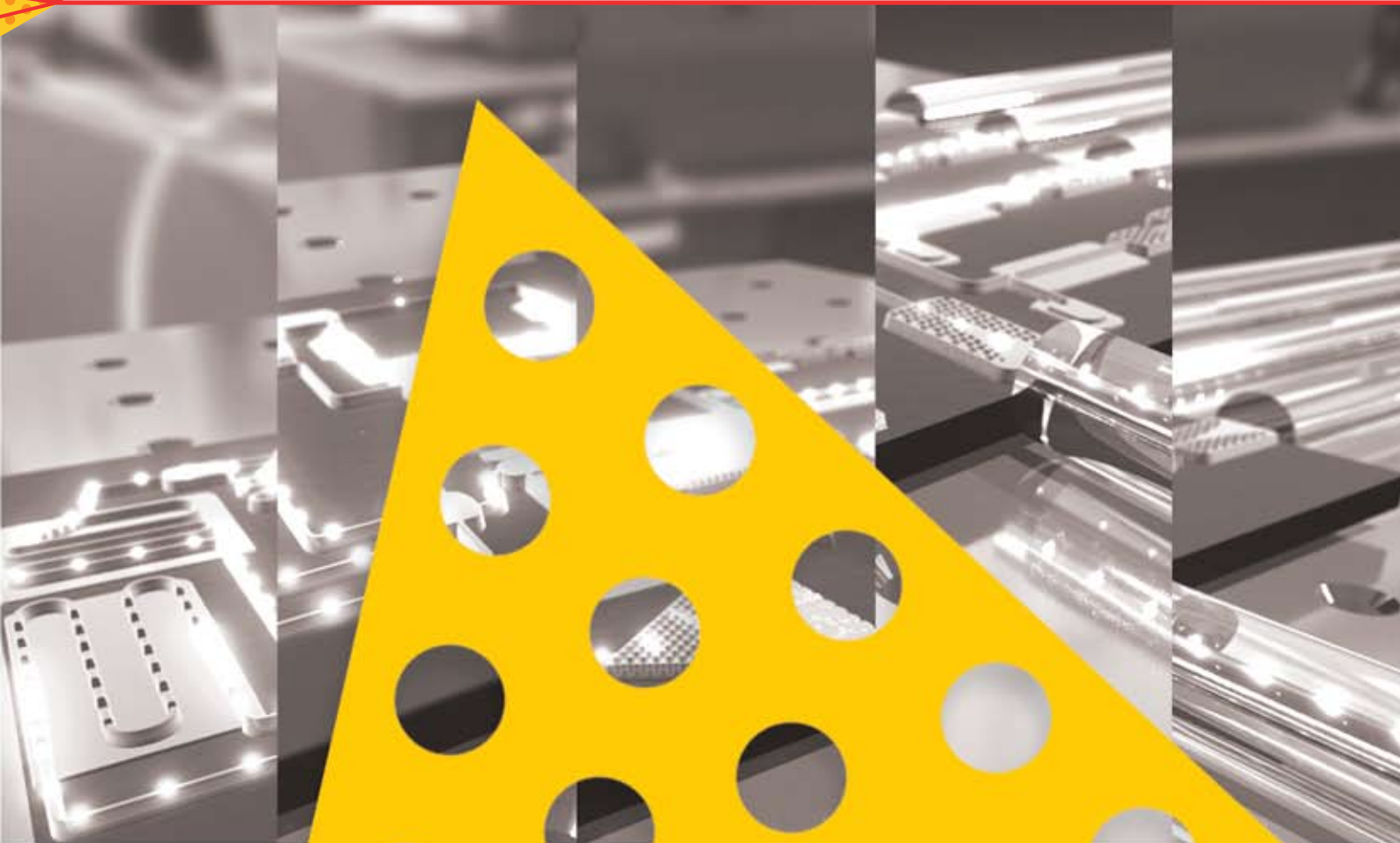




# CUDOS

The Centre for Ultrahigh bandwidth Devices for Optical Systems (CUDOS)  
An Australian Research Council Centre of Excellence



## Annual Report 2007



## ◀ Barry Luther-Davies

### Research Achievements

#### Nonlinear Materials and Planar Structures

Team:

Barry Luther-Davies  
Dukyong Choi  
Steve Madden  
Congji Zha (until May)  
Rongping Wang  
Andrei Rode  
Douglas Bulla  
Maryla Krolikowska  
Anita Smith  
Darren Freeman  
Amrita Prasad  
Ting Han

#### Overview

Our program underpins CUDOS-wide efforts to demonstrate high performance all-optical processors exploiting the third order nonlinearity of chalcogenide glasses. Our research includes the fabrication of novel glasses; studies of their basic physical and optical properties; film production and characterization; and film processing to create low loss optical waveguides and photonic crystals. Structures are supplied to the flagship projects “Chalcogenide Integrated Circuits” and “Compact Optical Switch”.

