Publishable Summary for 19ENG05 NanoWires
High throughput metrology for nanowire energy harvesting devices

Overview

Energy harvesting from renewable sources (solar, heat and movement) is a prominent solution to create small amounts of electrical energy in areas of difficult access, and energy harvesting devices have much potential to address our world energy problems. Nanowire (NW) based energy harvesting systems have achieved encouraging progress, but due to nanometre (nm) dimensions of the wires and large size (m^2) of the devices, they also bring challenges for testing and characterisation. Average properties of energy harvesting devices can be measured, but a quantitative link and correlation between the performance of single NWs and that of the overall device is lacking. This project aimed to develop reliable and high throughput metrology for the quality control of NW energy harvesting systems. Within the frame of this project, hybrid traceable metrology was developed for high throughput nanodimensional, nanoelectromechanical and thermoelectrical characterisation of NW devices made of innovative nanomaterials. All these innovative results will help the nanometric energy harvesting industry including developers and manufacturers in further supplying of more efficient and reliable products.

Need

Limited fossil fuel-based energy resources and their negative effect on the environment have resulted in enormous efforts being made over several decades to make energy supply and consumption more sustainable. Scavenging energy from renewable sources like solar, waste heat and mechanical movement is seen as a prominent solution to our world energy problems.

Over the past two decades, major efforts have been made to develop energy harvesting devices from macro and microscales down to nanoscale. Due to their extremely small physical size and high surface to volume ratio, NW based energy harvesting systems, including photovoltaic solar cells, thermoelectrical and electromechanical energy nanogenerators, have gained tremendous interest and encouraging progress has been achieved. In particular, it has been confirmed that the efficiency of NW solar cells can be enhanced from 17.8 % currently to its ultimate limit of 46.7 % by means of nanophotonic engineering.

While novel designs and materials for various energy harvesting devices indeed offer many potential benefits, they also bring challenges for testing and characterisation. For example, the quantitative link and correlation between the performance of a single NW and that of the overall device is still missing. Moreover, no reliable metrology for large area NW arrays (from cm^2 to several m^2) with diameters between 50 nm and 1 µm is currently available. Quality control of these energy harvesting systems is therefore highly challenging, and high throughput metrology is necessary, which requires the development of traceable measurement methods and models for the characterisation of NW energy harvesters, solar cells and devices.

Objectives

The overall goal of this project was the traceable measurement and characterisation of energy harvesting devices based on vertical NW. The specific objectives were:

1. To develop traceable measurement methods for high throughput nanodimensional characterisation of NW energy harvesters (> 10^8 NWs/cm^2) including 3D form (cylindrical, prismatic, pyramidal) and sidewall roughness.
2. To develop traceable measurement methods for high throughput nanoelectrical characterisation of semiconductor NW solar cells using conductive AFM for current-voltage in the current range 100 fA to 1 mA, SMM for doping concentration variation (between 10^{15} and 10^{20} atoms/cm^3 with an accuracy better than 10 %), and MEMS-SPM for lateral resolution (< 50 nm).
3. To develop and validate traceable measurement methods and models for high throughput nanomechanical characterisation of NW devices, and electromechanical energy harvesters taking into account local bending and compression of NWs including the development of a traceable MEMS-SPM

(< 10 pm depth resolution) for fast simultaneous nanomechanical and electrical measurement of semiconductor and polymer piezoelectric NWs.

4. To develop and validate traceable measurement techniques for thermoelectrical characterisation, based on fast areal thermal imaging, of NWs (thermal conductivity lower than 10 W/(mK) with an uncertainty < 10 %) under different scanning speeds and tip-surface contact.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, standards developing organisations (IEC TC 113 and IEC TC 82) and end users (solar cell and energy generator manufacturers).

Progress beyond the state of the art

High throughput nanodimensional characterisation of NW energy harvesters

A set of demonstrator NW arrays made of different materials (e.g. Si, ZnO and GaN) with diameters below 100 nm, cylindrical shape, aspect ratios between 20 and 100, densities between 10^8 NWs/cm^2 and 10^9 NWs/cm^2 and sidewall roughness ranging from nm to tens of nm have been developed for the first time.

A new high throughput optical measuring method, imaging scatterometry, which works by capturing a series of images of an illuminated sample at several wavelengths, has been used and further developed in this project.

The combination of advanced AFM and Scanning Electron Microscopy (SEM) metrology together with scatterometry and Mueller matrix ellipsometry (MME) showed the capability of this hybrid metrology to measure the complex 3D geometry of large size NW arrays made of their different materials. Access to the geometrical form and size of the nanostructures has become possible locally as well as globally.

