive testing. The same processing technique can be used to create more complicated selective mirrors, for example with multiple depths. This work is described in:

"Wavelength selection for the far-infrared p-Ge laser using etched silicon lamellar gratings," T. W. Du Bosq, R. E. Peale, E. W. Nelson, A. V. Muravjov, D. A. Walters, G. Subramanian, K. B. Sundaram, and C. J. Fredricksen, *Accepted Optics & Laser Technology* 2004,

"Wavelength selector developments for the far-infrared p-Ge laser," R. E. Peale, A. V. Muravjov, E. W. Nelson, Todd Du Bosq, K. B. Sundaram, and C. J. Fredricksen, in *OSA annual meeting* (OSA Technical Digest, Washington DC, 2002) paper WO6,

"Fixed wavelength selection for the far-infrared p-Ge laser using patterned silicon etched mirrors," T. W. Du Bosq, R. E. Peale, E. W. Nelson, A. V. Muravjov, D. A. Walters, G. Subramanian, K. B. Sundaram, C. J. Fredricksen, in *Chemical and Biological Sensing IV*, edited by P. J. Gardner, Proc. SPIE v. 5085, pp 119-125 (2003), and

Intracavity wavelength selectors for the p-Ge far-infrared laser, T. W. Du Bosq, Master's Thesis (University of Central Florida, Orlando, FL, 2003).

Multi-layer mirrors capable of >99.9% reflectivity at ~100 μm wavelengths were constructed using thin silicon etalons separated by empty gaps. Due to the large difference between the index of refraction of silicon (3.384) and vacuum (1), calculations indicate that only three periods are required to produce 99.9% reflectivity. The mirror was assembled from high purity silicon wafers, with resistivity over 4000 ohm-cm to reduce free carrier absorption. Wafers were double side polished with faces parallel within 10 arc seconds. The multi-layer mirror was demonstrated as a cavity mirror for the far-infrared p-Ge laser. A future application for such mirrors is a far-IR cavity ring-down spectrometer for ultra-trace detection. One publication describes this work,

"High reflectivity intracavity Bragg mirrors for the far-infrared p-Ge laser," T. W. Du Bosq, A. V. Muravjov, R. E. Peale, in Terahertz for Military and Security Applications II, edited by R. J. Hwu and D. L. Woolard, Proc. SPIE v. 5411 (2004).

A collection of different laser lines produced with the different selectors developed under this project is shown in Fig. 1.

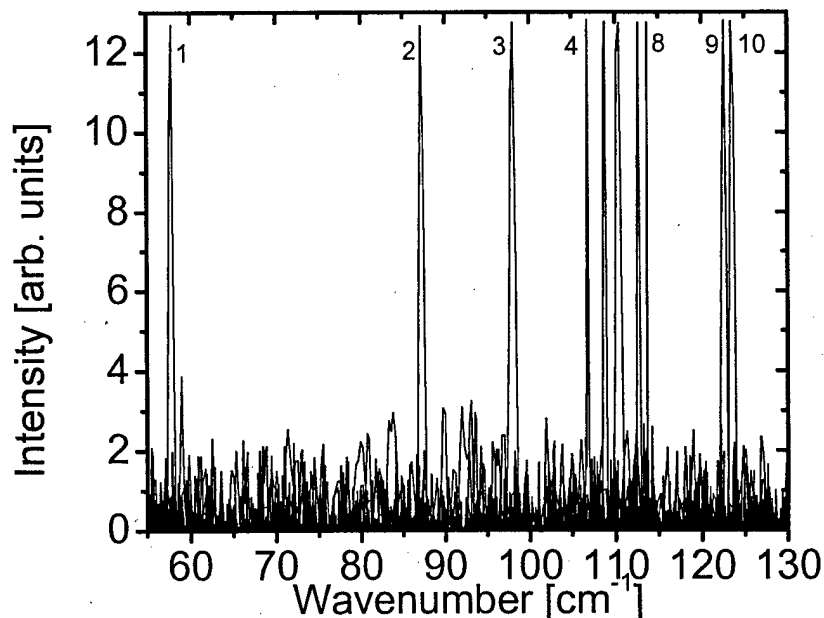


Fig. 1. Ten superimposed p-Ge laser emission spectra. Different intracavity selection devices are used in each case.

