Publishable Summary for 18SIB10 *chip*S·CAL*e* Self-calibrating photodiodes for the radiometric linkage to fundamental constants

Overview

Optical power measurement is vitally important for spectrally resolved measurements in photonics across industry, environment, health and science. Using today's costly and work intensive methods based on electrical substitution, the optical power uncertainty is limited by the properties and insufficient stability of current silicon transfer standard detectors. *chip*S·CAL*e* aims to improve and simplify the traceability chain by taking advantage of the intrinsic quantum properties of silicon photodiodes where their response to 99 % is determined by the values of fundamental constants. Developing new and improved technology, accompanied by new metrology, *chip*S·CAL*e* generates for the first time an "NMI-on-a-chip" for optical power. *chip*S·CAL*e* will demonstrate that responsivity over a wide spectral range can be predicted based on measurements at one wavelength only.

Need

The European Union has defined Photonics to be one of six Key Enabling Technologies (KET) which is increasingly used in climate monitoring, medical treatment, health and photonic industries, energy saving illumination (LEDs), science, and many more applications. The technological development trend moves in the direction of more integrated measurement systems and distribution of standalone sensor systems in possibly remote locations. Current metrological systems are not capable of calibrating detectors in integrated systems nor remote locations. Therefore, in both the European technology platform Photonics21's strategic roadmap, "Europe's age of light" from 2021 to 2027 and Quantum Flagship's Strategic Research Agenda March 2020, integration of self-calibrating systems and products are highlighted as one of the technology, research and innovation challenges ahead.

Provided by the best laboratories in the world, the current state-of-the-art spectrally dependent uncertainty of around 0.1 % is limited by the properties and stability of current silicon transfer standard detectors. Previous projects have developed the Predictable Quantum Efficient Detector (PQED), which has proven to have an extremely low external quantum deficiency (EQD) of around 0.01 % and an undetectable drift over 9-10 years. This means that to 99,99 % the responsivity of the PQEDs are determined by the values of fundamental constants. These properties make the PQED a very attractive calibration standard detector and complies well with the low-cost, high-accuracy, transfer standard requested by CIPM's Consultative Committee for Photometry and Radiometry (CCPR). However, the low availability and lack of experimental techniques to predict the PQEDs internal quantum deficiency (IQD) has to date prevented it from being exploited as a stand-alone primary standard.

Furthermore, CCPR and EURAMET express the need to measure fundamental constants ratio e/h radiometrically as the ultimate comparison of the two accepted radiometric primary standard detectors, with no preference for either of them, as a contribution to a strengthened and coherent SI system defined by fundamental constants.

Objectives

The overall objective of the project is to develop new and improved experimental techniques for optical power measurements over a wide spectral and dynamic range by the production of an "NMI-on-a-chip" detector developed as a self-calibrating silicon photodiode.

The specific objectives are:

1. To develop improved and validated 3D charge-transfer models to predict the PQED internal quantum deficiency. The target prediction uncertainty is 10 % of the internal quantum deficiency value.

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2. To develop the best possible PQED photodiodes for cryogenic operation by using the improved 3D models and evaluation of passivation layer materials, passivation strategies and charge increasing techniques. To manufacture a batch of optimised PQED photodiodes and to acquire bare-chip photodiodes for room temperature operation.
3. To develop instrumentation and packaging enabling self-calibration of photodiodes. The photodiodes should be operated in both photocurrent and electrical substitution mode with sufficient sensitivity and equivalence between optical and electrical heating over a temperature range from 20 K to 300 K.
4. To provide traceability of the self-calibrating photodiodes to the revised SI by measuring the fundamental constant ratio *e/h* to 1 ppm uncertainty at cryogenic temperatures and to 0.05 % uncertainty at room temperature for wavelengths from 400 nm to 850 nm over a dynamic range from 10 nW to 10 mW.
5. To facilitate the take up of the technology and measurement infrastructure developed by the project by engaging standardisation bodies and international organisations (CCPR, CIE, EURAMET and other RMO TC-PR), the measurement supply chain (accredited laboratories, instrument manufacturers) and end users (photonics industry).

Progress beyond the state of the art

Radiometric measurements of radiant power at discrete laser wavelengths are possible with cryogenic radiometers (CR) with an uncertainty down to 0.005 %. Cryogenic radiometers are accurate for most applications, but are bulky, expensive and require a high skill level to operate. Dissemination outside discrete wavelengths is carried out with silicon trap detectors and interpolation functions. The trap detectors' properties and insufficient stability is limiting the comparison agreement at the NMI level to a spectrally dependent dispersion around 0.1 %.

