Final Publishable JRP Summary for ENG53 ThinErgy
Traceable characterisation of thin-film materials for energy applications

Overview

EU targets for the use of renewable energy and energy efficient devices are driving rapid growth in the global market for low-carbon goods and services, resulting in increasing demand for advanced materials and related technologies. Thin films, with a thickness from a fraction of a nanometre to several micrometres, are key components in numerous energy applications such as solar cells, LEDs, energy efficient windows and solid state power electronics which are used to control the flow of electricity from the grid.

The lack of reliable measurement protocols and calibration procedures for thin films has hampered the development of these technologies. A key challenge is that thin film materials typically have complex structures, requiring multiple characterisation techniques to analyse them adequately.

This project developed a measurement framework for reliably characterising thin films, and has delivered new instrumentation, industrial consultancy, calibration services, standards documents, new solar cell technology, and a spin out company.

Need for the project

A single technology will not be sufficient to ensure Europe's shift to a low carbon economy. This requires a wide range of technologies, including power electronics (based on multilayers of thin film semiconductors), solid state lighting, solar energy and energy efficient windows. This project builds on some of the outputs of project *IND07 Metrology for the manufacturing of thin films* to develop new traceable measurement methods for these devices.

Energy technologies require manufacturing of devices, such as solar cells and LEDs, in high volumes. Prior to this project, the uptake of advanced thin film materials used in such devices was slow due to the challenges in demonstrating the required performance and reliability. Traditionally these devices were first manufactured and their different characteristics subsequently separately measured, meaning that adjustments to the manufacturing process had to take place after manufacture, which was an expensive and time consuming process. In addition, more batches were rejected during this quality control process than expected, partly due to the lack of clarity about which of the multiple measurement parameters were the most important to optimise for best performance. In order to accelerate innovation in energy technologies, provide confidence in the adoption of advanced thin film materials and reduce costs in manufacturing, it is critical to understand (i) which parameters need to be measured, (ii) which parameter correlate with device performance and reliability, (iii) what the accuracy of that measurement needs to be, (iv) which measurements should be done during the manufacturing process and (v) which can be performed after manufacture.

The different energy technologies mentioned above are based on complex thin film materials which often have non-uniform composition and are notoriously difficult to measure. That means that key measurement challenges are common to multiple energy technologies and if solved can have significant impact in the field. Such technical challenges include maximising performance, durability and cost-effective manufacturing. Innovative measurement methods and modelling are also needed to enhance device functionalities and improve competitiveness. Measurements are needed to characterise the structure of thin films and their novel electronic and thermal properties, and models are needed to help interpret the measurements and relate them to product performance. The complexity of these thin films and the associated measurement challenges requires a coordinated multi-method approach that cannot be achieved by a single institution alone.

The specific measurements needed for the different devices include:

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- Power electronics, based on multilayers of thin film semiconductors: wavelength dependent dielectric function (how a material responds to an electric field), structure
- Photovoltaics, used for the production of solar energy and thin film coated: relationship between structure and performance, product quality control, electrical and optical properties
- Solid state lighting, based on layers of thin films: material performance of large area devices
- Energy efficient windows, coated with low emissivity thin films to ensure high transmittance in the visible part of the spectrum, and low heat transmittance: optical characteristics and thermal parameters
- Thermal solar energy absorbers, coated with thin films: dielectric characterisation, thin film refractive index, effect of temperature stress on thin film performance (temperatures become very high when the transfer fluid is not circulating)

Scientific and technical objectives

The goal of this project was to develop complementary metrology tools for thin film characterisation, and included the following objectives:

1. Development of models for the interpretation of advanced materials measurements and their correlation to product performance.
2. Traceable determination of the correlation between material composition and electronic structure over a broad spectral range. This should include the production of reference standards, calibration samples and reference measurement techniques.
3. Validation of measurement techniques for elemental depth, selectivity and sensitivity for thin film energy materials such as novel compound materials with matrix elemental depth gradients, organic/inorganic hybrids, multi-layered structures and nano-structured surfaces, layers and interfaces.
4. Development of validated methods for the thermal characterisation of thin films as a function of temperature and for multi-parameter characterisation of energy thin film materials under specific stress conditions.
5. Development of large-area characterisation methods for process optimisation in thin-film energy material production, including fast contact and non-contact methods.

