VERSATILE INFRARED LASER SOURCE FOR LOW-COST ANALYSIS OF GAS EMISSIONS

FINAL ACTIVITY REPORT

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Consortium members:

Participant name	Short name	Country
Thales Research & Technology (Coordinator)	TRT	France
Norsk Elektro Optikk	NEO	Norway
Heinrich-Heine Universität Düsseldorf	HHUD	Germany
University of Southampton	ORC	United Kingdom
Universidad de Valladolid	UVA	Spain

Dissemination level for present deliverable: PU
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Project web site: http://www.neo.no/village/.

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1. PROJECT EXECUTION

1.1. Executive summary

Recent demonstrations of tunable infrared laser sources based on novel nonlinear optical materials have largely renewed the interest for vibrational molecular spectroscopy. Specific absorption features in the mid-infrared (MIR) range of the spectrum are indeed recognized as a powerful and often unique way to provide high sensitivity detection and identification of a large array of molecules. This is particularly relevant in the gas phase in order to avoid preconditioning steps associated with other detection methods (wet chemistry, gas chromatography, mass spectroscopy). Yet, many promising results have remained confined to laboratories for lack of suitable MIR sources, leaving complex Fourier-Transform spectrometers as the only alternative.

To promote direct MIR spectroscopy as a competitive solution for gas analysis, the main technical and scientific objective of the VILLAGE project is the development of a cost-effective widely tunable MIR laser source of high spectral purity. This source combines a 2 µm Thulium (Tm)-doped fibre laser device including a tunable Bragg grating stage and a nonlinear frequency converting crystal (Orientation-Patterned Gallium Arsenide, OP-GaAs) implemented either in a Difference Frequency Generation (DFG) setup or in a high spectral purity optical parametric oscillator (OPO) cavity as shown in the Figure 1.

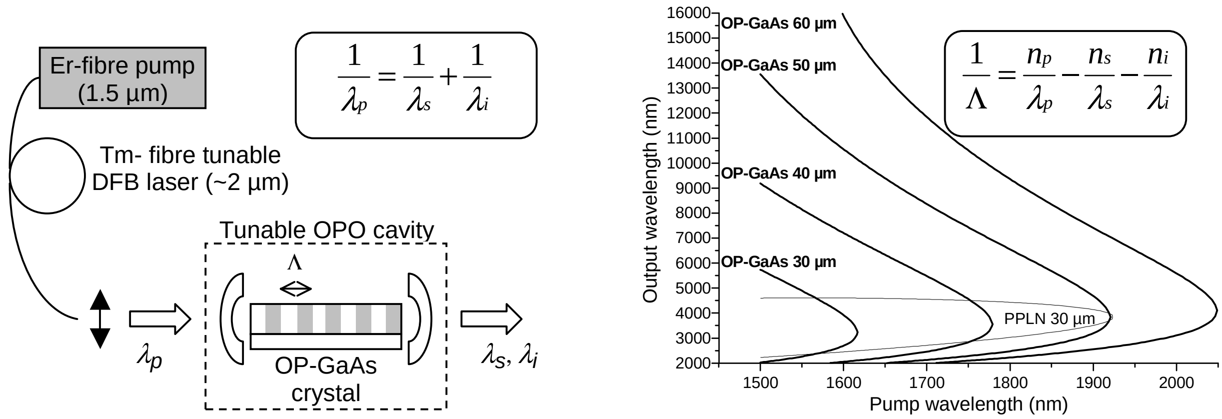


Figure 1 : a) Village source concept (left). b) Typical tuning curves (right).
Insets : key equations linking pump, signal and idler wavelengths, indices and QPM period.

Such a design has the potential for unprecedented performance in terms of both primary specifications and suitability to target multigas analysis of main pollutants generated by and emitted from industrial processes and more specifically of the gases believed to contribute to global warming.

In agreement with the work plan, the first twelve months of the project have enabled the VILLAGE Consortium to specify and fabricate all the subparts needed to implement a first version of MIR tunable source in order to provide useful feedback to the design of the targeted spectrometer. The corresponding technical achievements thus included:

- The fabrication by ORC of prototype Tm-doped fibres with high thulium concentration and the demonstration of narrow-linewidth Tm-doped DFB fibre lasers at 1935 nm and 1943 nm with output power > 0.3 W, further scaled to ~ 1 W. This has been the highest output power so far reported for a Tm-doped DFB laser operating in this wavelength regime.
- The design, fabrication and delivery by TRT of an OP-GaAs sample exceeding initial expectations in terms of propagation losses and geometrical characteristics, suited to DFG around 8 µm and the microscopic characterizations by UVA of various samples to validate a growth model.
- The selection by NEO of interference-free absorption lines by software supported simulations in the wavelength range from 4 µm to 14 µm.
- The implementation by HHUD of a preliminary DFG experiment around 3 µm and of the planned DFG source around 8 µm.

Building on the knowledge and sub-parts obtained during the first year, an upgraded version of this DFG-based MIR source has been implemented, leading to an exceptional tunability from 7.6 to 8.2 µm (1200 to 1300 cm^{-1}) and to the first spectrometric experiment of the project, demonstrating methane detection. In parallel with such a successful technical achievement, the second year of the project also enabled to model numerous OPO configurations and more precisely assess the main technical options for the final implementation. This led the VILLAGE partners to choose to pursue the OPO development work in HHUD laboratories and to select a DFG design as the preferred solution for the final spectrometer prototype assembled by NEO.

The first of those two options was early identified by the VILLAGE Consortium as the most challenging and reaching the oscillation threshold of a continuous wave OPO based on OP-GaAs has indeed remained elusive to date. Nevertheless, the fruitful collaboration between the partners recently yielded a comprehensive body of both theoretical and experimental results at the state-of-the-art. They demonstrate that the designed OPO cavity, resonating both pump and signal (PR-SRO), is most appropriate to be successful in the targeted experiment in a very near future.

From the industrial point of view, the last period of the project proved rewarding because it was possible in a very limited time to turn the laboratory DFG experiment into a compact MIR source and to validate the potential of this source in a spectrometer prototype, through the monitoring of nitrous oxide around 7.5 µm. This setup has been implemented in a 19 inches rack format in the premises of the SME partner of the project. The latest characterization results make it worth contemplating campaigns of field tests.