Intracavity modulator for modelocking

Optical quenching of the THz inter-sub-band p-Ge laser (tunable in the wavelength range 70-200 μm with ~1W output power) by Nd:YAG laser radiation was investigated. YAG laser pulses were coupled into a p-Ge laser cavity through a SrTiO₃ laser mirror, which is highly reflecting at cryogenic temperatures for THz frequencies and transparent for visible and near-IR light. Fast quenching of the p-Ge laser emission intensity was observed and attributed to free carrier absorption by optically generated electron-hole pairs in a thin layer of the active p-Ge crystal end surface. The effect also

occurs when the interband absorption is confined to optically stimulated intracavity Si or GaAs spacers, which are transparent in the far-IR, placed between the SrTiO₃ laser mirror and the active crystal end face. Such fast quenching of the p-Ge laser might be used to sharpen the trailing edge of the far-IR emission pulse for time-resolved or cavity-ring-down spectroscopic applications. Direct-gap semiconductor spacers might be used as fast, optically controlled intracavity modulators for active mode-locking. This work is described in

"The far-infrared p-Ge laser: Cavity and modulation advances," R. E. Peale, A. V. Muravjov, E. W. Nelson, C. J. Fredricksen, S. Kleckley, S. G. Pavlov, V. N. Shastin, in CLEO-QELS 2002 (OSA Conference Program, Washington DC, 2002), p. 107, and

"Intracavity modulation of THz p-Ge laser gain by interband optical excitation," C. J. Fredricksen, A. V. Muravjov, and R. E. Peale, in Solid State Lasers XIII: Technology and Devices, Proc. SPIE v. 5332 (2004).

Gain improvements

A far-infrared p-type germanium laser with active crystal prepared from ultra pure single-crystal Ge by neutron transmutation doping (NTD) was demonstrated. Calculations show that the high uniformity of Ga acceptor distribution achieved by NTD significantly improves average gain. The stronger ionized impurity scattering due to high compensation in NTD Ge was shown to have insignificant negative impact on the gain at the moderate doping concentrations sufficient for laser operation. Experimentally, this first NTD laser was found to have lower current-density lasing threshold than the best of a number of melt-doped laser crystals studied for comparison.

A controllable, repeatable process such as NTD will allow higher yield of good laser rods per kg of material. Given current prices for melt-doped (~\$3/g) and ultra-high purity Ge crystals (~\$5/g), the yield need only be twice better to make NTD economically preferable. The neutron-irradiation fee at UFTR is \$100/hour, which is negligible compared with other costs of producing a commercial p-Ge laser (primarily cryogenics and specialized electronics). Residual radiation is equivalent to background, so properly aged NTD Ge poses no health risk. High-purity Ge starting material for NTD is commercially available while suitable melt-doped Ge is not a standard commercial item. Publications resulting from this work are listed next.

"Neutron Transmutation Doped Far-infrared p-Ge laser," E. W. Nelson, M. V. Dolguikh, A. V. Muravjov, E. S. Flitsiyan, T. W. Du Bosq, R. E. Peale, S. H. Kleckley, C. J. Fredricksen, W. G. Vernetson, Accepted J. Appl. Physics, April 2004.

"Neutron Transmutation Doped Far-infrared p-Ge laser," E. W. Nelson, E. S. Flitsiyan, A. V. Muravjov, M. V. Dolguikh, R. E. Peale, S. H. Kleckley, W. G. Vernetson, and V. Z. Tsipin in High-Power Fiber and Semiconductor Lasers, M. Fallahi and J. V. Maloney, Eds., Proc. SPIE Vol. 4993, pp. 10-19 (2003).

"Intravalence-band THz laser in selectively-doped semiconductor structure," M. V. Dolguikh, A. V. Muravjov, and R. E. Peale, in Novel In-Plane Semiconductor lasers III, Proc. SPIE v. 5365 (2004),

"Selectively doped germanium THz laser," M. V. Dolguikh, A. V. Muravjov, and R. E. Peale, in Terahertz for Military and Security Applications, edited by R. J. Hwu and D. L. Woolard, Proc. SPIE v. 5411 (2004), and

Patent disclosure, University of Central Florida, April 2004.

Applications development

There is still no significant commercial market for THz devices. One can say that the commercial application for THz lasers has yet to be discovered, despite the rapid progress in THz technology during the past 10 years. One of our objectives was to investigate the suitability of p-Ge lasers for different proposed applications. We have chosen to attend large professional society meetings that cover a range of topics, have a large vendor show, and preferably have some session on THz technology. At the same time, laboratory experiments using the p-Ge laser and a Fourier spectrometer were performed to better define the possibilities.