Efficient MME with FEM-based data analysis has been developed. Thus, for ideal NW array samples for the first time, a high-volume metrology for reliable determination of order parameters of large arrays of coordinated nanosystems such as Si nanowire arrays has become possible. Further research is necessary to measure non ideal NW arrays.

High throughput nanoelectrical characterisation of semiconductor NW solar cells

The project has gone beyond the state of art by developing traceable methods to perform I-V spectroscopy on individual NW junctions in the dark and under illumination using a new conductive AFM equipped with a MEMS SPM probe and coupled to a commercial version of a contact-mode AFM. The expected relative uncertainty on the data determination was achieved in the order of 10 %. In addition, using a technique that allows 3D nano-printing conductive tip materials, new optimised tip geometries for nanowire characterisation were realised, that allowed the nanoelectrical characterisation of NWs.

Traceable Scanning Microwave Microscope (SMM) measurements of doping concentration profile have been carried out for the first time by probing individually the top of vertical doped NWs (attached to the substrate on the bottom part) in non-destructive contact mode and under environmental conditions compatible with NW solar cell design. The target relative uncertainty of about ± 10 % has been achieved for doping concentration up to 10^{19} atoms/cm^3.

High throughput nanomechanical characterisation of NW devices

An innovative conductive MEMS-SPM with 10 pm resolution and 10 mN maximum testing force has been developed for traceable measurement of direct and converse piezoelectrical properties of NW used in motion-to-electricity EH nanodevices. The functionalities and specifications of this new MEMS-SPM head was validated by means of quantitative measurements of reference resistors, a 100 nm reference pitch standard, free-standing GaN micropillars.

In addition, innovative molded pyramid-like diamond tips and novel 3D nano-printed conductive AFM tips were developed which allow stable simultaneous electric and mechanical measurements of individual NWs. An AFM probe equipped with a standardised (ISO 14577) indenter tip, allowed for the first time to link the mechanical measurements of nanomaterials using AFMs with those nanomechanical measurements using traditional validated nanoindentation instruments.

To further increase the throughput of mechanical measurements a microprobe-based high-speed contact resonance (CR) microprobe was developed. The rate of tracked resonance frequency samples was increased to 100 kSamples/sec.

Fast areal thermal imaging of NWs

The Scanning Thermal Microscopy (SThM) device has been improved to decrease the uncertainty of thermal conductivity measurements from 20 % to 10 % in the case of flat isotropic samples and for materials with a thermal conductivity lower than 10 $W \cdot m^{-1} \cdot K^{-1}$. Non-contact techniques have been combined for fast imaging of local defects in active devices based on nanowire arrays, like solar cells.

Results

High throughput nanodimensional characterisation of NW energy harvesters

Comprehensive fabrication technologies including bottom-up approach, nanospheres self-assembly and metal assisted chemical etching, optical lithography and cryogenic deep reactive ion etching (cryo-DRIE), Zn sputtering/annealing and CBD, and Metalorganic vapor phase epitaxy (MOVPE) have been, for the first time, utilised to fabricate sets of NW arrays made of typical materials for energy harvesting (Si, ZnO and GaN).

NW samples with different nanowire doping types (p- and n-type), diameters (100 nm and 250 nm), heights (from 300 nm up to 8 µm) and periodicity (190 nm and 400 nm) have been characterised by SEM and room temperature photoluminescence that is proportional to the porosity of the nanowires.

The fast in-line optical metrology tools developed in this project work perfectly for ideal NW arrays consisting of high quality uniform structures. The energy harvesting devices developed in this project work well but are not very uniform. This is still a challenge for the developed fast optical methods.

The project consortium created the design of the NWs which met the shape, uniformity as well as aspect ratio requirements and were suitable for multiple measurement techniques. The fabricated nanowires were measured by SEM, SPM, AFM, optical coherent and partially coherent Fourier scatterometry and Mueller ellipsometry. These different types of measurement techniques were combined to determine the geometrical parameters such as the diameter, the height, and the pitch of the NWs. The diameter of nanowires was measured by partners with a 5-10 nm uncertainty, while the pitch and the height could be measured with an uncertainty of 3 nm and 2 nm respectively. Therefore, as a set of demonstrator NW arrays made of different materials (e.g. Si, ZnO and GaN) with diameters below 100 nm, cylindrical shape, aspect ratios between 20 and 100, densities between 10^8 NWs/cm^2 and 10^9 NWs/cm^2 and sidewall roughness ranging from nm to tens of nm have been developed, the overall objective was successfully achieved.