Objective 1 aims to improve the predicted values of PQED response with 3D simulation software. The aim has two purposes:

I. improve the uncertainty in the responsivity to go beyond the IQD losses and
II. use the model in a new experimental technique to independently extract the photodiode model parameters.

In this way, 3D models are used to put intelligence into the measurement system providing improved understanding and enables new measurement techniques to be developed and applied.

The aim of objective 2 is to develop the best possible PQEDs with lower internal losses at cryogenic temperatures. The IQD in the PQED is limited by surface recombination velocity (SRV) and fixed oxide charge (Q_f). These parameters are found from standard material characterisation techniques as capacitance voltage (C-V) and charge carrier lifetime measurements. The passivation material / process giving the best PQED is effectively found by standard material characterisation techniques combined with improved 3D models without going through a full photodiode manufacturing process. The surface characterisation will be performed both at room and cryogenic temperatures.

In objective 3, we aim to develop the needed instrumentation and packaging to operate PQED type photodiodes also as a cryogenic electrical substitution radiometer (CESR). This will realise a self-calibrating dual-mode detector, which can be operated as both primary standards with the same absorber. Heat equivalence and signal to noise ratio (SNR) at sufficient accuracy for cryogenic and room temperature operation to meet the target uncertainties in objective 4 will be studied. Developing the PQED also as a CESR will eliminate present calibration limiting errors such as optical window and photon absorption differences from geometry or imperfect blackness. In this dual mode detector, the same number of absorbed photons will generate the signal in either mode of operation.

The metrological applications of the self-calibrating photodiodes at room temperature and cryogenic temperature to unprecedented uncertainties and simplicity will be demonstrated in objective 4. There are two different routes for self-calibration: i) photocurrent mode with fitted models and ii) dual-mode photodiodes. Exploiting the design of the self-calibrating photodiode, the relative measurement, and PQEDs with improved passivation and prediction models, will allow for radiometric measurement of e/h with 1 ppm uncertainty compared to the 100 ppm uncertainty normally provided by the established standard CESR. The same technology can be used to operate and build self-calibrating photodiodes into applications as an "NMI-on-a-chip" and by that removing the need to move instruments to the lab for calibration. They can calibrate themselves in their own, possibly remote, and unattended location to a lower cost.

Results

Objective 1: Simplified 3D simulation models have been developed and fitted to four I-V measurement curves of a PQED trap detector (produced in an earlier project) at 488 nm using one set of parameters. In the measured change in photocurrent with bias voltage, the IQD vary several orders of magnitude from tens of percent to tens of ppm depending on optical power, bias voltage and beam size. A remarkably good fit of the predicted IQD to the relative photocurrent measurement at different power levels and bias voltages (in the order of 20 % of the IQD value) was achieved despite the simplified 3D simulation model used. The excellent fit shows that the model describes very well the performance of the photodiode and demonstrates that a very good understanding of the physics of the device is achieved. A simplified model is needed as more advanced models would not produce results quickly enough due to computational limitations. Based on the fitted parameters the spectrally dependent IQD was predicted to range from 100 ppm to 10 ppm with an uncertainty around 50% of the value.

Currently, we examine if the simulation model can be used to independently predict the photodiode responsivity by varying a minimum number of photodiode defining parameters. The prediction models are sensitive to geometrical structure and beam size. In order to achieve a unique solution and quantitative correct results more measurements with variations of beam (wavelength and size) and photodiode structure and parameters are needed. Work on more advanced models and I-V measurements on various types of PQED detectors is ongoing.

Objective 2: Photodiodes with excellent external quantum deficiency is successfully manufactured in *chip*S·CAL*e* and has thereby reached the most important goal of the project already. This realised for the first time PQED photodiodes with SiN_x passivation and a stack of SiO_2 and SiN_x based on preceeding material research for optimum working PQEDs. Different passivation materials (SiO_2, SiN_x and Al_2O_3) was studied. Surface recombination velocity and fixed oxide charge are, based on simulations, highlighted as the key parameters needed to be improve for optimum working PQED photodiodes. The SiN_x passivation process recipe without any optical absorption and with fixed oxide charge 2-5 times higher than well working qu-candela photodiodes was achieved. Preliminary measurements of their responsivity shows that the photodiodes manufactured in *chip*S·CAL*e* are the best ones for calibration purposes ever produced. High fixed charge improves the linearity of the photodiodes and enables the dual mode techniques to be exploited at higher power and thereby with better signal to noise ratio (SNR). The batch of Al_2O_3 photodiodes are undergoing the final processing steps.

