017501

PI-OXIDE

Photonic Integrated Devices in Activated Amorphous and Crystalline Oxides

Specific Targeted Research or Innovation Project (STREP)

IST – NMP - 3

Publishable final report

Period covered: from 01. 09. 2005 to 30. 11. 2008

Date of preparation: 28. 08. 2009

Start date of project: 01. 09. 2005 Duration: 30. 11. 2008

Project coordinator name Prof. Markus Pollnau

Project coordinator organisation name University of Twente

Revision [2]

Project title	Photonic integrated devices in activated amorphous and crystalline oxides
Project acronym	PI-OXIDE
Project Number	017501, STREP
Web site	http://pi-oxide.el.utwente.nl
Coordinator	Name: POLLNAU, Markus Tel: +31 – 53 – 489 1037 Fax: +31 – 53 – 489 3343 Email: m.pollnau@ewi.utwente.nl UNIVERSITEIT TWENTE Faculty of Electrical Engineering, Mathematics, and Computer Science Integrated Optical MicroSystems (IOMS) Group Hogekamp, Room 7220 P.O. Box 217, 7500 AE Enschede, The Netherlands
Timeline	**Start Date:** 2005-09-01 **End Date:** 2008-11-30
Budget	**Overall Cost:** 2.85 million Euro **Funding:** 1.85 million Euro
Project Partners	Vacotec SA, Switzerland Phoenix BV, The Netherlands Ecole Polytechnique Fédérale de Lausanne, Switzerland Universität Hamburg, Germany Consejo Superior de Investigaciones Científicas, Spain

Project objectives

The general objective of this project was the realization and demonstration of integrated optical devices combining passive and active functions on a single chip. In order to achieve this goal, various host materials, like amorphous aluminium oxide (Al_2O_3) and crystalline sesquioxides (Y_2O_3, Sc_2O_3, etc.) were applied. These materials, which can be grown with good optical quality by pulsed laser deposition (PLD), were doped with rare-earth ions (Er^{3+} or Er^{3+} / Yb^{3+}) in order to achieve the active functionality. For the implementation of the integrated optical functions, dry etching processes on the micro- and nano-scale had to be developed and optimized. The path towards the design and realization of these active / passive integrated devices was completed with the development of novel software tools, which enable the simulation of integrated optical functions based on micro- and nano-scale features while taking the specific properties of the active materials into account. After having shown the feasibility of the application of rare-earth-ion doped oxide materials in integrated optical functions, the realization of more complex devices can be envisaged for the long term. Such devices could, for example, satisfy the future need for large bandwidth in various applications relevant to the community, e.g. METRO and Access networks.

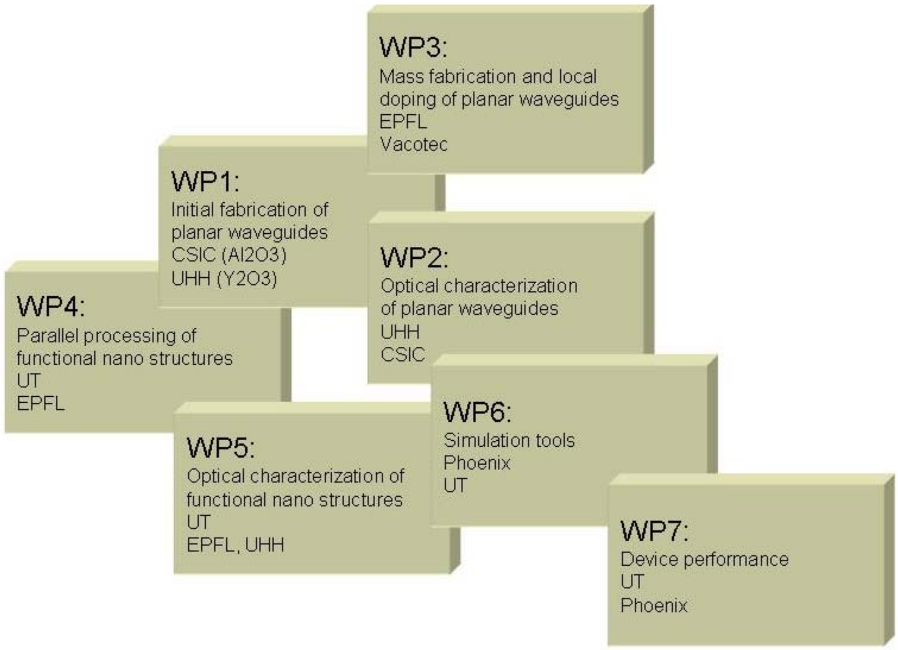
The second key aspect of the PI-oxide project was low-cost production of the aforementioned materials on a large scale, which means that access to mass-production enabling technology had to be ensured. Since PLD is not expected to be suited for processing beyond the laboratory scale, the second research path focused on the development of equipment and processes for rare-earth-ion doped Al_2O_3 and Y_2O_3 based on CVD technology. With this technology, high volume processing was expected to be realized. Moreover, direct integration of active and passive functions by optically activated local doping in a single-layer deposition step was envisaged.