1.2. Project partners and roles

The project has been supported by the European Commission under the Information Society Technologies priority of the Sixth Framework Program, and involves close collaboration partners from five countries: Thales Research and Technology (TRT, France), acting as the coordinator, Norsk Elektro Optikk (NEO, Norway), the Heinrich Heine University in Düsseldorf (HHUD, Germany), the Optoelectronics Research Centre of the University of Southampton (ORC, United Kingdom) and the University of Valladolid (UVA, Spain).

Thales Research and Technology (TRT):

Thales is one of the world premier professional electronics groups and a major player in numerous commercial markets. It has a revenue of over €10 billion, 65,000 employees and is present in over 40 countries. Thales Corporate Research Centre (formerly Thomson-CSF LCR), located near Paris, is the main multidisciplinary research unit of the Thales group. With over 250 highly skilled staff, 13000 sq. m of labs of which 1700 sq. m is clean rooms, its research teams perform pioneering work in the most advanced areas of optics & optoelectronics, electronic components for microwave applications, advanced interconnect and packaging, materials, software architecture and cognitive science.

In the Laser & Nonlinear Optics Laboratory, research has been active on Quasi-Phase Matching since 1989, and on orientation-patterned GaAs for IR applications since 1993. It presently holds a dominant position in this new promising field. Specific skills and equipment suited to enable further progress in this domain include:

- a pressure and temperature-controlled wafer bonding apparatus adapted to 2” wafers,
- a Hydride Vapour Phase Epitaxy (HVPE) machine adapted to 2” wafers,
- technical personnel in charge of dicing and polishing semiconductor samples,
- optical laboratories equipped with various infrared laser sources for linear and nonlinear characterization.

TRT is be in charge of the VILLAGE project coordination.

Web site: http://www.research.thalesgroup.com

Norsk Elektro Optikk (NEO):

Norsk Elektro Optikk A/S is a SME which was established in 1985 as a privately owned research oriented company within the field of electro optics. The founders had their scientific and technical background from the Norwegian Defence Research Establishment, which for the last 30-40 years has been the leading research organisation in electro optics in Norway.

The company has since its start grown to be one of the largest independent research and development organisations in electro optics in Norway, and has during the last 5 years established itself as a world leading manufacturer of gas monitoring instruments for industrial applications and emission control based on single mode tuneable NIR diode lasers. The company’s main areas of expertise are in activities involving various spectroscopic techniques, the use of lasers and NIR detectors, and the use of digital (CCD) cameras, both single line and two dimensional cameras.

TDL based instruments in the NIR spectral range have been widely accepted by the industrial users and NEO alone has more than 1500 instruments installed in more than 30 countries in Europe, America, Asia and Australia.

Web site: http://www.neo.no/

Institute of Experimental Physics, Heinrich-Heine Universität Düsseldorf (HHUD):

The group headed by Prof. S. Schiller is devoted to the development and application of lasers to high resolution optical measurement techniques. Its activities include the development of new, ultra-stable single-frequency sources for fundamental and applied spectroscopy, in part based on nonlinear optical frequency conversion, and their use for applied spectroscopy (trace gas analysis), frequency metrology and fundamental tests of physics (determination of fundamental constants, tests or Special Relativity).

The group has been working on cw-OPO development and applications since 1993. In the past it has worked closely together with producers of periodically poled crystals to extend the performance of its cw-OPOs, demonstrating wide emission ranges, and wavelengths as long as 4 µm. All cw-OPO devices developed were single-frequency sources.

In parallel with the development work on the cw-OPOs themselves, the systems were applied to spectroscopic studies, in part within collaborations with other institutions. The group was the first to demonstrate that cw-OPOs are suited for advanced spectroscopic methods such as Doppler-free spectroscopy, hole-burning spectroscopy, photoacoustic spectroscopy, cavity ring-down spectroscopy. The group's most important contributions to the device side of cw-OPOs may be summarized as follows:

- Highest conversion efficiency (81%)
- First demonstration of mode-hop-free oscillation over essentially unlimited time
- First reliable and single-frequency cw-OPO using periodically poled crystals (in 1997)
- The largest spectral coverage by a single OPO system (0.56 - 2.67 µm)
- First demonstration of active frequency stabilization

The developments also resulted in a US and EU patent that was licensed to the German company LINOS AG. It manufactures a cw-OPO based on this patent which was the first cw-OPO commercially available in the world.

On the applications side, the group has recently shown that cw-OPOs are suitable sources to perform trace-gas detection with highest sensitivity (at the level of parts per trillion).

Web site: http://www.exphy.uni-duesseldorf.de/

Optoelectronics Research Centre, University of Southampton (ORC):

The ORC has a 35-year history of research and development in the fields of optoelectronics and laser science. It is best known for its pioneering contributions to the field of fibre technology, where it has a strong track record of innovation and where it has been responsible for the development of a wide range of new fibres (including rare earth doped fibres, micro-structured fibres and novel fibre geometries) and novel fibre devices (including the erbium-doped fibre amplifier and all-fibre distributed feedback laser).

The centre has a state-of-the-art fibre fabrication facility, UV fibre grating writing and clean room facilities, and over 60 laser/photonics laboratories with over 130 research-active staff and students. The ORC has throughout its history had strong links with other academic institutions and with industry, and has been actively engaged in a number of successful collaborative research programmes funded by the European Union and other sponsors. The ORC has also been very active in seeking commercial exploitation of its research output through the formation of very close links with industry and through the formation of spin-out companies (e.g. SPI, Stratophase).

Of particular relevance for this project is the collective expertise of the ORC investigators in the areas of high-power (cladding-pumped) continuous-wave and pulsed fibre lasers, DFB fibre lasers, fibre fabrication, fabrication of in-fibre Bragg gratings, and techniques for linewidth control and wavelength tuning. The ORC was one of the first groups to demonstrate over 1kW output power from an Yb-doped fibre laser operating in the ~1.0-1.1µm regime, as well as narrow linewidth and single-polarisation master-oscillator power-amplifier (MOPA) devices at the multi-hundred watt power level and high-average-power pulsed MOPA sources. The ORC has in parallel played a lead role in investigating power scaling of fibre lasers based on erbium and thulium operating in the 1.5µm and 2µm regimes. The ORC is also responsible for the first demonstration of an all-fibre distributed feedback (DFB) fibre laser, and, in collaboration with industrial partner SPI, the ORC has demonstrated the highest power (~400mW) so far achieved from a single DFB fibre laser in the ~1 µm wavelength regime without external amplification.

