MINERVA project newsletter #5

June 2015

Welcome to the fifth MINERVA project newsletter!

This has been a busy period for the project, and there are several exciting areas of progress to report. This newsletter provides an update on the zirconium fluoride fibre supercontinuum sources being developed at NKT Photonics, which have been optimised in line with requirements from the bio-photonic experts in the project. There is a description of the crystal growing technology at BBT, the world leaders in the production of calomel: an exciting material for mid-IR photonics. Finally there is some more information on the chalcogenide fibre optic development at University of Nottingham (UK) in collaboration with the Technical University of Denmark which led to the longest wavelength supercontinuum generation ever recorded. An excellent example of simulation and experimental scientists working together!

There is much more information available from the project website (www.minerva-project.eu). For any other questions, further contact info is given below.

University of Nottingham fibre fab

The Mid-Infrared Photonics Group at the University of Nottingham (UK), led by Prof. Angela Seddon has world-class facilities for mid-IR photonics including a Class 10000 cleanroom housing a customised Heathway draw-tower.

In MINERVA, the group is fabricating new, highly transparent mid-IR optical fibres for ultra-low loss transmission as well as specially designed fibres for new broadband and narrowband, bright mid-IR light sources. See pg 4 for an update on the SCG fibres.

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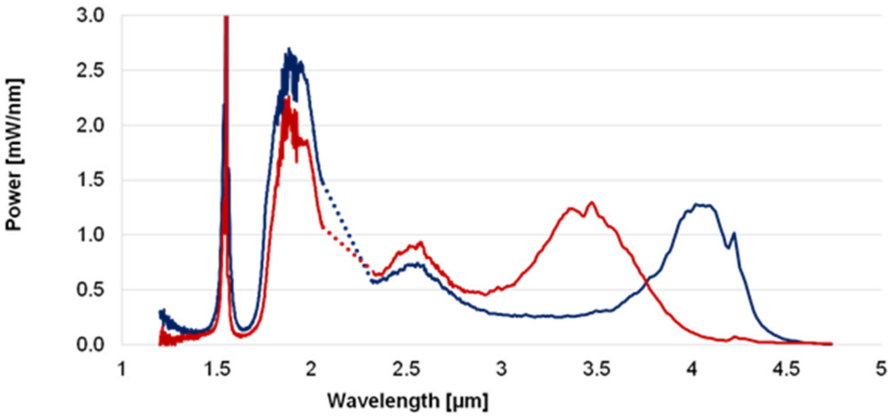
MINERVA improved medical diagnostics

Optimised ZrF_4 mid-IR supercontinuum sources for spectroscopy

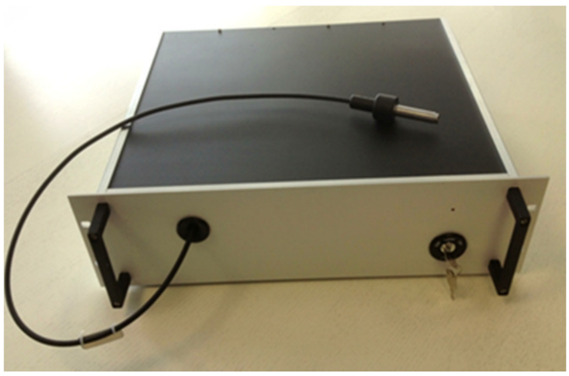
NKT Photonics the power of light

It is well known that spectroscopy in the mid-IR region can give access to a wealth of information on the chemical composition of a sample. Indeed this fact is the central theme of the MINERVA project. However, access to this information has until now been difficult because of the lack of bright broadband light sources in the mid-IR region. In MINERVA it is the goal of NKT Photonics to develop supercontinuum sources which cover the 1.8-4.5 µm region to supplement the existing line of visible to near-IR supercontinuum sources which NKT already sells and which are commonly applied in high-end fluorescence microscopes.

In the first 2½ years of the MINERVA project NKT has already developed several ground-breaking mid-IR supercontinuum sources with output power of up to 2.5 W. These sources are more than a million times brighter than most thermal light sources and even brighter than a synchrotron! NKT has shown the potential of zirconium fluoride-based systems by setting a new record for the longest wavelength supercontinuum generated at 4.75 µm. [See P. Moselund et al., "Highly Stable, All-fiber, High Power ZBLAN Supercontinuum Source Reaching 4.75 µm used for Nanosecond mid-IR Spectroscopy," in Advanced Solid-State Lasers Congress, G. Huber and P. Moulton, eds., OSA Technical Digest, paper JTh5A.9 (2013).] However, the chemometric specialists in MINERVA found that the main region of interest was the spectrum from 2.5-3.8 µm, so NKT has also shown how the power in the output spectrum can be shifted down in wavelength by altering the design of the non-linear fibre. These first supercontinuum sources are already at work in the development of the “MINERVA Lite” microscopy set-up which will soon be applied to bio-sample imaging.