#### Bulk chalcogenide glasses and their properties

Chalcogenide glasses comprise a wide range of amorphous materials containing the chalcogen elements S, Se and Te compounded with network forming elements such as As, Ge, Si, etc. We have chosen these materials because they have high refractive index, large third order nonlinearity, and photosensitivity whilst being free of absorption across the whole of the near- and mid-infra-red. A challenge with chalcogenides arises from their relatively weak chemical bonding which results in low melting temperatures and structural instability. This leads to a range of exotic phenomena such as quasi-crystallization and enhanced photosensitivity even at wavelengths well beyond their band edge.

We have continued to study glasses in the Ge-As-Se system to identify compositions with the best combination of physical properties for all-optical processing. Of interest is the optical loss; the refractive index; the third order nonlinearity; the optical gap; the glass transition temperature ( $T_g$ ); the glass structure and its stability. A wide range of Ge-As-Se bulk glasses have been fabricated and analysed using techniques such as differential scanning calorimetry (DSC); Raman spectroscopy; x-ray photoelectron spectroscopy (XPS); Vis-IR spectrophotometry; photo-thermal deflection spectrometry (PDS); and prism coupling to obtain values for the linear and z-scan the nonlinear refractive indices; etc.

The glass properties have been correlated with glass structure, characterized by the mean co-ordination number (MCN) calculated from the sum of the products of the valency of the constituent atoms times their elemental concentrations. Using this measure glasses with low MCN are generally over-stoichiometric in Se and are characterised as having a “floppy” glass network containing large numbers of Se-Se chains or rings; whilst those with the

Barry Luther-Davies is a Federation Fellow and Professor of Laser Physics at the Australian National University with 37 years research experience in the diverse areas such as lasers, laser-matter interaction physics, photonics, optical materials and nonlinear optics. He completed a BSc in Electronics and PhD in Laser Physics from the University of Southampton, UK.

Barry oversees the Centre’s work at ANU fabricating planar optical waveguide devices and photonic crystals in chalcogenide glasses and is also science leader of the CUDOS flagship project **Compact Optical Switch in 2-D Photonic Crystal** which combines the skills of researchers at ANU, The University of Sydney and the University of Technology Sydney. His broad experience contributes to all aspects of the CUDOS projects that spans materials science; film deposition and patterning; optical characterization; and device design.

Barry is a Fellow of the Australian Institute of Physics, the Optical Society of America and the Australian Academy for Technological Sciences and Engineering. He was awarded the Pawsey Medal of the Australian Academy of Science in 1986 for his contribution to laser-plasma interaction physics. He is currently a topical editor for the Journal of the Optical Society of America-B.

### Roles and responsibilities within the Centre

Group Leader: Laser Physics Centre, RSPHysSE, ANU;

Science Leader: Compact Optical Switch in 2-D Photonic Crystal.

### Key areas of research activity

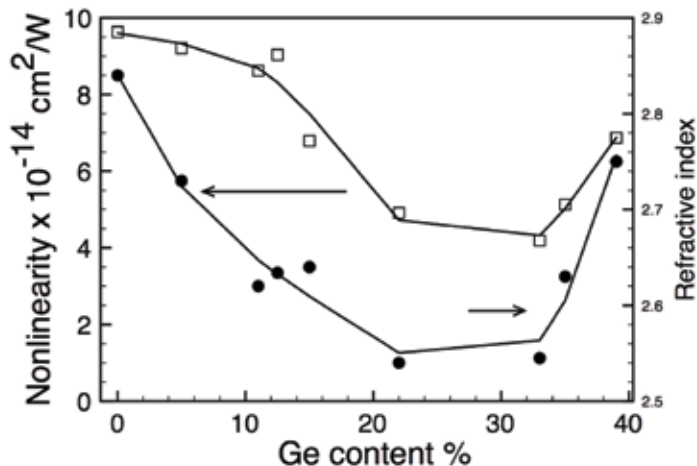
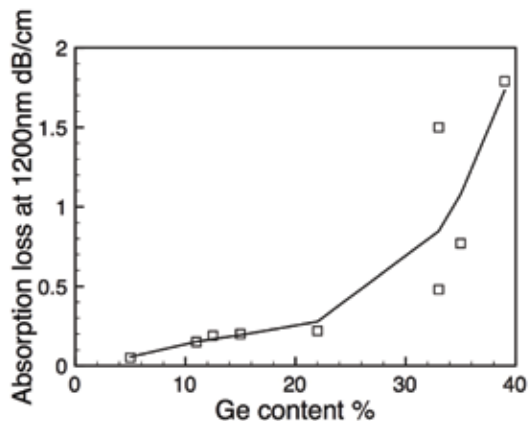
Barry Luther-Davies oversees research into properties of new chalcogenide glasses; thin chalcogenide film production by pulsed laser ablation; film processing for waveguide and photonic crystals; device and materials testing. Devices are supplied to the flagship projects Integrated Chalcogenide Glass All-optical Regenerator and Compact Optical Switch in a 2-D Photonic Crystal.