Results & Conclusions

Development of models for the interpretation of advanced materials measurements and their correlation to product performance.

A combination of X-ray and optical methods were demonstrated on highly complex thin films used in energy applications, such as solar cells and power electronics. The new data analysis models developed in this project represent a significant advance of the state of the art, and allow reliable characterisation that was not possible before. The new insight into the effect of film anisotropy (the difference in properties when measured along different axes) on optical properties and the ability to determine the depth gradient of composition with high accuracy allows the intelligent optimisation of product performance. These methods are now available for companies in Europe to use. Some project highlights are:

- A new data analysis procedure combining Grazing Incidence X-Ray Diffraction and Grazing Incidence X-Ray Fluorescence (GIXRF) was developed that allows the determination of the in-depth distribution of chemical elements in Copper-Indium-Gallium-Sulphur (CIGS) thin-film solar cells without prior knowledge about the gradient nor restriction on the elemental composition within the thin film. This is crucial for the optimisation and improvement of efficiency of complex thin film solar cells. Funding for future R&D development through a new EMPIR project 16ENG03 HyMET has been secured to transfer this capability into an online process control method during film deposition.
- A new model that allows measurement of complex refractive index of rough samples, such as thin film solar cells, was successfully developed and demonstrated. It includes a new data analysis method to combine Mueller Polarimetry data (a mathematical model) with the real surface roughness allowing

the characterisation of samples where it was not possible before using traditional ellipsometry (an optical technique for investigating dielectric properties). The new method, in a broadband wavelength range (300 nm to 1700 nm) was applied to Copper-Indium-Gallium-Selenide and was able to identify internal stress in selected samples. Internal stress introduces unwanted polarisation dependent absorption that reduces the efficiency of the solar cell. The ability to measure internal stress in such complex thin films allows manufacturers to optimise the manufacturing process to improve performance.

- Innovative ellipsometry models for materials with anisotropic permittivity were developed that allow investigation of optical properties of complex thing films such as those with periodic structures used for increasing performance of solar cells or reducing energy losses in light emitting diodes.

Traceable determination of the correlation between material composition and electronic structure over a broad spectral range.

The expansion of the spectral range for electronic structure characterisation allows for more accurate measurements and facilitates correlation with material composition as it provides a complete image of the electronic behaviour of thin film energy materials under working conditions. The work in this project and the demonstration of applicability of such methods for thin films used in different energy applications highlighted the need for standard and reliable calibration methods for ellipsometry. Significant input from this project has helped to generate a new standard at the German Institute for Standardisation (DIN) and is expected to lead to an international (ISO) standard in the near future. Highlights delivered by the project include:

- A new data analysis method was developed, taking into account a broad spectral range (from ultra violet to infrared) in order to determine traceable optical properties of surfaces and thin films. This method was demonstrated in transparent conducting oxide layers used in a range of energy applications, such as power electronics, solar cells and solid state lighting. Additionally, the effects of UV irradiation on the optical and electronic properties of the layers were analysed. Stability of the optical and dielectric constant is crucial for energy applications as the performance of energy devices rely on these parameters.

- The consortium contributed to an inter-laboratory study between DIN and the International Organisation for Standardisation (ISO) related to the ellipsometry calibration methods developed in this project. This study comprised the traceable determination of dielectric functions of different classes of carbon-based thin layers and will inform the development of new standards.

Validation of measurement techniques for elemental depth, selectivity and sensitivity for thin film energy materials such as novel compound materials with matrix elemental depth gradients, organic/inorganic hybrids, multi-layered structures and nano-structured surfaces, layers and interfaces.