A set-up making charge carrier lifetime measurements from 80 K to 300 K has been successfully developed and used to evaluate different process test samples. To the best of the consortium knowledge this is the first time lifetime measurement over such large temperature range is performed. Simulation models was used to separate bulk lifetime and surface recombination velocity from the lifetime measurement. Unfortunately, the lifetime is reduced on all test samples when reducing the temperature. This means that photodiode parameters are changing with temperature and the expected reduction of losses at low temperature will not be fully achieved. From lifetime measurements, prediction of the expected photodiode responsivity if they were manufactured with the various passivation layers was done. This demonstrates a new independent measurement technique of the photodiode responsivity from material characterisations and simulations only. At room temperature, the predicted IQD around 1 -10 ppm and 10-100 ppm of the two produced photodiodes are qualitatively consistent with the first preliminary measurements of photocurrent ratios between previous PQEDs and *chip*S·CAL*e* PQEDs.

The insensitivity of IQD performance with passivation layer thickness enabled us to minimise the reflection losses (for both Al2O3 and SiNx) based on spectroscopic ellipsometry. This resulted in passivation materials with known refractive indexes and photodiodes with optimised EQD. The excellent quality of the manufactured photodiodes reduces the need for accurate prediction of the EQD value. Preliminary examinations indicate that responsivity of these photodiodes mounted in a trap structure is VERY CLOSE (1 ppm range) to an ideal photodiode whose responsivity is given by fundamental constants and the applied wavelength only. In the next period work on how good these PQEDs are and comparison between evaluation techniques will be conducted.

Objective 3: Packaged devices for both room temperature and cryogenic temperatures have been developed based on thermal models with COMSOL Multiphysics. The heating of the photodiode optically is made in the center of the photodiode, whereas electrical heat occurs around the edges. With the current design, simulations show that optical and electrical heat equivalence meeting the target uncertainties is achievable. For room temperature heat equivalence is limited by radiation losses. However, with 11x11 mm^2 photodiode dimensions target equivalence to 500 ppm was achieved. The heat equivalence between optical and electrical heating is approximately 0.1% times the photodiode emissivity per mm displacement. Work on quantifying the effective emissivity is ongoing.

At cryogenic temperatures, below 1 ppm heat equivalence in the simulated design has been achieved. Different dual-mode modules have been manufactured according to the design for both room and cryogenic temperature and has been tested for experimental set-ups under objective 4.

Dual mode operation depends on heat equivalence and optimised signal to noise ratio. Evaluation of various temperature sensors for room and cryogenic temperature was conducted to find the best ones for the purpose, and will be implemented with the *chip*S·CAL*e* produced photodiodes. New mechanical trap detectors optimised for dual mode set-ups has been designed and manufactured. The very first dual mode detectors based on *chip*S·CAL*e* photodiodes and best temperature sensors are soon ready to be assembled in the trap.

Objective 4: Experimental set-ups for dual-mode photodiode operation is established, one for room and one for cryogenic temperatures. Work has triggered new ideas and two different types of measurement procedures have been developed and tested, compared to the initial planned one. Time constants in the dual mode modules have been measured both optically and electrically. The good agreement between the modelled and measured time constants supports the thermal simulations. Room temperature dual mode experiments has improved significantly. A room temperature dual mode module based on black silicon photodiodes has been tested. Work on thermal stabilisation, electrical shielding and improved measurement bridge has resulted in excellent measurement reproducibility of the IQD to better than targeted 500 ppm uncertainty for power levels higher than 400 µW. The two different measurement procedures agree within the targeted uncertainty and paves the way for a possibly third measurement procedure. Optimisation and exploration of achievable uncertainty and validation of the method will be the main focus ahead. The demonstration that we have sufficient SNR with typical 300 ppm Type A uncertainty is encouraging and the accuracy is likely to be limited by heat equivalence due to beam position and radiation losses. The high fixed charge of *chip*S·CAL*e* photodiodes ensures linearity at higher power levels and the room temperature dual mode experiment can therefore be run with improved SNR. Measurements of *chip*S·CAL*e* photodiodes trap modules are expected to start soon.

Measurements of dual mode detectors at cryogenic temperatures will have improved heat equivalence to better than 1 ppm and has reduced thermal noise, lower heat capacity, increased heat conductivity, lower time constant and better photodiode linearity as compared to room temperature. Measurements on modules with UVG bare chip photodiodes has a typical Type A uncertainty of 50 ppm with the open circuit method and 6 ppm in forward bias mode. The measurements has revealed that improved thermal stabilisation is needed at the cryogenic measurements, which is expected to be improved with the new modules.