Left: Output spectra of the two MINERVA NKT source designs. [Blue: original source. Red: power shifted down to region of interest.]



Right: Photo of the prototype packaged source.

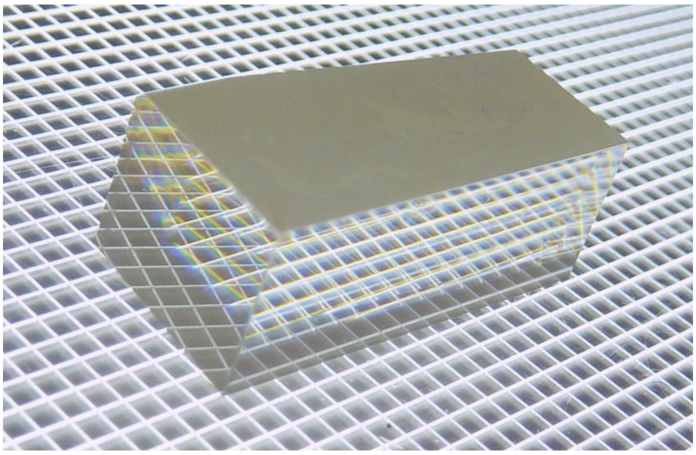
Meanwhile NKT is pushing onward in the development of the mid-IR supercontinuum sources. The initial sources were based on rather long pulse nanosecond pump lasers with relatively low pulse repetition frequencies. This made the sources incompatible with most of the Fourier transform spectrometers (FTIRs) that many researchers use in the mid-IR region. In addition the low repetition rate made it time consuming to counter any noise in the source by averaging over many pulses. Within the MINERVA project, NKT is therefore now developing sources based on much shorter pump pulses and with higher repetition rate in order to reduce noise and make the sources compatible with standard FTIRs.

For more info contact Dr. Peter Moselund: pmm@nktphotonics.com

Mercurous halides: unique optical materials for the infra-red

Mercurous halide (chloride, bromide and iodide) single crystals are highly promising infra-red optical materials for prospective high-tech applications ranging from satellites and tracking systems to medical diagnostics. They find a role in a great number of optical components: polarisation optics, acousto-optic tuneable filters (AOTFs), infrared cameras, modulators, delay lines, microwave components etc.

In MINERVA, calomel (mercurous chloride, Hg_2Cl_2) single crystals have been selected as a basis for AOTFs in the mid-IR (MIR). Calomel is one of the few materials that supports the anisotropic acousto-optic (AO) interaction and exhibits good optical transmission from the visible region to 20 µm with very low shear acoustic wave velocity and high birefringence.



Polished calomel AOTF substrate

BBT is a world leader in the growth and processing of calomel single crystals with excellent AO properties and is a key member of the MINERVA consortium. At the beginning of the project, production technology enabled the growth of calomel crystal boules with a diameter of ~28 mm and length of ~55 mm. Through MINERVA, new technology is being developed to enable the growth of cylindrical boules up to 35 mm diameter, which is necessary for the manufacture of a new design of AOTF proposed by G&H.

The calomel crystal growth process is highly demanding, difficult and complex, especially for large boules. The target of 35 mm represents a huge advance in the state-of-the-art and would be a major achievement for BBT and the project.

The growing process is powered by a dynamic temperature field and corresponding axial and radial temperature gradients. The whole process has to be carefully maintained within narrow physical condition limits. All the equipment and accessories have had to be developed by BBT and adjusted to the specific conditions for growing of the 35 mm diameter crystals, including the temperature controllers. These controllers are equipped with brand new cultivation programs capable of handling the bigger material mass. A total of six crystallizers will be built within the project. Currently, four units are operational and tested.

Growing calomel crystal at BBT (diameter 28 mm)

Calomel crystals are grown using the physical vapour transport (PVT) method. The mass transport of calomel vapour from the source to the crystal seed is realized in a sealed ampoule at the saturated vapour pressure and is driven by well-controlled axial and radial temperature gradients under convective-diffusive conditions. The crystal growth can occur in a broad temperature interval from 120°C to 525°C, but in practice, crystal growth proceeds at temperatures from 320°C to 480°C at a growth rate of 1 to 12 mm/day, depending on the particular growth temperature, temperature gradients, crystal diameter, crystallographic orientation, and distance of the crystal growth phase interface from the source. An important limiting factor of single crystal growth is the width of the metastable region, i.e. the ability of the gas phase to become under-cooled. The growth temperature must be controlled within the corresponding limits to avoid parasitic crystal nucleation. Decreasing the temperature also leads to a decrease in the nucleation rate of calomel and a delay in the establishment of the thermodynamic equilibrium. To avoid internal stresses and plastic deformation arising during growth, which can greatly reduce the quality of single crystals, it is advantageous to grow crystals in the lower part of the possible temperature window.