highest MCN generally are over-stoichiometric in Ge and will contain large numbers of homopolar (Ge-Ge) or defect bonds (Ge-As). Of particular interest are so-called intermediate phase glasses with MCN around 2.4-2.5 in this glass system that have been shown to display some unique physical properties including a near absence of the latent heat of melting. This means that the glass configuration in the solid is the same as that in the corresponding molten phase. This intermediate phase results in stress-free but rigid glass networks.

Some of the results of our studies are as follows:

- We have identified features in the Raman spectra of this glass system that indicate both the appearance of the intermediate phase glasses for MCN around 2.5 and the transition to stressed rigid phase glasses for MCN=2.67;
- By de-convolving the Raman spectra into individual peaks we identified a growth in the number of defect bonds in the glass and found it correlates with increasing optical loss determined by PDS. This result shows that high MCN (high Ge content) glasses are too lossy for our applications: (figure 1);
- A generally monotonic increase in T<sub>g</sub> occurs with increasing MCN (provided regions of known phase separation are avoided);
- Qualitatively the relation between nonlinear refractive index and linear refractive index obeys the semi-empirical Miller's rule, namely high index of refraction implies high nonlinearity. Of the glasses tested those with 2.6 > MCN > 2.9 had the highest linear and nonlinear indices (figure 2).
- More detailed studies of the relation between optical nonlinearity and Tauc gap confirmed these glasses conform to the model proposed by Sheik-Bahae et al. 1 for direct band gap semiconductors which predicts resonant enhancement of the optical nonlinearity near the two photon absorption transition.
- XPS spectra have been used to probe the local bond structure of these glasses. We have found for example that whilst GeSe<sub>4/2</sub> tetrahedral and AsSe<sub>3/2</sub> pyramidal units as well as Se trimers decrease within increasing mean coordination numbers at the same time most defect bonds increase in agreement with Raman data. However, whilst the appearance of Se trimers is reasonable in Se-rich samples, they never vanish even in Se-poor samples.

Our conclusion from this work is that the best combination of properties is obtained using glasses with around 11% Ge which also happens to correspond to an intermediate phase glass.



**Fig 2: Linear and nonlinear refractive index values vs Ge content for Ge-As-Se glasses**

### Relevant Papers and Presentations

1. Zha, Congji; Wang, Rongping; Smith, Anita; Prasad, Amrita; Jarvis, Ruth A.; Luther-Davies, Barry. Optical properties and structural correlations of GeAsSe chalcogenide glasses. *Journal of Materials Science: Materials in Electronics* (2007), 18(Suppl. 1), S389-S392.
2. Wang, R. P.; Zha, C. J.; Rode, A. V.; Madden, S. J.; Luther-Davies, B.. Thermal characterization of Ge-As-Se glasses by differential scanning calorimetry. *Journal of Materials Science: Materials in Electronics* (2007), 18 (Suppl. 1), S419-S422.
3. Zha, Congji; Smith, Anita; Prasad, Amrita; Wang, Rongping; Madden, Steve; Luther-Davies, Barry. Properties and structure of Ag-doped As<sub>2</sub>Se<sub>3</sub> glasses. *Journal of Nonlinear Optical Physics & Materials* (2007), 16(1), 49-57.
4. Wang, Rong-Ping; Rode, Andrei; Madden, Steve; Luther-Davies, Barry. Physical aging of arsenic trisulfide thick films and bulk materials. *Journal of the American Ceramic Society* (2007), 90(4), 1269-1271.
5. Prasad, A., Zha, C., Smith A., Madden S., Bulla D., Rode A., Wang R., and Luther-Davies B., Characteristics of Ge-As-Se chalcogenide glasses and films Proceedings of COIN-ACOFT, Melbourne 24-27 June (2007). [ISBN 978-0-9775657-3-3, IEEE catalog number 07EX1889C].