Web site: http://www.orc.soton.ac.uk

University of Valladolid (UVA):

The group involved in the project is a University department specialized in the characterization of semiconductor materials and devices. Several semiconductors such as InP, GaAs, SiC, GaN, InGaP, SiGe, GaAs/Si, laser diodes, Si, SOI/SIMOX and SiGe have been studied in the past.

A wide range of experimental equipments allowing optical characterization with micrometric or sub-micrometric spatial resolution are currently available in this group, including Transmission Electron Microscopy (TEM), Micro-Raman spectroscopy, Spectrum image cathodoluminescence, Optical interferometry in the Phase Stepping Mode.

A SNOM (Scanning near field optical microscope) is also be available. Only a few laboratories at an European scale actually combine these experimental techniques applied to the study of III-V compounds.

Web site: http://www.uva.es

Thus, the MIR tunable source and resulting spectrometric sensor at the core of the VILLAGE project benefit from several proven trends in the field of emerging photonic components in which the partners have leading positions. Their respective contributions over the whole duration of the project are further described in the four next paragraphs.

1.3. Tunable pumping fibre lasers around 2 microns

The main objective of VILLAGE fiber laser activities, led by the ORC, is to develop continuous-wave and pulsed laser sources with relatively high (multi-watt) output power, narrow linewidth and broad wavelength tunability in the ~2 µm wavelength regime exploiting the broad gain bandwidth of Tm-doped silica fibre and using in-fibre Bragg gratings for wavelength selection and control. These sources provide the starting point for nonlinear frequency conversion to the mid-infrared spectral regime in OP-GaAs crystals, via the two following routes.

Optical Parametric Oscillation (OPO) is the most promising way to obtain mid-IR sources with very large tunability. On the other hand, the power requirements are quite significant and a Master Oscillator and Power Amplifier (MOPA) must therefore be implemented. The development of Tm-doped fibers and DFB fiber lasers has eventually gathered most of the complexity of the project. Because of some technical issues in fabricating custom polarization-maintaining and polarization-discriminating fibers, it was decided to deliver the MOPA in three stages over the whole project duration. The first of these was based on a single amplifier. The second was based on two amplifiers with a maximum output power of 6 W. The master oscillator was a Thulium-doped DFB fiber laser emitting two modes close in wavelength and with crossed polarizations, that was later changed for a DBR structure to obtain truly single frequency operation. Further development of polarization-discriminating Tm-doped fibres recently allowed the demonstration of improved DFB fiber laser oscillators and one of them was used to make a new MOPA with a maximum output power of 15 W.

Difference Frequency Generation (DFG) between a 2 µm Tm-fiber laser and a commercial EDFA (1.5 µm) has been chosen as the preferred solution for the final spectrometer prototype assembled by NEO. For that kind of experiments, the spectral and temporal stability requirements are as strict as above, but a lower power is requested. The last prototype delivered to NEO is thus a Tm-doped DFB fiber laser emitting a maximum output power of 0.8 W around 1960 nm. Its spectral linewidth is below 100kHz and the wavelength can be tuned without mode hops over 20 GHz thanks to a piezo-electric stage.

1.4. OP-GaAs crystals

The research activities carried out by TRT have enabled the definition and study of all the parameters suited to the fabrication of Orientation-Patterned GaAs crystals for mid-infrared generation with an unprecedented quality. The precursor flux and growth speed are well adapted to faithful thickening of the chosen template patterns, preserving both period and duty cycle over the targeted geometrical characteristics. The control of residual impurities and their incorporation enabled a strong decrease in propagation losses compared to the state-of the-art at the beginning of the project, that led to the demonstration of very efficient Difference Frequency Generation and makes future Optical Parametric Oscillation compatible in terms of threshold with the power available from latest fibre pumps. To the best of our knowledge, the obtained 0.016 cm^{-1} value, twice lower than the objectives, constitutes a world record.

Given the excellent results obtained at the end of the first Review Period, it has been possible to proceed during Year 2 with the fabrication of several samples exceeding expectations in terms of losses and geometrical characteristics and suited to both the preparation of OPO experiments and the fabrication of the DFG-based spectrometer prototype. It subsequently occurred that the tuning objectives may benefit from an increased degree of freedom in the choice of grating period. The latest VILLAGE mask and growth runs thus included adjacent gratings with slightly different periods.

In parallel with making available those samples, a significant effort has been devoted during the whole project to pursue microscopic and optical material characterizations thanks to the close collaboration between TRT and UVA. They now offer a coherent body of results, opening preferred routes toward future quality control tools and procedures or improvements in process yield. Last but not least, it recently helped HHUD to validate a model explaining their experimental observations of parasitic back reflections in OPO cavities.

1.5. Turning pumps and crystals into tunable MIR sources

This part of the research activity has been led by HHUD in collaboration with all the partners. The main result of the activity during Year 1 was the implementation of a mid-IR source, based on difference frequency generation (DFG) in orientation patterned GaAs (OP-GaAs). Emissions from a high-power Er-doped fiber amplifier (EDFA, commercial) and a medium-power Tm-doped DFB fiber laser (first version of the Tm- laser from ORC) were mixed in the OP-GaAs crystal supplied by TRT to get a tunable mid-IR output in the 7.6 - 8.2 µm range. The performance of the DFG mid-IR source was subsequently improved while unexpected issues in the implementation of a high power 2 µm pump suited to OPO experiments led the Consortium to chose the DFG design as the preferred solution for the final spectrometer prototype assembled by NEO and to pursue the work toward OPO demonstration in HHUD laboratories.

The results obtained with the first DFG-based setup implemented by HHUD largely corroborated the risk analysis made by the Consortium members at the time the proposal was written. Later published in Optics letters, they emphasized the following points:

- OP-GaAs crystals obtained by TRT constitute unique frequency converters in the mid-infrared range in terms of conversion efficiency.
- They enable to benefit from developments in the field of tunable lasers around 1.5 and 2 µm.
- They are appropriate to reach a milliwat power level from a few watts of pump light, which proves suited to the development of a DFG-based spectrometer.
- A single OP-GaAs grating offered a 100 cm^{-1} tuning range around 8 µm with non-optimized components.

A significant part of the research effort from NEO in the second half of the project has thus been devoted to the valorisation of those achievements by turning a compact version of the above setup into a gas monitoring spectrometer, as described in the next paragraph.