Impact

Impact on industrial and other user communities

The production volume of the European Photonics industry accounted for € 69.2 billion in 2015. Industry in general requires accurate and cost-efficient calibration methods to maintain traceability to an SI unit of their methods and equipment. Working out an exploitation plan and making the self-calibrating photodiodes commercially available are the most important criteria for uptake of the technology developed in *chip*S·CAL*e*. Commercialisation of developed products is likely to be targeted in a follow up Support for Impact Project (SIP) project. The two planned animation videos will help users understand how the devices work and make it appealing to use the devices. The videos will also be instrumental in the promotion of the project in scientific talks, on the website and in the approach to the wider user community.

Photonics21 is one of the European Technology Platforms, supporting the Key Enabling Technologies (KET) defined by the EU, and has more than 2500 members from the photonic industry, research institutes, academia and public service. The established contact between the consortium and Photonics21 simplifies the transfer of knowledge about project outputs to this important technology platform. In Photonics21 WG5 strategic roadmap “Europes Age of Light! How photonics will power growth and innovation” for the period 2021-2027 they have already implemented the ideas of *chip*S·CAL*e* and request “maintenance-free, self-calibrating sensors” as both a technology challenge and a research and innovation challenge for optimised value.

Impact on the metrology and scientific communities

The principles and methods developed in *chip*S·CAL*e* will support and strengthen the implementation of the new SI system and the radiometric community's position within the SI. The CCPR has requested the possibility of making radiometric measurement of fundamental constants as will be demonstrated here with unprecedented accuracy by comparing two independent and inherently different primary standards in one device. With the commercialisation of the self-calibrating photodiodes the community will have a new chip-scaled device for measuring optical power with unprecedented accuracy in possibly remote operation.

The validated 3D simulation tool will be used to establish a new service for owners of PQEDs based on customer's own relative characterisations. The new service would support laboratories to make independent realisations based on PQEDs alone. The self-calibrating device will also help a number of project partners to reduce their CMC uncertainty for in-house customer calibration service. In a perspective beyond *chip*S·CAL*e*, other European NMIs can develop their capacity based on the self-calibration technology.

There exist concrete plans in granted projects to exploit PQED photodiodes in conjunction with photonic integrated circuits as an absolute embedded standard for extended dynamic range of optical power. This is intended as a first demonstrator and one possible way of exploiting photodiodes in integrated optics which existing standards are incapable of doing. The embedded reference may be exploited in the future as this technology becomes more mature and wide spread in various applications.

Impact on relevant standards

CIE is the international standardisation committee for light and lighting. CIE – Division 2: Physical Measurement of Light and Radiation has requested that the new experimental techniques developed in *chip*S·CAL*e* is fed into the ongoing revision of TC2-81 Update of CIE065:1985 (Absolute Radiometers). CIE welcomes the idea of an "NMI-on-a-chip" and has agreed to join the stakeholder committee to closely follow the progress of *chip*S·CAL*e.* The technical committee leader of TC2-81, a consortium partner in *chip*S·CAL*e*, presented the project at the CIE 29th Quadrennial Session in Washington June 2019, and will ensure that project outputs can be effectively implemented in the new standards.

Longer-term economic, social and environmental impacts

Photonic sensors are key enablers in a wide range of industrial manufacturing and service sectors including: healthcare, surveillance, and automotives. The resultant leverage makes photonic metrology and sensors a multi-billion euro industry. Improved sensors and simplified traceability will contribute to an improved efficiency in the photonic sensor industry.

The fraction of the population above 65 years age is increasing and this will put more pressure on the health care system. Optical methods are used increasingly both for diagnosis and therapy. Supporting optical methods with improved and simplified calibrations will contribute both on the improved diagnosis and optically based therapy side, as they are known to be faster and less invasive compared to previous surgical methods.

About 2/3 of the Essential Climate Variables (ECV) used to monitor impact of climate change require some sort of optical measurement. SI traceable measurements over decadal timescales require new instrumentation to detect small trends in ECVs from a background of natural variability. A self-calibrating instrument making climate quality measurements in the field, ideally in an autonomous manner would meet this requirement and is mentioned as an important application of basic science in the environmental roadmap for photometry and radiometry. The self-calibration detector proposed in this project is a first step towards such an instrument, and will have a major impact on the quality of Earth Observation data.

List of publications

None at this stage.

Project start date and duration:	01 June 2019, 42 months	
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