**Fig 1: Absorption loss at 1200nm for a series of Ge-As-Se glasses is compared with the number of defect bonds determined from analysis of Raman spectra**

1 M. Sheik-Bahae, D. Hutchins, D. Hagan, E. Van Stryland, Dispersion of Bound Electronic Nonlinear Refraction in Solids *IEEE J. Quant. Electr.* 27, 1296 (2001).



- Barry Luther-Davies, Congji Zha\*, Amrita Prasad, and Anita Smith Optical Properties and Structural Transitions in Ge-As-Se Glasses in Conference on Lasers and Electro-Optics/Quantum Electronics and Laser Science Conference and Photonic Applications Systems Technologies 2007 Technical Digest (Optical Society of America, Washington, DC, 2007), CMGG7, ISBN: 1-55752-834-9.
- C. Zha, B. Luther-Davies, R Wang S Madden, High Optical nonlinear Ag-doped As<sub>2</sub>Se<sub>3</sub> glasses, Proceedings of COIN/ACOFT IEEE catlog #07EX1889C (2007). ISBN 978-0-9775657-3-3

## Film properties and processing

Previously we concentrated on the use of pulsed laser deposition to produce high quality thin chalcogenide films for device fabrication. However for most of 2007 our pulsed laser deposition (PLD) system was out of action due to a major hardware failure at the end of 2006 and hence we have changed to simple thermal evaporation (TE) to produce films. This poses a number of difficulties. In general, TE films have a different stoichiometry from the bulk glass since chalcogenides often phase separate on melting, resulting in species with different volatility being present in the melt. A secondary effect is that phase separation causes violent boiling of the melt, with the resultant splashing of material from the boat heavily contaminating the films. A final issue with TE (and PLD) material is that the bond structure of the glasses is markedly different from the bulk glass because both deposition methods lead to film formation in non-equilibrium conditions. We reported last year for example that AMTIR-1 films deposited by PLD had markedly higher refractive index than the bulk (2.69 compared with 2.54) whilst in general As<sub>2</sub>S<sub>3</sub> films deposited by TE have indices of <2.3 compared with the bulk value of 2.43. Whilst it is possible to anneal these films post deposition to change their indices to bulk values this is generally accompanied by shrinkage or expansion of the film resulting in stress that causes problems during device processing.

We have developed ways of overcoming these problems. For example, film contamination from splashing has been eliminated by using baffled boats normally reserved for SiO deposition. Evaporation rate has a strong effect on the film stoichiometry and appropriate choice of boat temperature can result in films with relatively small differences in stoichiometry from the bulk. However changes in stoichiometry cannot be totally eliminated and in fact for Ge-As-Se glasses the film stoichiometry changes with time during deposition. The best we have achieved so far is to deposit films that are slightly Se-poor compared with the bulk glass, with Ge and As both correspondingly richer. In inappropriate deposition conditions, however, the relative content of Ge and As can deviate strongly (by up to 40%) from the bulk composition, one being richer and the other poorer. Hence we conclude that adequate control of stoichiometry can be obtained from TE especially using glasses whose properties do not depend strongly on composition. Films deposited in optimised conditions are virtually defect-free and excellent for subsequent device fabrication.

The difference in bond structure of the films compared with the bulk glasses has posed a range of problems for devices. The film stability can be poor with bond structure changing on exposure to heat or illumination with near band edge light. The films can be oversensitive to chemical attack, and stress concentration at defects after annealing can lead to film cracking during device processing.

Ideally one would like to be able to deposit films whose properties were near those of the bulk and which can be processed by annealing to relax their bond structure without changing the film

properties. We have therefore examined annealing of films deposited from glasses with compositions close to the intermediate phase glass composition identified above as the best for all-optical processing. Annealing was carried out in good vacuum (<10<sup>-5</sup> torr) to prevent oxidization of the film surface. Interestingly we find that glasses near the intermediate phase form films whose refractive index differs insignificantly from the bulk glass. This contrasts strongly with non-intermediate phase glasses such as AMTIR-1. Furthermore upon annealing the intermediate phase glasses do not change in refractive index nor in thickness within the accuracy of our measurements (figure 4). Finally these glasses can also be annealed well above their glass transition temperatures without any deterioration of their cosmetic quality. This is a very interesting observation since it suggests a new and important property for intermediate phase glasses, namely that their physical properties remain bulk-like even when produced in film form in non-equilibrium conditions. XPS studies will now be performed to identify if the same bond rearrangements occur during annealing as seen from our studies of AMTIR-1.