The development and characterization of the DFG source provided important inputs for our estimates on the feasibility of parametric oscillations in OP-GaAs in CW and QCW regimes of the Tm- laser pumping. Approaches for wavelength control and tuning of the OP-GaAs OPO were developed using HHUD expertise in this field. As a result of preliminary work, the PR-SRO design option (resonating both the pump and the signal waves) has been chosen for implementation. We subsequently spotted, evaluated and resolved various issues that may impede future implementation of a CW OP-GaAs OPO, as highlighted below:

- Thermal optical effects in the PR-SRO cavity:

Relatively high threshold (tens of watts) in combination with residual losses in the OP-GaAs (few percent) result in various thermal optical effects potentially complicating the control and stabilization of the OPO cavity. We evaluated thermal optical effects by means of computer simulation. As a result, the developed PR-SRO cavity is “immune” to the thermal lensing.

- Properties of the nonlinear material:

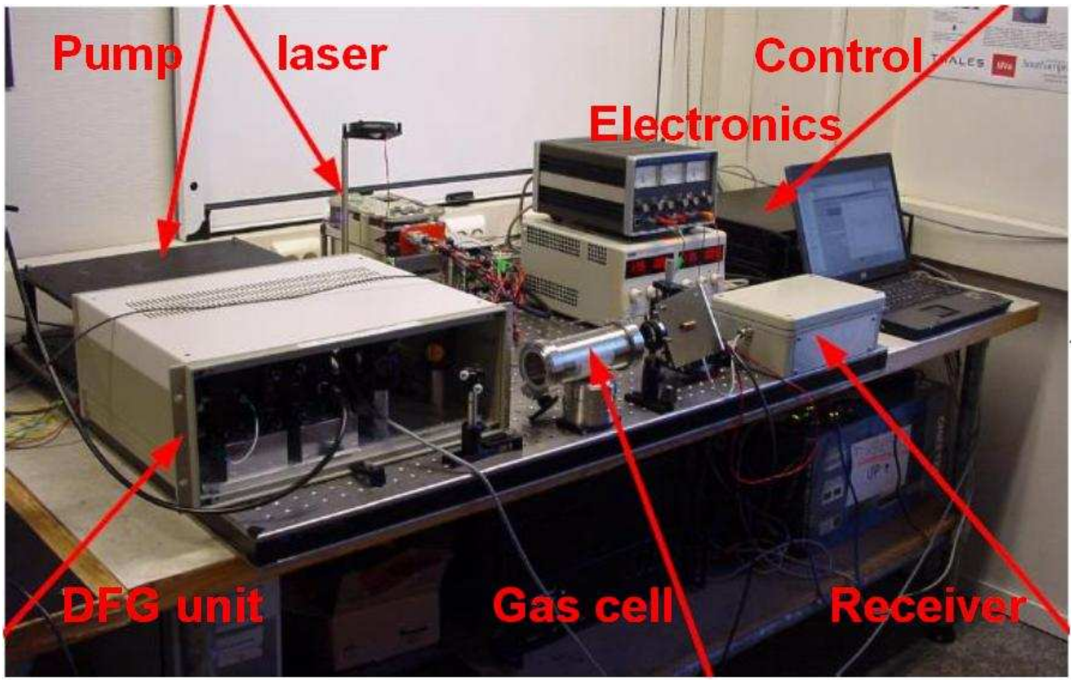
Low parasitic losses in the OP-GaAs is the crucial factor for implementation of the OPO device and remarkable progress has been made during the research project in this respect. Another interesting property of the OP-GaAs crystal, namely an anomalously strong back-reflection from the QPM grating has been revealed only recently, during first experiments with the PR-SRO setup. Consequently, the model of the ring cavity with a back-reflecting OP-GaAs crystal is our latest development.

- Properties of mid-IR optics:

Fabrication of a high performance dielectric coated optics suited to the VILLAGE requirements proved a challenging technical problem. As a result of joint efforts of TRT and HHUD, we found a good compromise solution for the PR-SRO optics, thus minimizing delays in our actual experimental work.

1.6. Development of a spectrometer prototype

As described in the three previous paragraphs, significant progress in all the key subparts of the targeted mid-Infrared source have eventually enabled NEO, the Norwegian SME in charge of the validation of the VILLAGE concept, to successfully build a spectrometer prototype, shown in the photo below and suited to the continuous monitoring of a N_2O absorption line around 7.5 µm (1300 cm^{-1}):



Even if the packaging of the last version of the fibre laser from the ORC is not sufficiently reliable for installation of the DFG spectrometer at an industrial site, the following conclusions have been reached:

- The issues related to the simultaneous emission of two polarizations modes in the Tm-doped fiber lasers that somehow slowed our progress have been solved in a robust manner, so that the expected power level is available.
- Noise issues have been investigated and the results compare well with QCL-based experiments.

1.7. Perspectives

The above demonstration constitute a key achievement of the VILLAGE project and can be further put in perspective with future developments:

- The OP GaAs crystal is of high interest for several research groups and competitors and may be sold as a stand alone product for a number of spectroscopy-related applications.
- We have demonstrated a DFG-based gas analyzer based on OP GaAs that is, to the best of our knowledge the first of its kind. Moreover, the spectroscopic measurements using the DFG source have demonstrated that sensitivities achieved are comparable with state-of-the-art gas analyzers based on Quantum Cascade Lasers (QCL).
- The DFG system has proved to operate as expected. Yet, future multi-gas instruments could still benefit from a widely tunable OPO. More work is necessary to demonstrate that CW operation is achievable, but this seems feasible thanks to the developed PR-SRO setup.

2. DISSEMINATION AND USE PLAN

2.1. Disseminating knowledge

The first milestone of the VILLAGE dissemination strategy has been the project website. It has been hosted by NEO since the end of 2006 and constantly updated at the address:

http://www.neo.no/village/

The VILLAGE partners have further disseminated their research results over the three yearly reporting periods of the project, at international and national conferences as well as in refereed research journals. The full list of publications and communications appears in the next tables, demonstrating that the quality of the results is expected to enable the dissemination of VILLAGE achievements well beyond the end of the project.

Dissemination activities toward other communication channels have also been sought. The key results are listed below :

- Registration of the project to the OPERA2015 website.
- Networking with other related European project such as VERTIGO (Versatile two micron light source).
- Participation to the ICT 2008 Conference and Exhibition (Lyon, France, 25-27 November 2008), reported in detail in a separate publishable Deliverable.
- Promotion by NEO of the project outputs to industrial end-users, as explained in the next section on exploitation of results.