In order to achieve the above described project goals, six project partners with complementary skills in the relevant fields joined the consortium.

Research organization

The research of the PI-oxide project had been organized in seven work packages (see scheme, below).

The basic technologies (deposition, patterning) developed in WP1 and 4 provided during the first half of the project the thin films and structures for the optical characterization in WP2 and 5. Feedback was delivered by the results of the optical measurements in order to allow for the optimization of the deposition and etching processes. Furthermore, the measured optical and structural parameters served as input for the developed simulation tools of WP6. Based on the properties of the optimized active materials in combination with the device feasibility study, the final demonstrator devices (amplifier, laser) were designed, realized and tested in WP7. Furthermore, the experience gained from the material optimization in WP1 served as input for the development of the novel, mass-fabrication enabling deposition tool (WP3). The realization of ready-to-sell deposition equipment was envisaged.



Achievements summary, project course & lessons learned

Year 1: During the first year of this project, the research focussed on work packages 1, 3, 4 and 6. The following results have been achieved.

Within WP1, rare-earth-ion doped Al_2O_3 and sesquioxide (Y_2O_3 and Sc_2O_3) thin films were fabricated and characterized by CSIC and UHH, respectively. Er^{3+} and Er^{3+} / Yb^{3+} doped amorphous Al_2O_3 waveguide layers with nano-scale-controlled dopant distribution were fabricated with a thickness of ~ 1 μm, which was calculated to be suited for the envisaged amplifier applications. Refractive index (1.678) and uniformity (<6%) were measured. In order to improve the optical quality, loss values and luminescence as well as the impact of annealing on the Al_2O_3 material were studied. Furthermore, samples combining rare-earth-ion-doping and Si-nanoparticles were fabricated and investigated. From preliminary results improvement of lifetime and loss was expected. Er^{3+} and Er^{3+} / Yb^{3+} doped crystalline Y_2O_3 and Sc_2O_3 waveguide layers with a pre-defined range of dopant concentrations and thicknesses between 1-2.5 μm were fabricated under various deposition conditions in order to optimize the growth process. Deposition on quartz and sapphire substrates was studied. Due to temperature related phase transitions in the quartz substrate deposition on these substrates was terminated after year 1. Compositional (e.g. stochiometry, dopant concentration) and structural studies (e.g. lattice match, surface roughness) of the samples grown on sapphire were carried out. Refractive index and uniformity (±6.2%) of the crystalline films were measured.

First results within WP3 were obtained at EPFL by the successful deposition of amorphous Al_2O_3 thin films with the HV-CVD technique. 1-μm-thick waveguide layers were fabricated and characterized. Waveguide losses as low as 2.3±0.3 dB/cm at 1.5 μm wavelength were measured.