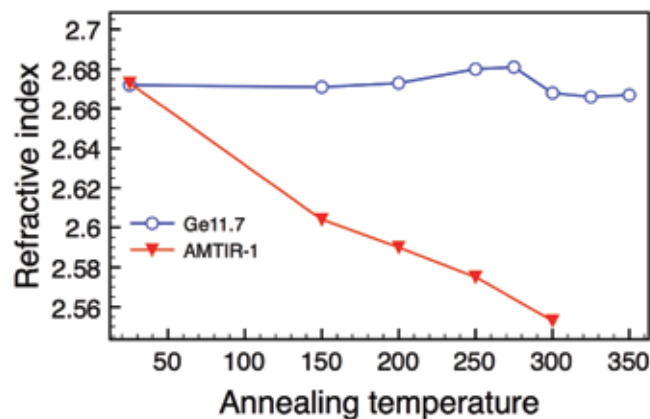


Fig 4: Change in refractive index upon annealing for Ge11.7 film in the intermediate glass phase compared with AMTIR-1 film from the stressed rigid glass phase

## Relevant Publications and Presentations

- Choi, Duk-Yong; Madden, Steve; Rode, Andrei; Wang, Rongping; Luther-Davies, Barry. Nanoscale phase separation in ultrafast pulsed laser deposited arsenic trisulfide (As<sub>2</sub>S<sub>3</sub>) films and its effect on plasma etching. *Journal of Applied Physics* (2007), 102(8), 083532/1-083532/5
- Jarvis, R. A.; Wang, R. P.; Rode, A. V.; Zha, C.; Luther-Davies, B.. Thin film deposition of Ge<sub>33</sub>As<sub>12</sub>Se<sub>55</sub> by pulsed laser deposition and thermal evaporation: Comparison of properties. *Journal of Non-Crystalline Solids* (2007), 353(8-10), 947-949.
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- Wang, R. P.; Choi, D. Y.; Rode, A. V.; Madden, S. J.; Luther-Davies, B.. Rebonding of Se to As and Ge in Ge<sub>33</sub>As<sub>12</sub>Se<sub>55</sub> films upon thermal annealing: Evidence from x-ray photoelectron spectra investigations. *Journal of Applied Physics* (2007), 101(11), 113517/1-113517/4.
- Duk-Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Barry Luther-Davies, Plasma etching of As<sub>2</sub>S<sub>3</sub> films for optical waveguides, *J. Non-Cryst. Solids* (accepted).

13. R.P.Wang, A.V.Rode, D.Y.Choi, and B. Luther-Davies, Surface Oxidation of  $\text{Ge}_{33}\text{As}_{12}\text{Se}_{55}$  films, J. Am. Ceram. Soc. (accepted).
14. Duk-Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Adrian Ankiewicz, Barry Luther-Davies, Etched Surface Roughness in  $\text{As}_2\text{S}_3$  films: Its Origin, Improvement and Effect on Scattering Loss in optical Waveguides, IEEE-TNANO (in press)

## Device fabrication

There are three routes to reducing the operating power of all-optical devices based on chalcogenide waveguides: to use glasses with enhanced optical nonlinearity; to reduce the area of the propagating modes; and increase to effective propagation length in the device. The effective propagation length is limited by linear or nonlinear optical losses. In the case of the chalcogenides nonlinear losses are generally negligible and two sources of optical losses generally determine the effective propagation loss: absorption in the glass and scattering of light from the interfaces. In most glasses absorption losses are small and hence loss is dominated by scattering from the interfaces which increases markedly as the mode area is reduced in high index contrast waveguides. Thus large propagation lengths and ultra-small mode volumes are unlikely to be achieved simultaneously.

Up until now the best optical losses we obtained for our rib waveguides fabricated in  $\text{As}_2\text{S}_3$  had been 0.25dB/cm. In 2006, however, we introduced several new approaches to device processing that led to a significant reduction in the roughness of the etched surfaces down to about 1.5nm RMS. During this year therefore we fabricated and characterised our first devices using these new approaches. A new photo-mask was employed which allowed the production of waveguides up to about 24cm in length – a factor of four longer than previously possible. Using our new approach a record low optical loss of 0.05dB/cm at 1550 nm was achieved with mode areas of about  $7\mu\text{m}^2$ , a five fold-reduction on our previous best result. With a smaller mode volume  $\approx 2.5\mu\text{m}^2$  losses of 0.17 dB/cm were obtained. These waveguides had nonlinear parameters of  $1700(\text{W}\cdot\text{km})^{-1}$  and  $4700(\text{W}\cdot\text{km})^{-1}$  respectively. They have been used in experiments on wavelength conversion up to 80GB/s reported elsewhere.