Title	Author(s)	Journal/ Event	Planned/ Actual Date	Type	Type of audience	Countries addressed	Size of audience	Partner involved
REPORTING PERIOD P1								
Development of mid-IR CW narrowband 5 – 15 µm tunable laser source for molecular spectroscopy	S Vasilyev, S Schiller, A Nevsky, A Grisard, D Faye, E Lallier, Z Zhang, M Ibsen, A Clarkson, P Kaspersen, A Bohman, P Geiser	DPG AMOP meeting 2007 Düsseldorf Germany	mars-09	Poster	Research	Germany	1000	HHUD/ ORC/ TRT/ NEO
REPORTING PERIOD P2								
Thick Orientation-Patterned GaAs (OP-GaAs) for Mid-Infrared Laser Sources	D. Faye, E. Lallier A. Grisard, B. Gerard	ICCG 07, Salt Lake City, USA	août-07	Conference	Research	All	300	TRT
Systems and applications at Norsk Elektro Optikk AS	P. Kaspersen	FLAIR 07, Florence, Italy	sept-07	Conference/ Exhibition	Research/ Industry	Europe	100	NEO
Development of a Mid-IR CW narrowband 5-15 µm tunable laser source for molecular spectroscopy	S. Vasilyev, A. Nevsky, S. Schiller	HRMS 07, Dijon, France	sept-07	Poster	Research	Europe	300	HHUD
Thulium-doped distributed-feedback fiber laser with > 0.3W output at 1935 nm	D. Y. Shen, Z. Zhang, A. J. Boyland, J. K. Sahu, W. A. Clarkson and M. Ibsen	BGPP/NP 2007, Quebec	sept-07	Oral presentation	Research	All	400	ORC
Tunable mid-IR CW narrowband laser source for molecular spectroscopy	S Vasilyev, S Schiller, A Nevsky, A Grisard, D Faye, E Lallier, Z Zhang, M Ibsen, A Clarkson, P Kaspersen, A Bohman, P Geiser	DPG AMOP meeting 2008 Darmstadt Germany	mars-09	Poster	Research	Germany	1000	HHUD/ ORC/ TRT/ NEO
Single-Frequency Tm-Doped Fiber Master-Oscillator Power-Amplifier with 10 W Linearly Polarized Output at 1943 nm	Z. Zhang, A. J. Boyland, J. K. Sahu, M. Ibsen, W. A. Clarkson	CLEO 2008, San Jose, USA	mai-08	Oral presentation	Research	All	2000	ORC
Broadly tunable sub-mW CW Narrowband mid-IR Laser Source for Molecular Spectroscopy	S. Vasilyev, S. Schiller, A. Nevsky, A. Grisard, D. Faye, E. Lallier, Z. Zhang, A. J. Boyland, J. K. Sahu, M. Ibsen and W. A. Clarkson	Modern Applications of Trapped Ions, Les Houches, France	mai-08	Poster	Research	Europe	300	HHUD/ ORC/ TRT/ NEO

Title	Author(s)	Journal/ Event	Planned/ Actual Date	Type	Type of audience	Countries addressed	Size of audience	Partner involved
REPORTING PERIOD P3								
Growth and characterization of orientation-patterned gallium arsenide with low optical losses for quasi-phase matched nonlinear frequency conversion in the mid-infrared	A. Grisard, D. Faye, E.Lallier, B. Gerard, M. Avella, J. Jimenez	ICPS 08, Rio de Janeiro, Brazil	juil-08	Conference/Exhibition	Research/industry	All	2000	TRT/ Uva
QPM-GaAs for Mid-Infrared applications	E. Lallier, D. Faye, A. Grisard, and B. Gerard	EPS-QEOD 08, Paris, France	sept-08	Conference	Research	Europe	300	TRT
Broadly tunable sub-mW cw narrowband mid-IR laser sourcefor molecular spectroscopy	S. Vasilyev, S. Schiller, A. Nevsky, A. Grisard, D. Faye, E. Lallier, Z. Zhang, A. J. Boyland, J. K. Sahu, M. Ibsen and W. A. Clarkson	HRMS 2008, Prague, Czech republic	sept-08	Conference	Research	Europe	300	HHUD/ ORC/ TRT/ NEO
High power Thulium-doped fiber distributed-feedback laser at 1943 nm	Z. Zhang, D. Y. Shen, A. J. Boyland, J. K. Sahu, W. A. Clarkson and M. Ibsen	Optics Letters 2008 Vol.33 (18) pp.2059-2061	sept-08	Publication	Research	All		ORC
Distribution of point defects in Orientation-Patterned GaAs crystals a cathodoluminescence study; Appl. Phys. Lett. 93, 151115 (2008)	D. Faye, A. Grisard and E. Lallier, B. Gérard, M. Avella, J. Jimenez	Appl. Phys. Lett. 93, 151115 (2008)	oct-08	Publication	Research	All		TRT/ Uva
Cathodoluminescence Study of orientation patterned OP-GaAs crystals for mid IR laser sources	H. Angulo, L.F. Sanz, M. Avella, J. Jimenez, D. Faye, A. Grisard, E. Lallier, B. Gérard	HeTech, Venice, Italy	nov-08	Oral presentation	Research	All	100	TRT/ Uva
Development of broadly tunable cw narrowband mid-IR laser source for molecular spectroscopy	S Vasilyev, S Schiller, A Nevsky, A Grisard, D Faye, E Lallier, Z Zhang, M Ibsen, A Clarkson, P Kaspersen, A Bohman, P Geiser	DPG AMOP meeting 2009 Hamburg Germany	mars-09	Poster MO 23.11	Research	Germany	1000	HHUD/ ORC/ TRT/ NEO
High power thulium-doped fiber lasers (Invited Paper)	W. A. Clarkson, L. Pearson, Z. Zhang, J. W. Kim, D. Y. Shen, A. J. Boyland, J. K. Sahu, M. Ibsen	OFC 2009, San Diego, USA	mars-09	Invited presentation	Research	All	2000	ORC