Based on its advances in MINERVA, BBT plans to use this technology to confirm its world-leading position, and establish itself as the only supplier in the world capable of growing and supplying large calomel crystals for scientific and industrial purposes.

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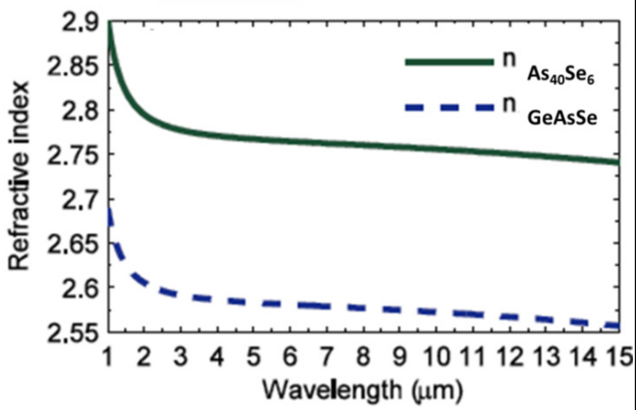
Three cultivation stations with the six crystallizer units

Ultra-high NA chalcogenide step-index fibre for world-record long wavelength supercontinuum

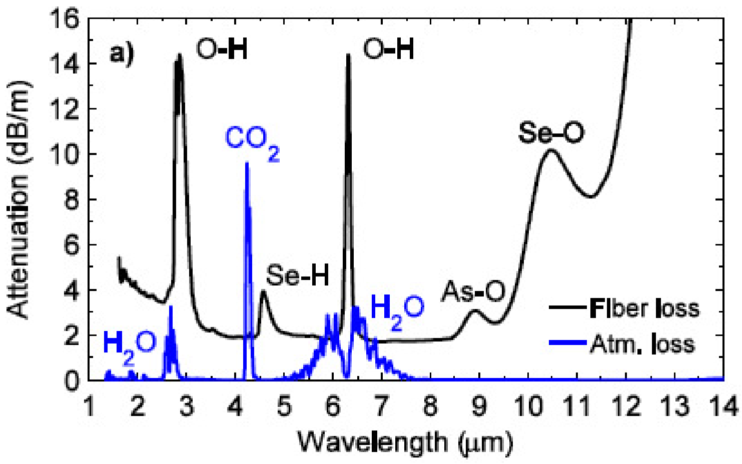
One of the most productive relationships in the MINERVA project has been the collaboration between the Mid-Infrared Photonics Group at the University of Nottingham (UK) led by Prof. Angela Seddon, and Prof. Ole Bang's Fiber Sensors and Supercontinuum Group at the Technical University of Denmark. Even before the project began, the world-leading simulation team at DTU was setting outrageous fibre specifications for Nottingham, which according to its models, would produce supercontinuum generation at wavelengths way beyond what had been observed.

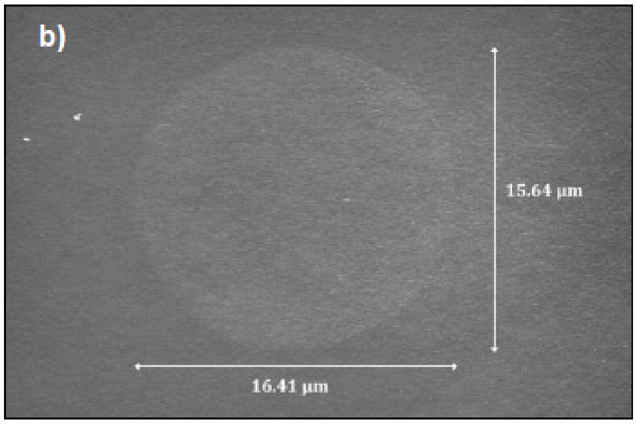
These fibres required numerical apertures (NAs) approaching, and even greater than one, which is not possible according to traditional ray optics (with air as the external medium). However, with propagating modes in a fibre it can be achieved by using core and cladding materials exhibiting a very large difference in refractive index, which can be achieved e.g. with certain chalcogenide glasses used at Nottingham..