In addition to fabricating waveguides in  $\text{As}_2\text{S}_3$  we fabricated our first structures in  $\text{Ge}_{11}\text{As}_{22}\text{Se}_{67}$  glass. The initial results on these waveguides were encouraging with losses below 0.25 dB/cm; mode areas around  $4\mu\text{m}^2$  and a resulting nonlinear parameter of about  $10500(\text{W}\cdot\text{km})^{-1}$ . These waveguides were used for tests of self-phase modulation using psec pulses achieving more than 20 radians of nonlinear phase change. From modelling of the SPM results we deduced the nonlinear figure of merit of the waveguides ( $n_2/\beta\lambda$ ) was around 60 emphasizing that two-photon absorption is very small.

## Relevant Publications and Presentations

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16. Choi, D.-Y., Madden, S., Rode, A., Wang, R., Luther-Davies, B. Fabrication of low loss  $\text{Ge}_{33}\text{As}_{12}\text{Se}_{55}$  (AMTIR-1) planar waveguides (2007) Applied Physics Letters, 91 (1), p. 011115.
17. Choi, D.-Y., Madden, S., Rode, A., Wang, R., Luther-Davies, B. Nanoscale phase separation in ultrafast pulsed laser

deposited arsenic trisulfide ( $\text{As}_2\text{S}_3$ ) films and its effect on plasma etching (2007) Journal of Applied Physics, 102 (8), art. no. 083532, .

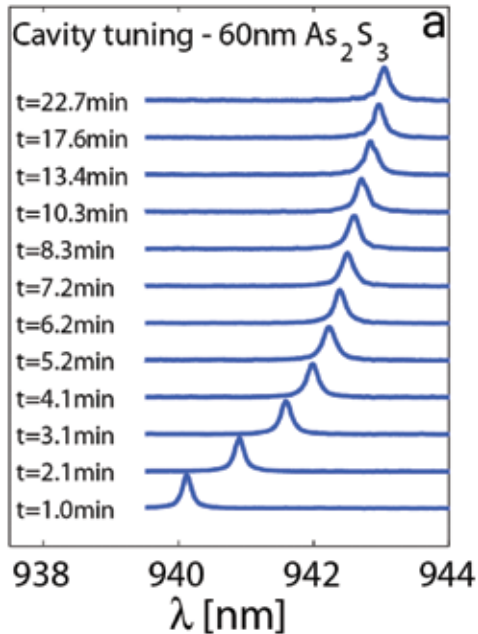
18. Choi, D.-Y., Madden, S., Rode, A., Wang, R., Luther-Davies, B., Baker, N.J., Eggleton, B.J. Integrated shadow mask for sampled Bragg gratings in chalcogenide ( $\text{As}_2\text{S}_3$ ) planar waveguides (2007) Optics Express, 15 (12), pp. 7708-7712.
19. Finsterbusch, K., Baker, N.J., Ta'eed, V.G., Eggleton, B.J., Choi, D.-Y., Madden, S., Luther-Davies, B. Higher-order mode grating devices in  $\text{As}_2\text{S}_3$  chalcogenide glass rib waveguides (2007) Journal of the Optical Society of America B: Optical Physics, 24 (6), pp. 1283-1290
20. DukYong Choi, Steve Madden, Andrei Rode, Rongping Wang, Barry Luther-Davies, Neil J. Baker, Benjamin J. Eggleton, Novel Shadow Mask Structure for Sampled Bragg Gratings in Chalcogenide ( $\text{As}_2\text{S}_3$ ) waveguide, Technical digest of CLEO/QUEL2007, Baltimore 6-11 May (2007). [ISBN 1-55752-834-9, IEEE catalog number 06EX1468C].
21. Duk-Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Barry Luther-Davies, Neil Baker, Benjamin Eggleton, High Quality Comb Filters in Chalcogenide Rib Waveguides, Proceedings of COIN-ACOFT, Melbourne 24-27 June (2007). [ISBN 978-0-9775657-3-3, IEEE catalog number 07EX1889C].
22. Duk-Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Barry Luther-Davies, Fabrication Process Development for  $\text{As}_2\text{S}_3$  Planar Waveguides using Standard Semiconductor Processing, Proceedings of COIN-ACOFT, Melbourne 24-27 June (2007). [ISBN 978-0-9775657-3-3, IEEE catalog number 07EX1889C].
23. Duk Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Barry Luther-Davies, „Advanced processing methods for  $\text{As}_2\text{S}_3$  Waveguide Fabrication, COMMAD,06 Proceedings of the 2006 Conference on Optoelectronic Materials and Devices, Perth, Australia, 6-8 December 2006. [ISBN 1-4244-0578-5, IEEE catalog number 06EX1505C]
24. Duk-Yong Choi, Steve Madden, Andrei Rode, Rongping Wang, Douglas Bulla, Barry Luther-Davies, Fabrication of  $\text{As}_2\text{S}_3$  Planar Waveguides with Very Low Propagation Loss, Proceedings of IEEE Lasers and Electro-Optics Society 2007.