Title	Author(s)	Journal/ Event	Planned/ Actual Date	Type	Type of audience	Countries addressed	Size of audience	Partner involved
REPORTING PERIOD P3								
Quasi-Phase Matched Gallium Arsenide for Mid-Infrared Applications	E. Lallier, D. Faye, A. Grisard, and B. Gerard	MICS 2009, Trouville, France	juin-09	Invited presentation	Research	Europe	~100	TRT
High power two-micron fiber lasers	W. A. Clarkson, L. Pearson, Z. Zhang, J. W. Kim, D. Y. Shen, A. J. Boyland, J. K. Sahu, M. Ibsen	MICS 2009, Trouville, France	juin-09	Invited presentation	Research	All	~100	ORC
High-power single-frequency Thulium-doped fiber master-oscillator power-amplifier at 1943nm	L. Pearson, J. W. Kim, Z. Zhang, J. K. Sahu, M. Ibsen and W. A. Clarkson	CLEO/IQEC 2009 Baltimore	juin-09	Oral presentation	Research	All	2000	ORC
Single-frequency Tm-doped fiber DBR laser at 1943 nm	Z. Zhang, A. J. Boyland, J. K. Sahu, W. A. Clarkson and M. Ibsen	CLEO/Europe EQEC 2009 Munich	juin-09	Oral presentation	Research	All	2000	ORC
High power two-micron fibre source: Recent progress and future prosepcts	W. A. Clarkson, L. Pearson, Z. Zhang, J. W. Kim, D. Y. Shen, A. J. Boyland, J. K. Sahu, M. Ibsen	EOS Topical Meeting on Lasers, Capri, Italy	sept-09	Invited presentation	Research	All	~100	ORC
A widely tunable cw mid-infrared spectrometer based on difference frequency generation in orientation-patterned GaAs	P. Geiser, S. Vasilyev, A. Bohman, Z. Zhang, A. Nevsky, S. Schiller, M. Ibsen, A. Clarkson, A. Grisard, D. Faye, E. Lallier, P. Kaspersen	LACSEA 2010, San Diego, USA	févr-10	Poster	Research/industry	All	200	NEO/ HHUD/ ORC/ TRT
High-power linearly-polarized single-frequency thulium-doped fiber master-oscillator power-amplifier	L. Pearson, J. W. Kim, Z. Zhang, J. K. Sahu, M. Ibsen and W. A. Clarkson	Optics Express	Accepted for publication	Publication	Research	All		ORC
Contradirectional mode coupling in ring resonators with QPM nonlinear crystals and effects on the characteristics of cw OPO	S Vasilyev, H-E Gollnick, A Nevsky, A Grisard, J Jimenez S Schiller	Applied Physics B	Prepared for submission	Publication	Research	All		HHUD/ TRT/ Uva
High power, single frequency Thulium-doped fiber DBR lasers at 1943 nm	Z. Zhang, A.J. Boyland, J. K. Sahu, W. A. Clarkson and M. Ibsen	Optics Express	In preparation	Publication	Research	All		ORC

2.2. Direct exploitable outputs

The project objectives are actually twofold: they include a completely new tunable laser source in the Mid-Infrared, which is a product in its own right and is based on several subcomponents also worth exploiting in the long term, and a prototype MIR spectrometer that is specifically designed to form the basis for a new generation of multi-gas analysers based on spectroscopic techniques, for measurements of polluting gases generated by and emitted from industrial processes, and more specifically the gases believed to contribute to global warming.

The following table recalls the key characteristics of the corresponding outputs, listed according to the work package splitting further put in perspective in the five next paragraphs recalling the key achievements, issues and perspectives from an innovation point of view :

	Exploitable Knowledge	Exploitable product(s) or measures(s)	Sector(s) of application	Timetable for commercial use	Patents or other IPR protections	Owner & Other Partner(s) involved
1	Tm-doped DFB fiber laser design	Tunable DFB fiber laser	Research laboratories	> 2 years	Considered	ORC
2	OP-GaAs growth parameters	Wavelength converters	Research laboratories	3 to 5 years	Licensable know-how	TRT
3	DFG source design	Low-cost MIR tunable source	Spectroscopy	3 to 5 years		HHUD (NEO)
4	OPO source design	MIR tunable source	Spectroscopy	> 5 years	Considered	HHUD (NEO)
5	Spectrometer design	Multi-gas monitor	Emission- and process-control (e.g. refinery)	3 to 5 years		NEO (HHUD, ORC, TRT)

2.2.1. Tm-doped DFB fiber laser design

The main objective of Work Package 1 was to develop an efficient, high-power, narrow-linewidth Tm-doped fibre laser source with wide wavelength tunability in the wavelength regime around two-microns for frequency conversion to the mid-infrared. This source would then form the basis of a highly sensitive gas spectrometer system for accurate measurement of the concentrations of various pollutant gases. Our approach was based on the use of an in-band pumped Tm fibre DFB or DBR master-oscillator architecture to provide narrow-linewidth output and the means for tuning the lasing wavelength (i.e. by compressing and/or stretching the fibre grating) followed one or more Tm fibre amplifiers to achieve the desired power levels. A key feature of this approach is that it potentially provides access to a very wide range of operating wavelengths by virtue of the broad emission bandwidth for the two-micron transition in Tm-doped silica fibres, which extends from ~1750 nm to beyond ~2100 nm. We are not aware of any other laser architecture that offers comparable flexibility in operating wavelength combined with power scalability in such a simple format.

The project goals for the two-micron source in terms of linewidth, polarisation and output power have been achieved. However, the wavelength tuning range is currently limited to <10 nm. This does not represent a fundamental limit and hence further work aimed at refining the design of the tuning arrangement should yield a dramatic improvement performance. A wavelength tuning range of > 50 nm should be possible and achieving this is the subject of ongoing studies. An important feature of our DFB laser architecture is the use of a novel fibre design for polarisation selection. This fibre is still at the early stages of development and there is much scope for further optimisation of the fibre design to enhance DFB laser performance. Once again, this will be the subject of ongoing studies and we are considering applying for patent protection for this idea.

At present there are very few commercial fibre-based products that operate in the wavelength regime of interest to Village. We are only aware of one company (Koheras) that can supply a single-frequency fibre source in the two-micron wavelength regime. This is not a standard (off-the-shelf) product and hence is quite expensive. Moreover, the output power is rather low (~20 mW) and the wavelength tuning range is very limited (~1 nm), so these sources are not compatible with the VILLAGE requirements. Single-frequency Tm fibre MOPAs operating at much higher powers have been reported in research literature and at conferences by ourselves and others. At present, the world-record output power from an 'all-fibre-gain-element' single-frequency linearly-polarised Tm fibre MOPA is ~ 100 W and this work was conducted at the ORC using Tm fibre source technology developed in the Village project. This work is the subject of journal paper that has been accepted for publication in Optics Express.