Suggested use of THz carriers for airborne/satellite THz free-space laser communication seems to be at least 10 years ahead of any real commercial need. Information on possible near-term military need is unavailable to us. Hence, it seems premature to stress this particular application for the p-Ge laser. However, our efforts to make the p-Ge laser compact and light weight generally benefit this potential application. Applicability of the p-Ge laser for satellite communication is discussed in

"Far-IR semiconductor laser for future THz-carrier free-space communications," R. E. Peale, A. V. Muravjov, E. W. Nelson, C. J. Fredricksen, S. G. Pavlov, V.N.Shastin in Free Space Laser Communications Technologies XIV, edited by G. S. Mecherle, Proceedings SPIE 4635, 57-64 (2002).

There has been much recent emphasis on THz bio/chemical sensing for homeland security purposes. However, the THz spectra (as presented at 2003 meetings Photonics West, Aerosense, and CLEO) of biomolecules and chemical agents, including explosives, are not at all encouraging for this application. These spectra lack sharp characteristic absorption features, except in artificially simplified laboratory samples. The absorption that exists is of broad phonon-like character with insignificant differences between compounds. Humidity has a larger effect on the spectra than the spectral differences between different biomolecules. Sharp, characteristic THz absorption lines exist only in the rotational spectra of very small molecules, which are of limited interest.

The interest that does exist in identifying small molecules is mainly in the field of submillimeter-wave astronomy and atmospheric chemistry. A far-IR laser has value as a local oscillator for heterodyne spectroscopy, which is the workhorse method in those

fields. The recently introduced THz QCL operating above liquid nitrogen temperature with mW of output power may become suitable as a solid-state local oscillator for that technique. However, the requirement of cryogenic apparatus to achieve 80 K temperatures maintains a competitive edge for the modern far-IR gas lasers (e.g. Coherent-DEOS) used now. Suitability of the p-Ge laser for molecular spectroscopy is discussed in

"Far-infrared semiconductor laser for molecular spectroscopy," R. E. Peale, A. V. Muravjov, E. W. Nelson, C. J. Fredricksen, S. G. Pavlov, V. N. Shastin, in Laser Applications to Chemical and Environmental Analysis (OSA-TOPS vol. 64, Washington DC, 2002) pp ThD1-1 to ThD1-3.

Another astronomy-based application for p-Ge lasers is calibration of far-infrared spectrometers designed for airborne or satellite platforms. This application was suggested by our first likely customer (see Transactions), who is developing the PACS spectrometer for the ESA Herschel space telescope. The cost of purchasing a p-Ge laser system is beyond their budget, so a rental arrangement has been agreed upon and currently scheduled for July 2003. Reasons behind their interest are that the p-Ge laser has sufficient power in a sufficiently narrow line, which can be easily tuned. Their detectors are helium cooled, so that they have storage dewars on hand and no objection to using cryogenics.

A laboratory application for p-Ge lasers is laser spectroscopy of pulsed expansion gas jets. This was suggested by the inquiry of Prof. John Bevan at Texas A&M University (see Transactions). Prof. Bevan performs THz spectroscopy in expansion jets using harmonically multiplied backwards-wave oscillators (BWO), which are limited to frequencies below 2 THz, while the p-Ge laser tunes up to 4.2 THz. The low duty operation of the p-Ge laser is not an issue because the expansion jets are pulsed already at about 10 Hz.

Non-destructive testing is an application that can benefit from the high output power and high spectral density provided by the p-Ge laser. To test this, we prepared a number of plastic and composite samples and measured their transmission with a p-Ge laser operating narrow line at 109 cm^{-1} and detected with a room temperature Golay cell. Results are given in Table I. Note that the common structural plastic Plexiglass is completely opaque at 109 cm^{-1} . Measurement with a Fourier spectrometer over the $50\text{--}140\text{ cm}^{-1}$ tuning range of the p-Ge laser confirms that this opacity is broad band. Similarly, fiberglass printed-circuit-board substrate is opaque. Silica glass fibers are expected to be opaque. The transparency of polyester resin has not yet been separately determined. The importance of surface quality of the specimen is indicated by the 0% transmittance of single-side polished silicon. Silicon is known to have high far-IR transparency, so this result is attributed to Rayleigh scattering on the rough surface. (This shows that through-wall imaging is unlikely.)

Table I. Sample, sample thickness, and sample transmittance at 109 cm⁻¹.