With respect to the channel waveguide fabrication of amorphous Al_2O_3 and crystalline sesquioxide layers, reactive ion etching exploiting chlorine, bromine and fluorine chemistry was investigated at UT and EPFL, respectively. First results on microstructured channel waveguides with ~ 800 nm step height were obtained by EPFL. Etch process studies at UT focussed on a systematic investigation of all relevant parameters (etch rate, mask selectivity, quality of channels). For the Y_2O_3 material, first results at 73 nm/min etch rate were obtained. The channel quality was inspected (side-wall roughness, channel profile, etch surface roughening). The selectivity of Y_2O_3 etching to various mask materials (organic, inorganic, metals) was investigated. The best selectivity over resist is 0.7. Doped crystalline Y_2O_3 layers were patterned and channel waveguides with ~ 730 nm step height produced. The etch processes developed at EPFL and UT were applied for the channel fabrication of CSIC (Al_2O_3) and UHH (sesquioxide), respectively.

In close collaboration between PhoeniX and UT (WP6), a software module for active material simulation was successfully developed, implemented and tested.

<u>Year 2:</u> In the course of the second project year, the fabrication technologies (WP1, 3, 4) were further optimized, detailed optical characterization (WP2, 5) was carried out and first test structures (WP7) were realized exploiting the software tools developed in WP6. The main results are summarized below.

Applying the PLD process of WP1, optimized Al_2O_3 layers with 0.1at% Er and 0.4at% Yb concentration were deposited while controlling the spacing between Er and Yb incorporation on the nanoscale. Optical characterization of the as-deposited films showed, however, high losses (8 dB/cm at λ = 633 nm), short lifetimes (~1 ms) and no gain. Therefore CSIC focussed on studying the impact of annealing on the optical properties of Er/Yb:Al_2O_3. In parallel, UT introduced an alternative method for Al_2O_3:Er fabrication, reactive co-sputtering, which was developed outside the project scope. At UHH all optical and spectroscopic measurements on sesquioxide films and test structures for IO devices were carried out. The optical quality, however, was not satisfactory at this point. The main reason was found in the lattice mismatch between the sesquioxide films and the sapphire substrates. Detailed study of the growth mechanism of the (poly-) crystalline sesquioxides resulted in the observation of quasi-homoepitaxial growth for Sc_2O_3 and $(Gd,Lu)_2O_3$ on Y_2O_3. While the first combination yields lattice mismatch, the latter one results in perfect lattice match for a carefully chosen combination of Gd and Lu ions. With the demonstration of epitaxial growth in <100> and <111>-directions up to thicknesses of several micrometers a remarkable breakthrough was achieved in the deposition of sesquioxide materials. The progress in growth quality becomes evident from the AFM pictures of the growth of Sc_2O_3 and $(Gd,Lu)_2O_3$ on Y_2O_3 (Figs. 1 and 2, respectively). The latter one results in atomically smooth layers with terraces at the surface corresponding to steps of half an elementary cell (~5.3Å).

The research and development on the large-scale fabrication technology (WP3) was continued by the improvement of the optical quality of the amorphous HV-CVD-grown Al_2O_3 waveguides; losses as low as 2 dB/cm at 1.5 μm wavelength were achieved. An example of a patterned Al_2O_3 film grown at full wafer scale can be seen in Fig. 5. In order to enable the investigation of Y_2O_3 deposition and (local) Er incorporation a novel HV-CVD system was designed and realized by Vacotec in close collaboration with EPFL. Main features of the novel design were a high-temperature heating stage (up to 1200°C), a special effuser ring design, and an excimer-laser-based illumination module. However, preliminary Y_2O_3 and Er deposition tests were not successful.

At UT, the channel waveguide fabrication process for the Al_2O_3, Y_2O_3 and Sc_2O_3 films was optimized. Dry-etched waveguide channels with very smooth side-walls were realized. A SEM cross-section of a high-quality Al_2O_3 channel waveguide, obtained by applying HBr/BCl_3 chemistry, can be seen in Fig. 3. First functional nano-structures were fabricated in Al_2O_3 at UT and EPFL exploiting FIB technology. Resonant structures like Bragg gratings (Fig. 4) and photonic crystals were realized.