It is clear from the results obtained in the Village project that Tm fibre DFB MOPAs have enormous potential both in the application area of gas spectrometry directly relevant to Village as well as many other application areas. Indeed, our work in Village has brought the realisation of fibre laser products based on Village technology a giant step closer. Clearly, further work on optimising fibre design and laser architectures to enhance performance and reduce costs will be needed, but the prospects for success look very promising. The timescales for the required developments will depend very much on the intended application and hence performance levels required. For the power levels and performance specifications required by the Village application it seems likely that the timescales required for further development and optimisation could be relatively short (i.e. < 2 years). More demanding applications requiring higher average output power in cw and/or pulsed modes will require further fibre development hence will take a little longer. Nevertheless, the prospects for Tm fibre sources in these application areas still look very good indeed.

One of the main obstacles to the development of low-cost Tm fibre laser products is the lack of commercial fibre-pigtailed components (e.g. isolators, modulators, etc) suitable for the ~2 µm wavelength regime. This problem is particularly acute at high average power levels. Further developments in this area will be crucial for successful exploitation of two-micron fibre laser technology. The ORC has already engaged in discussions with component suppliers with a view to establishing research and development programmes aimed at addressing this problem. The Village project has already benefited from some very recent developments in component technology, but more work is needed in this area to improve performance and to bring down costs, even at the relatively modest powers required by Village. As far as we are aware, there is still no other competing laser technology that can rival Tm fibre sources in terms of wavelength flexibility and output power, so the prospects for successful exploitation of this laser technology look very good.

2.2.2. OP-GaAs crystals

The VILLAGE project has enabled the definition and study of all the parameters suited to the fabrication of Orientation-Patterned GaAs crystals for mid-infrared generation with an unprecedented quality. The precursor flux and growth speed are well adapted to faithful thickening of the chosen template patterns, preserving both period and duty cycle over the targeted geometrical characteristics. The control of residual impurities and their incorporation enabled a strong decrease in propagation losses compared to the state-of the-art at the beginning of the project. This led to the demonstration of very efficient Difference Frequency Generation and makes future Optical Parametric Oscillation compatible in terms of threshold with the power available from latest fibre pumps. To the best of our knowledge, the obtained 0.016 cm^{-1} value, twice lower than the objectives, constitutes a world record.

Whether implemented in a DFG or OPO configuration, the latest experimental results obtained demonstrate that further exploitation of OP-GaAs as a versatile wavelength converting material is fully justified. TRT therefore reviewed the fabrication process to get more precise exploitation data. To date, it is able to ensure small scale production of OP-GaAs crystals up to 20 to 30 samples per year. Above that number a preferred solution would be to subcontract the template fabrication and dicing/polishing tasks and license the HVPE growth step.

2.2.3. DFG source design

A narrow-line width mid-IR source based on difference frequency generation (DFG) in orientation-patterned GaAs has been developed in a framework of the VILLAGE project. The DFG source is pumped by a broadly tunable (1540-1570 nm) commercial Er-doped fiber laser system and a custom Tm-doped fiber laser, developed by ORC.

The source can be tuned to any frequency in the 7.6 – 8.2 mm range with an output power of 0.5 mW. A straightforward improvement of the source is an increase of the mid-IR output power by a factor of 3 by AR coating of the crystal facets. The tuning speed of the mid-IR source can be increased to several cm^{-1} per minute by using commercially available pump lasers with fast mode-hop free tunability. Besides, tuning range of the DFG source can be extended to 6.5 to 15 µm using multi-grating OP-GaAs chips and broadly tunable Tm-doped fiber lasers.

Such an OP-GaAs DFG source is an interesting alternative to current cw quantum cascade lasers because of its broad continuous tunability and spectral purity determined by near-IR pump lasers, for which precise wavelength measurement and stabilization techniques are available. Thus, this developed OP-GaAs DFG source is a unique instrument for variety of scientific applications, which rely on high-resolution molecular spectroscopy (e.g. spectroscopy of ultra-cold molecular ions).

On the industrial application side, it must be emphasized that the OP-GaAs DFG source benefits from a simple design without moving mechanical parts. Combined with capabilities of pumping fiber lasers, it allows to develop very robust and compact turnkey devices for field applications. For instance, it can be a part of mobile gas spectrometers for express on-the-site analysis of gas emissions, as discussed below.

2.2.4. OPO source design

As explained in the Activity report, the VILLAGE consortium calculated that reaching the oscillation threshold of a CW OPO would prove more challenging than predicted. Nevertheless, the most recent models and the review of the promises of this option in terms of power and tunability definitely make it worth pursuing.

Prof. Schiller has already successfully transferred a similar technology to a SME: it licensed its cw-OPO invention, after protection by a European patent, to the German company LINOS, now producing cw-OPOs in the 1.5 to 3.5 µm spectral range, for research laboratory use. With this background, HHUD is very aware of the need to protect IP and to stimulate companies working in diverse application fields. HHUD thus can be helpful in establishing direct links between NEO and third parties, as well as open up new directions of research and application of the GaAs sources.

2.2.5. Spectrometer design

The VILLAGE project has proved a major step on the road toward the development of a mid-infrared spectrometer suitable for several critical measurements in the process industry with significant market potentials due to technical limitations of existing products. Four different design options for the spectrometer laser source have been assessed. It has turned out that a laser emitting a continuous wave (CW) is preferable due to its spectral characteristics and simpler electronics implementation. The two remaining design options, namely a CW mid-infrared source based on Optical Parametric Oscillation (OPO) or on Difference Frequency Generation (DFG), were both followed and the last one selected to build the final prototype of the project.

We have thus demonstrated at DFG-based gas analyzer based on OP GaAs that is, to the best of our knowledge the first of its kind. Moreover, the spectroscopic measurements using the DFG source have demonstrated that sensitivities achieved are comparable with state-of-the-art gas analyzers based on Quantum Cascade Lasers (QCL). Other research groups, in the US in particular, have also worked on CW OPOs using OP GaAs but none of these have been able to demonstrate CW operation yet. Feedback from these groups has led us to believe that we are in the forefront of this research worldwide.