Material	Thickness (mm)	% Transmittance
Polypropylene (PP)	1.45	72.2
LDPE	3.70	61.1
Polystyrene (PS)	1.86	33.3
Teflon	3.17	66.7
Plexiglass	9.05	0
Plexiglass	3.06	0
HDPE	0.74	44.4
Double layer of Scotch Tape	0.12	27.8
Bumper Sticker	0.15	55.6
Bare Printer Circuit Board	1.55	0
Single Side Polished Si	0.64	0
Plastic drinking straw	2.12	61.1
Teflon with thin layer of grease	3.17	55.6

THz radiation might be suitable for imaging through soils to detect mines. To test this, we measured the transmission spectrum of a soil sample. The unpublished results are presented next. Spectra were collected using a Fourier spectrometer with a Hg arc blackbody source, mylar pellicle beamsplitter, and a 4 K silicon bolometer. Samples of dry sand were placed in a polyethylene cell to obtain a 1 mm long sample path length. "Dry" means that the soil was scooped on a warm-hot day from an exposed surface with little organic matter and without special drying procedure. A microscope image was collected to determine that the characteristic grain size of the sample was ~ 0.25-0.35 mm with only small size variations. Hence, scattering is expected to be strong for wavelengths beyond ~300 micron. Fig. 2 supports this expectation in that the transmittance is zero except beyond 300 microns (below 33 cm⁻¹). New money to continue these soil studies has been obtained through Northrup-Grumman.

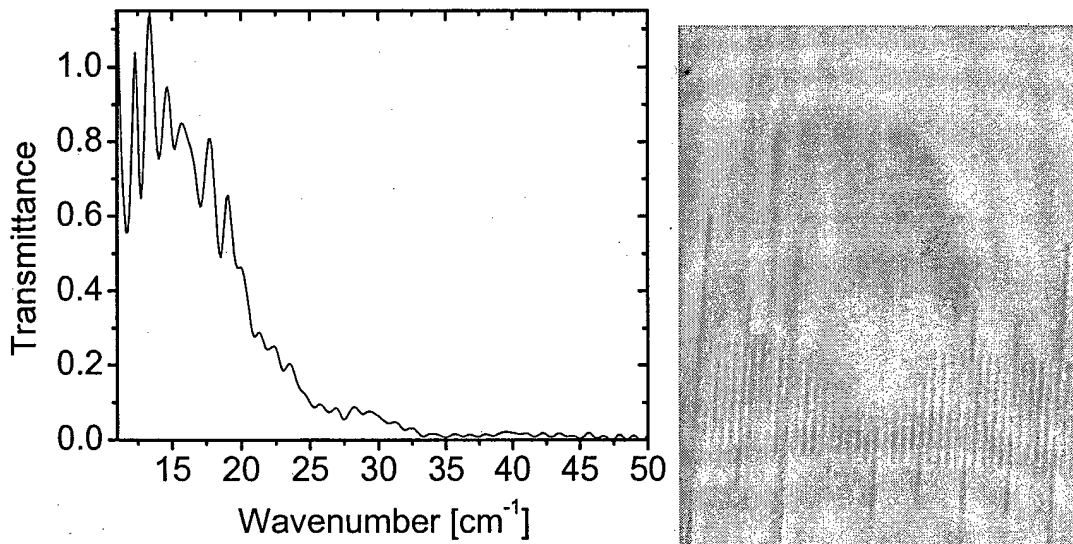


Fig. 2 (left). Transmission spectrum of sand. (right): Microscope image of characteristic soil particle. Major ticks are 100 micron apart. Florida soil is primarily quartz sand having a uniform size distribution and little organic matter.

Potential applications of the p-Ge laser will benefit from a broad range of THz technology improvements. For instance, long pass filters are important components in the detection system to reduce noise from the thermal background radiation. We have discovered a simple way to prepare long pass filters with cut off wavelengths beyond 10 microns by high temperature diffusion of Cu in GaAs. Copper was evaporated to thickness ~ 100 nm on both sides of double-side polished 3 mm thick GaAs samples, then sealed in evacuated quartz ampoules back filled with 250 torr of helium. The ampoules were heated at temperatures between 600 and 1200 C for periods of 15 min to 16 hours, then quenched in water. Infrared spectra were collected using a Fourier spectrometer at 1.7 K sample temperature, 500 to 3500 cm^{-1} range, and 1 cm^{-1} resolution. The well known Cu:GaAs sharp-line absorption spectrum was observed near 1200 cm^{-1} together with a strong photo-ionization band. The latter provides zero transmission for wavelengths shorter than 8 μm . This cutoff shifts to longer wavelengths as diffusion times and temperature increase. The process allows for the simple preparation of mid and far infrared long-pass filters.