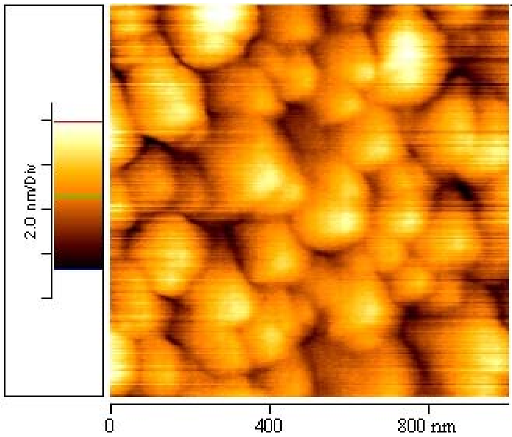


Fig. 1: AFM of 1-μm-thick Sc_2O_3 on Y_2O_3 substrate

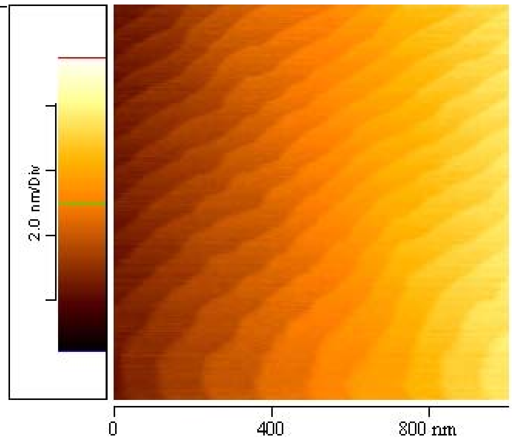


Fig. 2: AFM of 3.1-μm-thick $(Gd,Lu)_2O_3$ on Y_2O_3

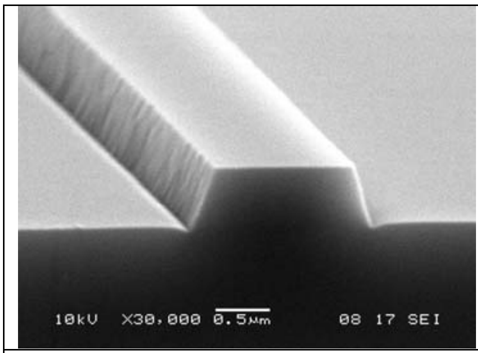


Fig. 3: 1.2-μm-wide and 550-nm-deep channel in Al_2O_3

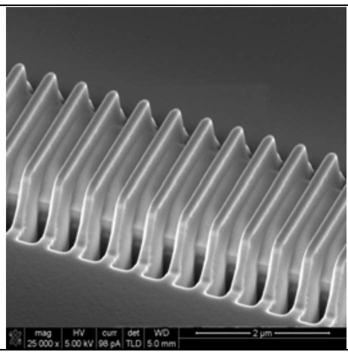


Fig. 4: 23-μm-long grating in Al_2O_3 with 550-nm period

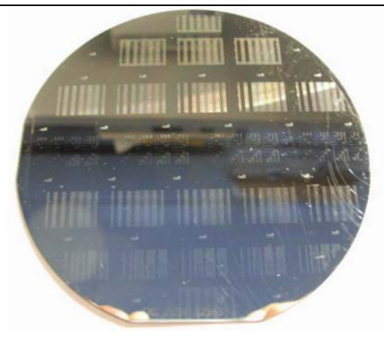


Fig. 5: Wafer view of patterned HV-CVD-grown Al_2O_3 waveguide

With respect to the optical characterization (WP2 and 5), the spectroscopic parameters of the amorphous and crystalline thin films were established. The optical loss of the PLD-grown planar waveguides was characterized and resulted in significant improvement. Upon annealing of PLD-grown Er/Yb:Al_2O_3, loss and lifetime ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) were improved to 2 dB/cm and 2.4 ms, respectively. From the optical characterization of the polycrystalline Er:Y_2O_3 films grown on sapphire, losses in the order of 4 dB/cm (λ = 800 nm) became evident. Gain calculations predicted an expected gain on the order of 1 dB/cm. Since this gain is not sufficient for compensating the losses, two routes were pursued by UHH after year 2: a) further investigation of epitaxially grown $(Gd,Lu)_2O_3$: Y_2O_3 system (lower losses expected), and b) investigation of alternative rare-earth ions like Nd^{3+} (higher gain expected). Microstructures were designed and fabricated for optical measurements. Al_2O_3 channel waveguides with propagation losses as low as 0.2±0.05 dB/cm were realized by combining the reactive co-sputtering deposition with the optimized BCl_3/HBr reactive ion etching process. Gain measurements were carried out over 6.4-cm propagation length in a 700-nm-thick Er-doped Al_2O_3 waveguide. By the end of year 2 net optical gain was obtained over a 35-nm-wide wavelength range (1525-1560 nm) with a maximum of 4.9 dB at 1533 nm.

PhoeniX combined the active materials module with the integrated optics BPM software and extended the Film Mode Matching bend mode solver with Bessel and Hankel functions. Upon implementation of the novel software modules, the full design of active / passive functions was enabled. This tool was exploited for the design of functional test structures in WP7. Bent sections, directional couplers, Y-splitters and race-tracks were realized and tested. Low-loss adiabatic bends with radii down to 250 μm were demonstrated. Combining the results on the active layer properties and channel waveguide performance with the feasibility study on active / passive functionality, several promising devices were selected for the year-3 research. As the unexpected problems around the Y_2O_3 growth and Er doping in the CVD processing became evident by the end of year 2, it was decided to increase efforts on the CVD deposition and to take the alternative wafer scale process for Al_2O_3:Er (reactive co-sputtering) into account in the outline business case of the amplifier devices and for the corresponding demonstrator fabrication in year 3.