## Photonic crystals

Work on photonic crystals produced by focussed ion beam milling has been sporadic this year due to the student involved focused on finalizing his PhD thesis. Future fabrication of photonic crystals will almost certainly be switched to more conventional electron beam patterning and ICP etching following the installation of the new Raith 150 e-beam writer at ANU at the end of the year.

We have engaged with researchers at Stanford University who are developing photonic crystal micro-cavities in GaAs membranes containing InAs quantum dot emitters for applications in quantum information processing. The performance of these devices relies on the possibility to post tune the wavelength of the quantum dots and PC cavities that can become detuned due to errors during fabrication. The purpose of this project was to demonstrate that photosensitivity of a chalcogenide glass layer deposited on top of the GaAs membrane could be used to as a local tuning mechanism.  $\text{As}_2\text{S}_3$  layers from 30-100nm thick were thermally-deposited onto patterned GaAs membranes so that the flux of material struck the sample close to normal incidence to minimise coating the side of the holes. The samples were annealed then loaded into a cryostat and the emission wavelength from the

quantum dots inside the resonators monitored as the refractive index of the chalcogenide was tuned by exposure to 543nm laser light. Whilst the presence of the  $\text{As}_2\text{S}_3$  degraded the Q of the resonators somewhat (between 5 and 30% for the 60 and 30nm thick films from values around 9000), the cavity resonance was successfully tuned over 3nm.