Refractive index dispersion of the core and cladding glasses used to make the ultra-high NA chalcogenide optical fibre for MIR-SCG.

*[See HG Dantanarayana, N Abdel-Moneim, Z Tang, L Sojka, S Sujecki, D Furniss, AB Seddon, I Kubat, O Bang, and TM Benson, "Refractive index dispersion of chalcogenide glasses for ultra-high numerical-aperture fiber for mid-infrared supercontinuum generation," Optical Materials Express **4**, p. 1444-1455 (2014).]*



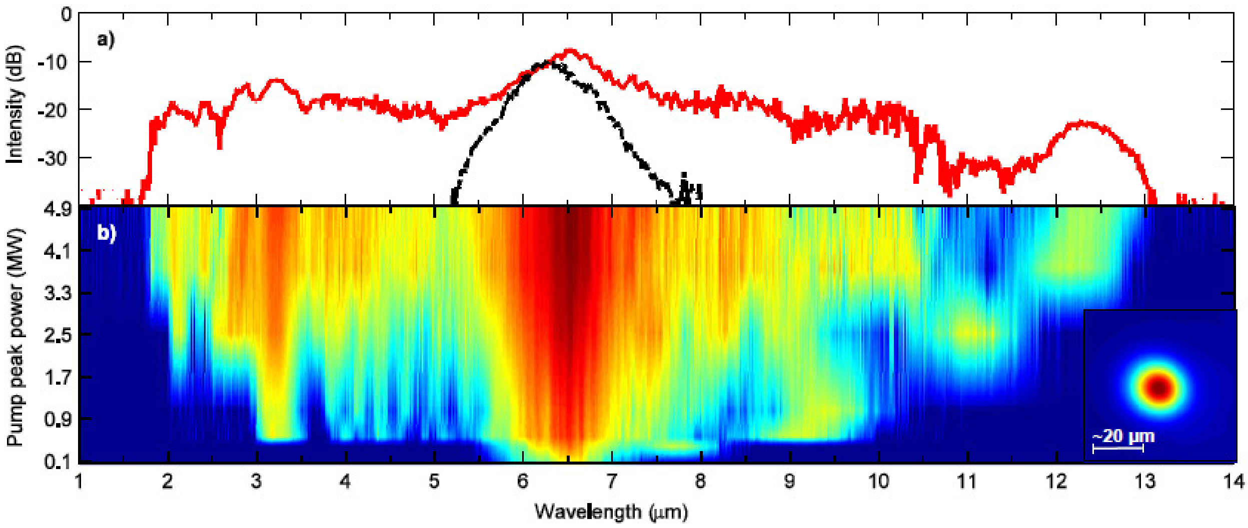


Left: Optical loss of ultra-high NA fibre for MIR-SCG using an intermediate-step fibre by standard cut-back.

Right: Scanning electron micrograph of core of ultra-high NA fibre for MIR-SCG.

[From: CR Petersen, U Møller, I Kubat, B Zhou, S Dupont, J Ramsay, T Benson, S Sujecki, N Abdel-Moneim, Z Tang, D Furniss, AB Seddon & O Bang, "Mid-infrared supercontinuum covering the 1.4–13.3 μm molecular fingerprint region using ultra-high NA chalcogenide step-index fibre," Nature Photonics ***8*** *p. 830–834 (2014).]*

Through its work in the MINERVA project, the Nottingham group produced specially engineered ultra-high NA MIR optical fibre targeting the specifications defined by DTU. Using this fibre with a pump source centred at 6.3 µm, Prof. Bang and his team demonstrated supercontinuum generation (SCG) beyond 13 µm: a great demonstration of simulation validated by experiment! As has been mentioned in a previous newsletter, the results were published in Nature Photonics in Oct-2014 (see reference above) and this paper was selected for Nature Photonics "News & Views" in Nov-2014 ("Entering the mid-infrared," G. Steinmeyer and J. Skibina Nature Photonics **8**). Since then a number of other groups across the world have started the pursuit of high NA mid-IR fibres for high average power supercontinuum sources reaching above 10 µm. The MINERVA team hopes to report further exciting results soon!



World record SCG 1.4-13.3 μm covering molecular fingerprint region, using ultra-high NA step-index fibre.
(a) Spectral profile at max. pump power: relatively flat: 1.8–10.7 μm (–20 dB) with strong spectral peak to 13 μm
(b) Spectral evolution with increasing pump power, with gradual red-shift of the distinct spectral peak and corresponding blue-shifted dispersive wave peaks.
Inset shows captured beam corresponding to spectrum in (a) for all wavelengths.
[Reference: Also from Nature Photonics ***8****, p. 830–834 (2014); see above.]*

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