Year 3: The focus of the final project year was on the realization of the two main project goals, (1) realization and testing of two final demonstrator devices for integrated optical amplification and lasing and (2) realization of ready-to-sell equipment for large scale fabrication of the novel oxide materials. The following results have been achieved.

The partners involved in WP1 and 2, UHH and CSIC, delivered optimized PLD-grown films and finalized the optical characterization. A final series of PLD-grown Al_2O_3 layers with optimized Er and Er/Yb dopant concentrations was fabricated. Several series of PLD-grown films (Y_2O_3 on sapphire, $(Gd,Lu)_2O_3$ on Y_2O_3 and Sc_2O_3 on Y_2O_3) with optimized concentrations of Er, Yb, Er/Yb and Nd were realized and delivered for gain optimization studies, test component realization, and laser experiments. The optical properties of the optimized PLD-grown thin films were investigated. After annealing of the PLD-grown amorphous Al_2O_3 waveguides at 200 °C, losses at 976 nm in passive waveguides are close to the resolution limit of ~1 dB/cm. Improvement of optical signal enhancement was achieved for the amorphous Er/Yb-doped Al_2O_3 PLD films. Optical loss and gain of 3.0 dB/cm and 4.8 dB/cm, respectively, were measured for $(Gd,Lu)_2O_3$:Er(0.6%) deposited on Y_2O_3.

Micro-structured channel waveguides in $(Gd,Lu)_2O_3$ were realized and optically characterized. Net gain was shown for the Nd- and Yb-doped materials. The gain performance of the Er-doped Al_2O_3 channel waveguides was optimized with emphasis on the achievement of net gain over a broad wavelength range. As can be seen from Fig. 6, 2 dB/cm peak gain and 80-nm gain bandwidth was demonstrated. For non-optimized amplifier length over 3.5 dB net amplification over the entire C-band was shown. According to calculations 18 dB over the C-band is expected upon amplifier length optimization.

The research on the feasibility of nano-scale resonant structures was continued by the design, realization and testing of Bragg gratings. Full grating characterization (transmission, reflection, loss figures) was established (WP5). Transmission values were shown to be according to the design specification; reflectivity and grating loss of 40% and 2.8 dB, respectively, were measured.