High throughput nanoelectrical characterisation of semiconductor NW solar cells

The objective of performing traceable measurements of I-V characteristics on individual NW junctions with C-AFM techniques and then providing reliable datasets of photovoltaic parameters of NWs turned out too difficult to be achieved within this project. This matter of fact originates to the highly challenging aspect in fabricating working photovoltaic NW junctions. From a multitude of technologies and materials in the design of NW photoelectric devices reported in the literature, only a few lead to high performance devices, i.e. efficiencies reaching 15 %. In this project, various NW samples were fabricated, i.e. arrays of pure n-doped and p-doped NWs and NW p-n junctions samples based on well-known materials but no photovoltaic properties were observed. Nevertheless, thanks to these samples, the works performed here can be considered as a first step for the metrological investigation of photoelectric NWs and provide some good insights of metrological developments to support the NW technologies in the photoelectric domain.

The high relevancy of using non-contact C-AFM techniques, like Electrostatic Force Microscopy (EFM) and Kelvin Probe Force Microscopy (KPFM) has been demonstrated as complementary techniques to the contact C-AFM such as the Resiscope in the detection and characterisation of photoelectric NWs. Investigations focused on error sources in C-AFM photoelectric measurements have shown that a significant error source is the parasitic illumination coming from the red laser of the AFM. We have also shown the interest to extend measurements on NW junctions at the microscale (using a probe station). They can reveal parasitic effects and error sources which undoubtedly exist at the nanoscale but are much easier to be investigated at the microscale (because of no issue due to the AFM tip).

The other challenging objective was to measure the doping concentration values with the SMM scanning the top surface of NWs in contact mode with an expected uncertainty below 10 % and to compare to values determined by Cathodoluminescence (CL) and Electron-beam induced current (EBIC) on similar NWs. To this end, SMM calibration methods were investigated, relying on fit for purpose 2D reference doped multilayer samples fabricated by partners. The key result is the completion of the traceable measurements of doping concentration performed for the first time on the top surface of the vertical pure doped NWs by SMM techniques. Dopant concentration values were found in the same order of magnitude as the expected values. Comprehensive uncertainty budgets have been established leading to combined relative uncertainties ranging from 10% to 35% for doping concentration values between 10^{15} atoms/cm^3 and 10^{19} atoms/cm^3. Error sources and uncertainties were also investigated in CL and EBIC measurements. Therefore, since the doping concentration couldn't be enhanced to 10^{20} atoms/cm^3 and no dataset of photovoltaic parameters was finally obtained the overall objective was achieved partially achieved.

High throughput nanomechanical characterisation of NW devices

With the help of FEM analysis, two MEMS-SPM heads with the maximum indentation force up to 10 mN and a lateral resolution < 50 nm for high-throughput nanoelectromechanical measurements of nanowires have been designed, fabricated, and prototyped. Two calibrated MEMS-SPM heads equipped with different AFM probes have been delivered to the partners of the consortium for nanomechanical and nanoelectrical measurements.

A new high-speed microprobe CR setup with upgraded electronics and firmware (lock-in amplifier, signal generator, micro controller) has been developed. Due to its modular architecture, this setup can be adjusted to future requirements by replacing the limiting components without the need of developing a completely new system.

First nanomechanical measurements of semiconductor and metal NWs using the newly developed high-speed microprobe measurement system have been performed.

Modelling of the tip-surface interaction has been performed by fitting quasistatic measurements using the Hertzian contact theory. A Good Practice Guide (GPG) on the "Characterisation of probe wear in AFM based topography and nanomechanical and surface deformation measurements" was summarised.

Fabrication of specialised AFM probes using a focused ion beam (FIB) facility and 3D nano-printing technology has been realised. The world-first silicon and diamond AFM Berkovich tips have been fabricated using FIB and was characterised using SEM. World-first molded single crystal Berkovich-like tips have been successfully fabricated on silicon wafers.

A new microshaker measurement tool with a bandwidth up to 10 kHz has been designed to characterise the averaged performance of nanowire-based energy harvesting devices. Initial testing of the project's NW devices indicates that the NW's exhibit a macroscopic piezoelectric response to mechanical excitation.