To further pursue the exploitation of these results the following technical points can be listed:

- The OP GaAs crystal is of high interest for several research groups and competitors and may be sold as a stand alone product.
- A widely tunable Thulium-doped fibre laser is the key to a widely tunable Mid-IR source and more work is needed to improve it and more specifically to make progress in the tuning and packaging specifications.
- The DFG system has proved to operate as expected but in the final multi-gas instrument a widely tunable OPO is still the preferred solution. More work is necessary to demonstrate that CW operation is achievable.

2.3. Further use of results by each partner

2.3.1. Thales Research and Technology (TRT)

Since the first successful growth runs carried out before the VILLAGE project, the strategy followed by TRT has been to trigger or join collaborative project to explore the application potential of OP-GaAs and thus pave the way toward a global market for those crystals above the critical size for profitability. As far as propagation losses are concerned, one of the key successes of VILLAGE is that low loss-demanding applications can now rely on state-of-the-art samples and TRT has started to receive informal requests for quotations.

On the other hand, the large body of characterization results obtained in collaboration with UVA opens preferred routes toward future quality control tools and procedures or improvements in process yield. TRT expects to further valorise those results to solve some issues that remain important for thickness-demanding applications.

2.3.2. Norsk Elektro Optikk (NEO)

We have demonstrated experimentally that a spectrometer based on the DFG concept developed and used in Village has basically the same performance (e.g. sensitivity) as similar spectrometers based on Quantum Cascade Lasers. The wavelength tuning of the current lab prototype is based on tuning the 2 micron Tm fibre laser as originally proposed. Wide tuning of the Tm fibre laser has proved to be more challenging than anticipated so with this limitation an alternative and wider tuning may be achieved by tuning the other pump wavelength through introducing a widely tunable seed laser for the 1.5 micron source. In this way adequate wavelength scanning across the absorption line can be achieved and by using different grating periods and temperature tuning of the OP-GaAs crystal an overall tuning range of 150 cm^{-1} is possible.

Further commercialization of a spectrometer based on the improved DFB source as described above will first of all depend on the availability of the two novel components/modules developed in the Village project, namely a robust and reliable 2 micron pump source and OP-GaAs crystals with the right specifications for grating periods, internal losses etc.

In the interim period until these critical components are available commercially the alternative for NEO is to use Quantum Cascade lasers in their commercial MIR spectrometers. Even if QCLs are offered by several vendors as “commercially” available the actual situation is somewhat different. QCLs have a very limited tunability and there are only a limited number of wavelengths available. For commercial applications we generally have, through spectroscopic tests, to identify specific gas absorption lines that has a minimum of cross-interference from other gases in the process. Depending on the process conditions (gas mix, gas temperature and pressure) we may have to use different absorption lines (i.e. different QC Lasers) even for the same gas. As available spectroscopic databases are not sufficiently detailed we need to verify this experimentally. To carry out these experiments a widely tunable source is essential and the improved prototype as described above will be a very valuable tool for these measurements until a widely tunable source can be commercialized.

Another aspect of the Consortium plans for exploitation has been to seek contacts with potential end-users of the spectrometer technology. A brisk demand for spectrometers able to measure sulfur oxide (SO_2), hydrogen sulfide (H_2S), methane (CH_4), nitrous oxide (N_2O), nitrogen oxide (NO_2) and water vapor (H_2O) with a single instrument motivated those contacts. The following applications and markets could thus be identified.

- *Measurement of SO_2 and H_2S in the petro-chemical industry:*

In all oil refineries accurate, reliable measurement of H_2S and SO_2 in the tail gas from a Claus sulfur recovery plant is critical. Unfortunately, tail gas analysis has historically been one of the most difficult applications because of problems with sample line plugging due to sulfur vapors present in the sample. The current instruments available on the market for this application are generally very expensive and maintenance intensive and the industry has for a long time been looking for (in situ) solutions without any sampling. The existing NIR laser-based analyzers are unfortunately not suitable as there are no SO_2 absorption lines in this range.

- *Measurement of SO2 in sulphuric acid plants*

The measurement of SO_2 during the recycling process of industrial waste is another important application. For recycling industrial wastes are incinerated in rotating kilns at high temperatures. The gas that is hereby released is cleaned and used as a raw material to produce high-purity liquid sulfur oxide. Existing extractive measurement methods are maintenance intensive (once per week) and unreliable. Online measurements after the rotating kilns are preferable to increase reliability and decrease maintenance time and costs.

Software simulations have led to the conclusion that interference-free measurements for these applications are possible in the 7.6 µm to 8.2 µm range. The DFG setup described above ideally covers this wavelength range.

2.3.3. Heinrich-Heine Universität Düsseldorf (HHUD)

DFG in OP-GaAs has a lot of application potential, since it can be implemented using commercial sources, which are increasing in output power, in particular Erbium amplifiers (now covering the C+L telecom range at 1.525 to 1.607 µm) and Tm sources (e.g. from IPG, Koheras). We soon will have a 15 W Erbium amplifier in our group and might perform another DFG experiment with this source and the ORC Tm fiber laser with a potential output power increase by a factor of 30 to the 10 mW level.

One essential output of the VILLAGE project was to enable us to apply for a joint academia-industrial project to develop a new prototype DFG source targeting another wavelength range, using periodically poled Lithium Niobate. This project uses our VILLAGE know-how as an input.

2.3.4. Optoelectronics Research Centre, University of Southampton (ORC)

The ORC will further develop the Tm fiber work conducted in the Village project to improve Tm fibre sources, and particularly Tm fiber DFB lasers, to extend wavelength coverage and increase output power in both continuous-wave and pulsed modes of operation. The improved sources will find applications in a number of areas (e.g. gas sensing, spectroscopy, LIDAR, medicine, metrology, defence and materials processing) and will provide an excellent starting point for frequency conversion to the mid-infrared spectral regime where there are many more applications. Further funding to support this ongoing work will be needed and the ORC is currently pursuing a number of different funding opportunities including direct sponsorship of some of the proposed activities from industrial end-users. The members of the ORC team are also considering establishing a Ph.D programme to pursue further fibre work in the two-micron wavelength regime.

2.3.5. University of Valladolid (UVA)

In the frame of the VILLAGE project, UVA has been involved in extended characterization tasks. They proved to be most suitable to better understand a number of issues related to the fabrication of OP-GaAs crystals. As mentioned by TRT, the large body of characterization results opens preferred routes toward future quality control tools and procedures or improvements in process yield and UVA is keen to further engage in the corresponding dissemination.