The inhibited growth of Y_2O_3 by HV-CVD was investigated. The main reason identified is insufficient heating of the substrate. Despite all efforts on improving the special heater design, only temperatures up to 700°C could be achieved at the sample surface, while the required deposition temperature was 800°C. Therefore, Y_2O_3 waveguide fabrication on a large scale is currently not feasible. Local HV-CVD deposition of amorphous Al_2O_3 sub-mm-sized structures was achieved by applying the novel excimer laser illumination system (Fig. 7). This demonstrates the feasibility of local deposition envisaged as local Er doping in the original project plan. The failure of controlled Er incorporation into the films was found to be due to growth inhibiting behaviour when combining the Al- and Er-precursors. Although some preliminary results on the growth of Er-containing deposits were demonstrated in year 3, the development of mass-fabrication enabling processes by HV-CVD will yet require more detailed research. A ready-to-sell HV-CVD system was realized in close collaboration between Vacotec and EPFL. This system is equipped with the novel high-temperature stage, effuser ring and an excimer laser illumination set-up.

Within WP6, PhoeniX developed and implemented a sub-system (circuit) level simulation tool (S-matrix tool). This tool offers a powerful method to calculate the spectral response of a circuit from a simple model of the constituting components.

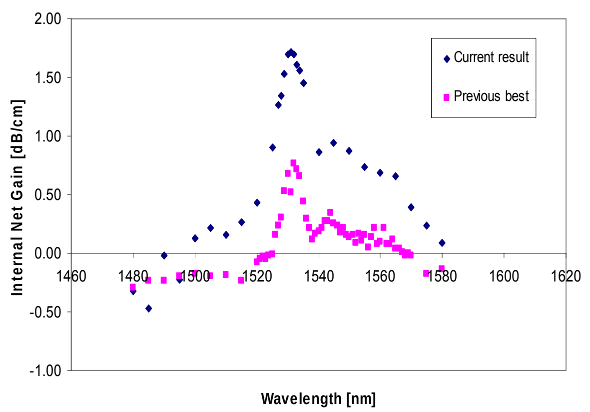


Fig. 6: Gain improvement in Er:Al_2O_3 channel waveguides

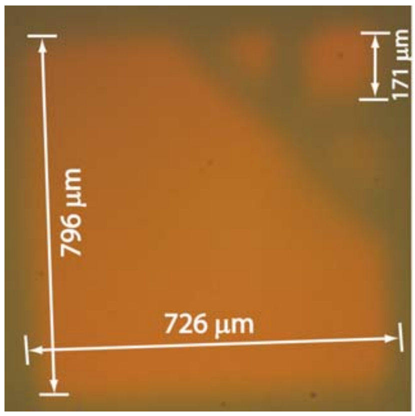


Fig. 7: Optical microscope image of local Al_2O_3 deposition

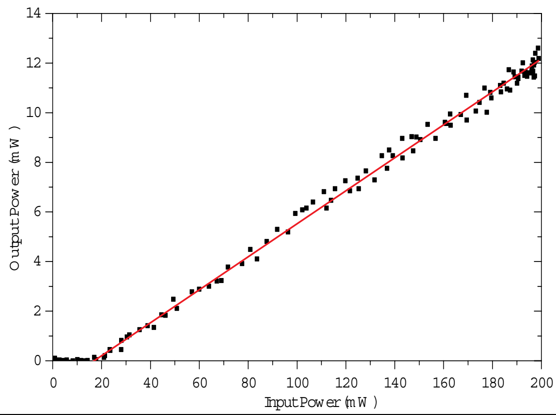


Fig. 8: Laser performance of 3%Yb^{3+}:$(Gd,Lu)_2O_3$ channel

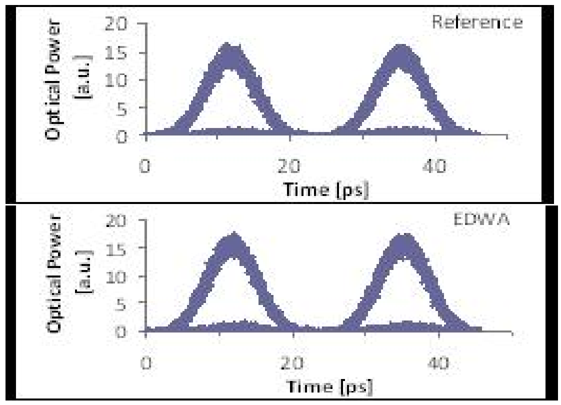


Fig. 9: $Al_2O_3:Er^{3+}$ amplifier response at 40 GBit/s data rate

In order to enable the final choice for the two demonstrator devices, the required device performance was summarized and figures on market need, fabrication cost and sales feasibility were collected. For each potential final device an outline business case was worked out (WP7). Based on the conclusions from the outline business case, two final demonstrator devices, a high-speed (≥ 40 Gbit/s) $Er:Al_2O_3$ amplifier and an integrated high-power sesquioxide laser for satellite clock applications were chosen.

By the end of the PI-oxide project, first channel waveguide lasing with facet-coated mirrors resulting in 12 mW output power, 17 mW laser threshold and 6.7% slope efficiency was demonstrated in $(Gd,Lu)_2O_3:Yb^{3+}(3\%)$ (Fig. 8). Although the demonstrated channel waveguide lasing in the novel sesquioxide material is highly promising for future integrated laser devices (impact1), an on-chip integrated $(Gd,Lu)_2O_3$-based laser could not be demonstrated within the project timeframe, because the realization and characterization of the test structures for the novel materials system, being an essential step towards the integrated laser design, could not