Therefore, the objective has been successfully achieved by means of (1) successful development of traceable MEMS-SPM with a depth resolution of 10 pm and a maximum indentation force higher than 10 mN to bridge the metrological gap among currently available nanomechanical measurement approaches, (2) realisation of high-speed CR microprobe measurement system for fast areal topography and mechanical measurement of NWs, (3) modelling and compensation of tip-NW interaction during the high-speed scanning, (4) development of ISO-compatible innovative AFM probe tips for materials testing, (5) development of innovative micro-shaker to characterise the properties of piezoelectric NW energy harvesting samples/ chips and devices.

Fast areal thermal imaging of NWs

The specifications of sample configurations and MEMS designs have been defined and agreed between the partners. Various samples have been fabricated and delivered, including silicon nanowires with n+ type doping, thin ZnO layers and samples with wide range of solar cells based on SiNWs.

The calibration facility for SThM probes has been updated to perform probe calibration for traceable thermal measurements with less influence of the calibration oven on the temperature gradients in the probe. To improve the possibilities of SThM for thermoelectric measurements, a transformer bridge based electronics was developed, providing galvanical separation of the probe from rest of the circuitry. Patterned substrates have been developed for individual nanowires measurements and nanowires were attached to them by micromanipulation. FEM modelling of new suspended MEMS platforms has been performed to minimise the uncertainty of the thermal conductance measurement of individual nanowires below 10 %. Based on these simulations, the new multi beam platform was constructed and tested which enables measuring the conductance of the NW without contribution of the thermal contact resistance. SThM experiments using advanced sampling via force-distance-energy measurements were performed, showing less dependence on the probe-sample contact formation and topography artefacts. A two-probe SThM setup has been used to measure heat flux through individual nanowires, as well as on reference bulk samples and thin films. On basis of these activities, two good practice guides (GPGs) describing the methodology for MEMS-fixing platform and multi-probe measurements of individual NWs and thermal measurements of NWs (thermal conductivity lower than 10 W/(mK)) with a target uncertainty of 10 % have been created.

An initial set of measurements has been performed on an already traceable infrared microscopy setup, where the different operating conditions of solar cell samples were tested and discussed. Novel sample configurations for thermal measurements were suggested. In parallel, the thermoreflectance setup was created for the local temperature measurements and measurements were performed on nanowires based solar cell samples.

Therefore, the objective (desired uncertainty for the particular range of thermal conductivities thermal conductivity lower than 10 W/(mK) with a target uncertainty of 10 %) was fully achieved by two methods (single probe SThM, MEMS device) and a wider set of thermophysical characterisation methods was improved for better compatibility to nanowire samples in terms of instrumentation, measurements capabilities, traceability, throughput and uncertainty analysis.

Impact

To promote the uptake of project results and to share insights generated throughout the project, results have been shared broadly with scientific and industrial end-users. 25 papers reporting project results have been published in open access peer-reviewed international journals or proceedings, another has been accepted and is awaiting publication, five have been submitted and two more have been drafted. In addition, an article was published in Metrology and Hallmarking, the Bulletin of the Central Office of Measures, 6 Bachelor and one Master thesis have been published as a result of the project. 51 presentations have been given at conferences and the project was presented to over 100 participants at two plenary sessions of NanoMaterials for Energy Applications. A stakeholder workshop was held at the 21st Annual Metrology Congress CIM2021 in September 2021 in Lyon, France and stakeholder meetings were offered online in February and November 2022 as well as July 2023. The project has been presented at national and international standardisation committees and posted regularly on social media through Instagram, Facebook and LinkedIn. A website containing all social media posts has been set up and is periodically updated, in parallel to the official project site, for a wider public. 19 training events were held in the form of workshops, online webinars, staff exchanges.

Impact on industrial and other user communities

New contact-based measurement modes will enable industry to simultaneously measure dimensions, electrical, thermal and mechanical properties of surfaces such as elasticity, stress, adhesion and thickness of nanomaterials. Conductive AFM and SMM techniques combined with EBIC and CL techniques have the strong potential to describe overall performance, unwanted loss mechanisms, optimal operating frequencies and aging within one device. Together, these techniques will certainly fit the need of upcoming industrial production. Advanced diamond probe technology will provide the robustness and longevity currently lacking from contact sensors today.

Industrial users have been contacted and relative key persons have joined the Stakeholder committee. Additionally, ELECTRO has developed their new piezoelectrical measurement tool which is now under commercial reviewing for industrial manufacture. ELECTRO has patented parts associated with it. The application of the new high-speed CR setup on NW arrays developed within this project for commercially available products (e. g., KlettWelding, KlettSintering and ZnO/Si solar cell on glass) has also been assessed and explored.