"Copper doped GaAs infrared filter for the 8-13 micron atmospheric window," F. R. Ruhge and R. E. Peale, in Infrared Technology and Applications XXX, edited by B. F. Andresen and G. F. Fulop, Proc. SPIE v. 5406 (2004).

Personnel Supported

Zaubertek: Chris Fredricksen, Principal Investigator; Dr. Elena Flitsiyan, Scientist; T. Joseph Mahaney, technician; Forrest Ruhge, research assistant; Melinda A. Wright, Business Manager.

UCF: Dr. Andrei Muravjov, scientist; Dr. Robert E. Peale, professor; Todd Du Bosq, graduate student, Maxim Dolguikh, graduate student.

Subcontractor: Carl Hallberg, Electronics Engineer.

Interactions/Transitions:

Presentations were made at the conferences listed below. Each conference had a large product show, at which material on the p-Ge laser was provided to representatives of laser manufacturers and distributors to explore strategic partnership opportunities. Some follow-up discussion resulted, but no deals so far. Established companies apparently do not perceive a near term market opportunity at present.

- High-Power Fiber and Semiconductor Lasers (Photonics West 03, San Jose)
- Solid State Lasers XII (Photonics West 03, San Jose)
- Chemical and Biological Sensing IV (Aerosense 03, Orlando)
- Laser Systems Technology (Aerosense 03, Orlando)
- CLEO/QELS 2003 (Baltimore)
- OSA Annual Meeting (Orlando)
- Solid State Lasers XII (Photonics West 2003 San Jose)
- High-power fiber and semiconductor lasers (Photonics West 2003 San Jose)
- Laser Systems Technology (Aerosense 2003 Orlando)
- Chemical and Biological Sensing IV (Aerosense 2003 Orlando)
- OSA Optics in the SouthEast 2003 (Orlando)

- Solid State Lasers XIII: Technology and Devices (Photonics West 2004 San Jose)
- Novel in-plane semiconductor lasers III (Photonics West 2004 San Jose)
- Terahertz for Military and Security Applications II (Defense & Security 2004 Orlando)
- Infrared Technology and Applications XXX (Defense & Security 2004)

Zaubertek continues to pursue a low cost marketing strategy of sending informational post cards to leads identified through conference participation. These postcards direct recipients to the Zaubertek website www.zaubertek.com. The rate of phone/mail/email inquiry has been about 2%. Zaubertek is listed in the Physics Today and Laser Focus World buyer's guides. Zaubertek's ZT-100 p-Ge laser is listed in the infrared sources catalog of electro-optics distributor Boston Electronics. Because of technical innovations developed under this contract, Zaubertek is able to offer a p-Ge laser system for a price of about \$45000, down from preaward estimates of \$80000. Official price quotes were provided per request to the following.

1. Dr. Helmut Feuchtgruber, MPI Extraterrestrische Physik, Giessenbachstrasse, Postfach 1312, D-85741 Garching, Germany.
2. Dr. Yunping Wang, Institute of Physics, Chinese Academy of Science, PO Box 603, Beijing 100080, P.R. China
3. Prof. John Bevan, Department of Chemistry, Texas A&M University, College Station TX
4. Dr. Danielle Chamberlin, Electronics Research Lab, Agilent Technologies, 3500 Deer Creek Road MS26L, Palo Alto CA 94304
5. Ken Puzey, Teracom Research Inc., PO Box 163, Essex Junction VT 05453
6. Prof. Ming Bao, Department of Electrical Engineering, UCLA, Los Angeles CA
7. Costel Biloiu, PhD, Researcher, Department of Physics, Hodges Hall, West Virginia University Morgantown WV 26506
8. Dr. Indra Mukhopadhyay, Dakota State University, Madison SD 57042
9. Tania Silver, University of Wollongong, Australia
10. Haim Grebel, New Jersey Inst of Tech, Newark NJ 07102
11. Andrey Chebotarev, Opthus, Inc., Stanford CA 94309

Of this list, only the first seems likely to result in near-term business. A five-day trip to the Max Planck Institute for Extraterrestrial Research in Garching Germany is planned for July 2004. Zaubertek will deliver and operate a ZT100 p-Ge laser with intracavity wavelength selection to calibrate the PACS spectrometer of the ESA Herschel Space Telescope.