be finalized within the project. The on-chip integration feasibility was, however, successfully demonstrated by the realization of an $Al_2O_3:Er$ laser, for which all required test structure characterization had been finalized in the 2nd project year. The slope efficiency and output power of the $Al_2O_3:Er$ laser were 0.11% and 9.5 μW, respectively. The threshold pump power varied from 6.4 to 15.5 mW. Due to the amorphous nature of Al_2O_3, resulting in a broad, unstructured Er gain spectrum, the observed laser output was spectrally highly multimode, hence this approach is not able to achieve the required specifications of the envisaged laser device and to replace the crystalline sesquioxide layers for this or similar applications.

40 GBit/s data rate transmission applying the amorphous $Er:Al_2O_3$ amplifier device was successfully demonstrated. Eye diagrams (Fig. 9) and bit-error rate (BER) were analyzed. No fundamental limitations for increasing the bitrate to 160 GBit/s are expected; corresponding measurements are ongoing after the project closure and will be reported in the literature.

Management, dissemination & exploitation:

The project results have been published by the consortium at international conferences and in peer-reviewed international journals. In total 20 papers have been submitted to / published in peer-reviewed journals; 71 conference contributions, 7 invited seminar presentations, and 5 book chapters have contributed to the dissemination. Four other dissemination activities involve the project webpage, product releases (flyer) and exhibitions. At current 4 journal publications reporting on the final results of the demonstrator devices are under preparation. A more detailed overview of the publications can be found on the project webpage, which was established at the beginning of the project and regularly updated.

The generated knowledge and results are exploited by all academic partners and the SME's of the consortium. On the academic level parts of the PI-oxide work are applied in education; in total ~ 14 lectures benefit from the project results. The assignments of ~ 10 master students have been based on PI-oxide related research. With respect to application of the gained knowledge in new research projects, 7 projects and research collaborations building on the achieved results have been (recently) started and at least two future project proposals are being written up. Several new research initiatives closely involve local SME's, institutes and other universities.

The industrial participants have defined a plan for exploitation based on their work carried out in the project. PhoeniX has started the commercialization of the novel simulation tools. A flyer with an executive summary of the active materials module was created and post news items for product announcements have been spread. The FDTD is also useable as a module independent of the active materials. As part of the commercialization activities, the new modules have been made available to the academic world via Europractice.