Impact on the metrology and scientific communities

Partners continued to interact with various scientific, metrological and industrial networks in order to disseminate project results. Three newly established European Metrology Networks (EMN) networks have also been contacted through the project representatives in these communities: Quantum Technologies, Advanced Manufacturing and Clean Energy Networks.

Experiences with advanced NW fabrication methods were documented in a “Report on the fabrication of NW array artefacts for demonstration of traceable measurement methods for high throughput nanodimensional characterisation of NW energy harvesters (> 10^8 NWs/cm^2) including 3D form”. First results have been presented by INRIM to VAMAS TWA2 Surface analysis in September 2021 and EURAMET TC Length in October 2021. This report was also presented at NanoWire Week in April 2022, to more than 200 attendees, and MNE2022 in September 2022 to more than 100 attendees A set of process parameters for cryogenic etching of silicon NWs established at TUBS has been transferred to INRIM to accelerate the introducing procedure of their new Oxford cryo etcher.

Experiences of NW fabrication were exploited with MBE-grown highly boron- and phosphorus-doped silicon for diffusion experiments in NWs of 300-1200 nm in diameter at WWU Münster. Furthermore, for Li-ion-battery applications silicon wires of large height (up to 15 µm) and aspect ratio (up to 22) were fabricated. Detailed results have been published in a paper and were presented at the Physical Colloquium, TU Kaiserslautern, in April 2022, and at the conference Sensoren und Messsysteme in May 2022, to more than 100 attendees.

Training courses for members of the consortium and external audience on nanomanipulation and positioning of single nanowires on MEMS structures were held in August 2021 and March/May/September 2022. Measurement and training services/courses on nanodimensional characterisation of NW arrays using scatterometry were organised.

A one-to-one training for the consortium has been held in September 2021 at DFM, with the aim to demonstrate/investigate the in-situ performance of the MEMS-SPM head with integrated readout electronics developed by PTB, and to integrate the MEMS-SPM head into the commercial AFM (NEX20, Park) at DFM. Furthermore, one-to-one trainings within the consortium have been held on topics of NW fabrication and MEMS-SPM for material testing in July and October 2022, respectively.

Impact on relevant standards

The metrological outputs of this project in the fields of NW solar cells have been presented to standardisation committees e.g. IEC TC 113 ‘Nanotechnology for electrotechnical products and systems’ and IEC TC 82 “Solar photovoltaic energy systems” to foster the creation of new standards. Good Practice Guides developed were disseminated to ISO TC 164 “Mechanical testing of metals”, and the German VDI/VDE-GMA Technical Committee 3.41 “Surface Measurement Technology in the Micro- and Nanometer range” aiming to standardise the new measurement modes.

The project was presented at the IEC TC113 WG13 "Wafer-scale system integration" meeting in November 2021. IEC TC113 WG11 “Nano-enabled energy storage” has recommended the establishment of one Preliminary Work Item (PWI) on “Metrology for nanowire energy harvesting devices”.

Longer-term economic, social and environmental impacts

The metrology developed within the framework of this project will contribute to quality control of newly developed devices for energy harvesting and storage, and consequently help to promote and accelerate the development and fabrication and enable new nanotechnologies for renewable energy industry. This will strengthen Europe’s response to human-induced climate change.

The high throughput metrology for quality control of innovative energy harvesting and storage devices will substantially improve the competitiveness of the European semiconductor and small energy industries. The developed high throughput SPM techniques can also be applied for ultrafast quality control of ultra-precision workpieces, therefore enhancing the competitiveness of European manufacturing industry.

List of publications

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This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

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<tr><td colspan="2">Project start date and duration:</td><td>September 01, 2020, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Uwe Brand, PTB   Tel: +49 531 592 5100   E-mail: uwe.brand@ptb.de
Project website address: https://www.ptb.de/empir2020/nanowires/</td></tr>
<tr><td>Internal Funded Partners:
1. PTB, Germany
2. CMI, Czechia
3. DFM, Denmark
4. GUM, Poland
5. INRIM, Italy
6. LNE, France
7. VSL, Netherlands</td><td>External Funded Partners:
8. Aalto, Finland
9. CNRS, France
10. ELECTRO, United Kingdom
11. GET, Austria (withdrawn from 31/08/2021)
12. PWR, Poland
13. TCD, Ireland
14. TUBS, Germany
15. UAB, Spain
16. QDM, Germany (joined from 01/01/2021)</td><td>Unfunded Partners:
17. CSI, France</td></tr>
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