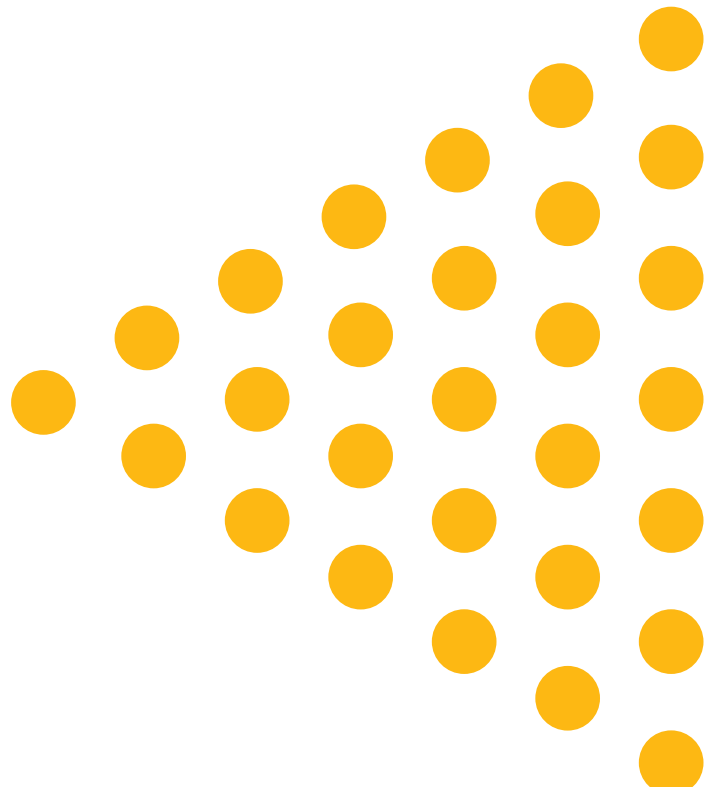


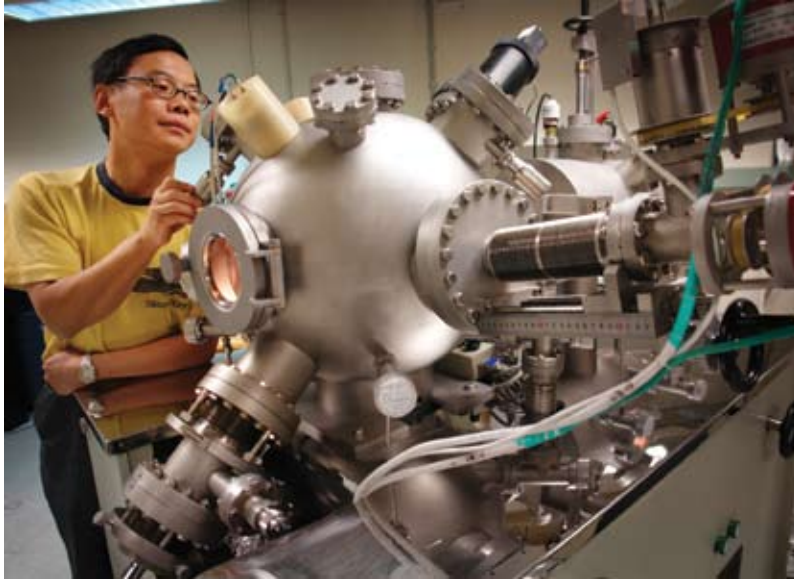
**Demonstration of post tuning of a PC cavity in GaAs membrane containing InAs quantum dots using the photosensitivity of a 60nm thick  $\text{As}_2\text{S}_3$  layer coated onto the membrane.**

Collaboration has continued with the University of Melbourne on the characterization of the optical properties of annular arrays of holes milled into metal films and with the group of the group of Hiroaki Misawa from Hokkaido University on femto-second nano-structuring structuring of bulk materials.

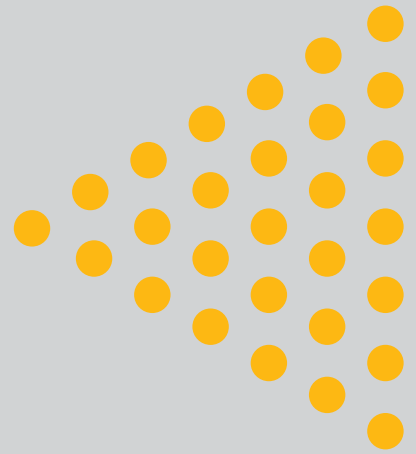
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26. Smith, Cameron L. C.; Wu, Darran K. C.; Lee, Michael W.; Monat, Christelle; Tomljenovic-Hanic, Snjezana; Grillet, Christian; Eggleton, Benjamin J.; Freeman, Darren; Ruan, Yinlan; Madden, Steve; Luther-Davies, Barry; Giessen, Harald; Lee, Yong-Hee. Microfluidic photonic crystal double heterostructures. Applied Physics Letters (2007), 91(12), 121103/1-121103/3.
27. Orbons, Shannon M.; Roberts, Ann; Jamieson, David N.; Haftel, Michael I.; Schlockermann, Carl; Freeman, Darren; Luther-Davies, Barry. Extraordinary optical transmission with coaxial apertures. Applied Physics Letters (2007), 90(25), 251107/1-251107/3.
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The new pulsed Laser Deposition system installed at ANU



## Facility Development

Three major pieces of hardware were funded in 2007 that will impact on the CUDOS program in 2008 and beyond. The first of these was a new high temperature pulsed laser deposition facility funded by the ANU's Major Equipment Committee. This apparatus will address an CUDOS goal stated in the initial CUDOS proposal to deposit novel photonic materials including crystalline materials in addition to chalcogenide glasses. The apparatus consists of a high vacuum chamber, substrate heater capable of operation above 900C; target carousel and RHEED system for monitoring epitaxial growth of crystalline films. The PLD source in this case is a KrF excimer laser (Lumonics IPEX). The final commissioning of this system will take place in early 2008.

### New pulsed Laser Deposition system installed at ANU

A K&S (ADT) 7100 advanced dicing saw has been acquired for wafer dicing. The system offers an indexing accuracy of  $\approx 1 \mu\text{m}$  and will be used for separating dies on our current masks and tested for end face dicing. Installation is planned in the first quarter of 2008.

### New K&S 7100 dicing saw

The final equipment funded by a successful LIEF grant in 2007 is a new Raith 150 electron beam lithography system which is now installed in a clean room facility supporting the ANU activity in NCRIS. The system is operational although some problems with environmental stability need to be addressed before the system can be used at its full potential. NCRIS funding in 2008 will in addition fund a new cluster tool including RIE and PECVD system; a new ion beam mill; and a tool for nano-imprint lithography.



The new K&S 7100 dicing saw