Vacotec has established the novel ready-to-sell HV-CVD machine, including a high-temperature stage, special effuser ring design and the excimer-laser based illumination system for local deposition and/or doping. The exploitation strategy for this new technology hardware has been outlined in the business plan, which was prepared by Vacotec as a part of this project.

Impact and conclusions

Important progress to the worldwide state of the art in PLD growth has been achieved by the homo-epitaxial growth of rare-earth-ion doped sesquioxide thin films. This research highlight has directly contributed to the first demonstration of lasing in Nd- and Yb-doped $(Gd,Lu)_2O_3$ channel waveguides. Output powers up to 12 mW and pump thresholds as low as 1 mW have been obtained. Some key parameters (linewidth, output stability, and polarization) for the envisaged space applications (satellite clocks and communication) still need to be measured. Although the realization of fully integrated on-chip lasers applying this novel materials class will require further development in future research collaborations, we expect a high impact and good market perspective of this integrated laser due to the following reasons: (a) linewidth and high-power performance of dielectric crystalline lasers is generally significantly better as compared to their semiconductor counterparts, and (b) the fabrication cost of an integrated version is calculated to be ~10x lower as compared to bulky solutions.

A reliable, wafer-scale amorphous Er-doped Al_2O_3 waveguide technology yielding excellent optical properties has been developed and implemented in this project. Gain measurements on channel waveguides with non-optimized length resulted in 2 dB/cm net gain, 80 nm gain bandwidth and over 3.5 dB amplification throughout the C-band. Calculations on amplifier length optimization predict future gain values above 18 dB throughout the C-band, which is comparable to currently available amplifier solutions. By demonstrating high-speed amplifier performance at 40 GBit/s, the high potential of the novel erbium-doped amorphous aluminum oxide technology has been shown. In order to finalize the development of stand-alone high-speed amplifiers based on the new technology the following steps need to be addressed in an end-user driven project: (a) amplifier design optimization with respect to device length (max. gain), polarization-dependent loss, fiber-to-chip coupling, and chip foot-print minimization, (b) full characterization including parameters like noise figure, gain ripple, polarization dependent loss & gain, and bitrates up to 160 GBit/s, and (c) development of assembly / packaging. According to the cost price calculation examples performed in the outline business case fully packaged high-speed amplifier devices are expected to become available at ~50 €

and ~15 € for 5-point connections (optical backbone) and point-to-point connections (business LAN), respectively.

Besides the realization of stand-alone devices, as focused on throughout the PI-oxide project, the Si-compatibility of the developed Al_2O_3 technology is expected to add particularly to the attractiveness and lead to a high impact upon future research on integration with Si-photonics. Monolithic integration of amplifier components on multi-functional optical chips will not face any competition from fiber amplifiers or stand-alone waveguide amplifiers.

Further achievements include the development of software tools to support the design process of new devices in the technologies described above. The realization of the active materials modules, their combination with integrated optical design methods and S-matrix calculation tools substantially expanded PhoeniX' software portfolio. Besides the direct contribution to the PI-oxide project, future projects on integrated optical amplifiers and lasers as well as PhoeniX' software market share will benefit from these results.

The demonstration of amorphous Al_2O_3 deposition and the feasibility for in-situ local deposition have shown the potential for future mass-fabrication facilities based on HV-CVD technology. Fabrication of sesquioxide thin films such as Y_2O_3 and the controlled incorporation of rare-earth ions into the growing films will, however, require further fundamental research. The realization of ready-to-sell manufacturing equipment by Vacotec is considered to be an important step towards the commercialization of this novel technique.

In conclusion, the research project PI-Oxide, "Photonic Integrated Devices in Activated Amorphous and Crystalline Oxides", funded in part by the European Union, has delivered several breakthroughs in the materials and technology development for the fabrication of optically active devices. First results on integrated on-chip light generation and high-speed amplification have been successfully demonstrated. The generated results form a solid basis for the future research collaboration between the partners of the PI-oxide consortium, and have enabled several follow-up R&D projects, in which together with industrial end-users integrated amplifier and laser products will be developed and introduced to the market.