Complex thin film energy materials often have non-uniform composition that directly affects their performance. Such variation in composition is notoriously difficult to measure. This project validated non-destructive new methods for determining elemental depth, selectivity and sensitivity of such complex thin films. In order to improve the accuracy and reliability of the new methods, the project also determined fundamental X-ray parameters of elements of interest for energy applications with significant lower uncertainties. These fundamental parameters, such as absorption cross section and fluorescence cross section, are used for the quantification of X-ray measurements, therefore a reduction in the uncertainty of fundamental parameters means more accurate determination of microstructures via X-ray measurements. The new methods are now available for EU companies to use, and additionally this work resulted in a Calibration and Measurement Capability entry within BIPM for quantification of thin CIGS layers. Some highlights are:

- A new X-ray standing wave method was developed for GIXRF and near edge X-ray absorption fine structure that allows the measurement of depth dependent elemental composition as well as chemical speciation (the distribution of an element amongst chemical species). This information is crucial for the development of highly efficient solar cells and was applied to nanostructured Si solar cells.

- A new measurement facility developed in this project that allows correlation of the effect of temperature and irradiation on parameters of thin films energy materials and is now available for users at the SOLEIL synchrotron, in France. The new instrumentation allows synchrotron-based X-ray Reflectivity and GIXRF of samples under controlled temperatures. To validate the facility optimised measurement protocols were developed to reliably and traceably analyse power electronic materials and transparent conductive oxide stacks under controlled temperature conditions.

- The X-Ray fluorescence yield of the K shell (the principal energy level) of Gallium (Ga) was determined with a low relative uncertainty of 4 %. This significantly improves the quantification of X-ray fluorescence analysis of thin films that rely on fundamental X-ray parameter values with low, reliable and traceable uncertainties. Ga is an important element in CIGS thin film solar cells and this result has allowed better quantification of depth profiling of these compound materials with matrix elemental depth gradients.
- The X-Ray fluorescence yield of the oxygen K-shell was determined experimentally with a significant reduction in uncertainty from approximately 20 % to 5.2% (in comparison with the available literature data) and represents a significant improvement in the current state of the art. This atomic fundamental parameter is important for reference-free quantitative analysis of oxides, which are formed on most surfaces in ambient air conditions and can lead to a loss of performance in energy products.
- X-ray spectroscopy measurements in grazing incidence geometry and analysis of a series of multilayer thin film samples based on dielectrics (high-k) on semiconductors for power electronic application were performed. The combination of different methods allowed the determination of thin film interface quality which is critical to high electronic performance. But it also demonstrated a slight change in substrate strain within the first atomic layers that could potentially lead to reliability issues in the longer term. The ability to perform such measurements and analysis will help manufacturers to optimise the performance of power electronic devices.
- A novel combined Grazing Incidence X-Ray Diffraction - GIXRF measurement method and analysis was developed to allow reliable characterisation of depth profiling. The new method was successfully validated through comparison with cross-section scanning electron microscopy measurements of a series of complex CIGS solar cells.

Development of validated methods for the thermal characterisation of thin films as a function of temperature and for multi-parameter characterisation of energy thin film materials under specific stress conditions.

This project successfully developed and validated a new facility for thermal diffusivity measurements on thin films as a function of temperature. It has also developed facilities, now available for EU companies that allow characterisation of thin film energy materials under controlled stress conditions. Highlights are:

- A new facility was developed for thermal diffusivity measurements on thin films as a function of temperature, from room temperature up to 1000°C. New protocols for temperature and frequency calibration were developed, and a best practice guide written which is now available on the project website and other online platforms.
- A world first measurement facility that allows high resolution mapping of transient photovoltage and transient photocurrent was developed. These measurements are critical for the analysis of performance loss in solar cells and the facility has already been used to identify and classify defects in organic solar cells.
- A leak-free portable environmental chamber that can be coupled to different measurement instruments was developed and is now commercially available. It permits in-situ measurements under different stress conditions, such as well controlled levels of humidity and oxygen, which allows identification of degradation modes and improvement of product lifetime. Through a collaboration with Surrey University, this chamber was used for assessment of the quality of manufacturing of large area printed solar cells (~ up to 100 cm^2). Further engagement with Surrey University and additional collaborators will continue in a new EC funded R&D project (H2020 CORNET).
- It was shown that integration of a portable environmental chamber with Muller Polarimetry can identify causes of performance loss in solar cells due to UV and O_2 exposure. Measurement protocols were also developed that allowed investigation of degradation in-situ via measurement of the chemical binding states using X-ray photoemission spectroscopy.

Development of large-area characterisation methods for process optimisation in thin-film energy material production, including fast contact and non-contact methods.

This project designed, developed and validated a series of novel instrumentation for fast, contact and non-contact large area characterisation of thin film energy materials. These methods provide low uncertainly, high sensitivity and a significant increase in measurement speed. These developments have attracted significant industrial interest, which so far has led to one patent application, consultancy agreements, a new calibration service and a spin-out company being formed. Highlights include:

- A new measurement facility and software was developed for fast photocurrent mapping measurements of homogeneity of solar cells. This method is significantly faster than the current state of the art for photocurrent mapping, and reduces the measurement time from hours to a few minutes, thereby opening up the possibility to implement such a technique as quality control during the manufacturing of photovoltaics.
- A theory translating the microwave implementation of the Electro-absorption interferometry technique to the optical domain was developed. This will allow the future development of an experimental setup for fast, non-contact optical characterisation of thin films over large areas.
- A non-contact, non-invasive, fast optical measurement facility was designed and built that allows optical characterisation of periodically patterned or flat thin films with sub-nanometer uncertainty. Integration of coherent Fourier scatterometry with a large-area robotic system with six-degrees of freedom was achieved which allows measurements over an area of 75 x 75 cm^2 on samples of different shapes and formats.
- A new two-colour Coherent Fourier Scatterometry system was designed, built and validated. It is able to perform optical characterisation of thin films with sub-nanometre uncertainty and is capable of measuring pitches down to 200 nm as well as thickness and refractive indices of materials.
- A non-contact, non-invasive measurement setup for spatial characterisation of performance of solar cells was designed, developed and demonstrated on a range of different photovoltaic technologies. The photomagnetic method allows contactless characterisation of thin films and complete solar cells for identification of defects and reduced performance in small and large area solar cells.
- A set of characterisation tools suitable for non-contact characterisation of the electrical quality of nanostructured thin films and related interfaces has been developed to allow fast mapping of samples up to 8 inches in size. The methods were tested with a variety of thin film samples to optimize the process parameters and from this the best parameters were selected to fabricate high-efficiency Si solar cells.

Actual and Potential Impact

This project developed an innovative and ambitious multi-faceted metrology framework for reliable characterision of thin films in energy applications, significantly progressing the drive to increase efficiency, reduce costs and reduce the energy used.

Dissemination of results

This project engaged with a wide range of stakeholders, resulting in the direct uptake of the technology and knowhow developed. The consortium delivered a series successful events:

- A large 4-day symposium on Analytical techniques for precise characterization of nano materials (ALTECH 2017) at the spring meeting of the European Materials Research Society including dedicated sessions and a workshop to disseminate the results of the ThinErgy project. 140 high quality papers from 23 countries were presented during ALTECH and allowed significant dissemination of project results to the 4000 attendees of the whole conference.
- 4 successful targeted workshops (in Portugal 2015, Germany 2016, UK 2016, France 2017), two on Fundamental X-ray parameters and two on Advanced optical characterisation.
- Summer School on Metrology for Thin Film Materials targeted at early stage engineers and scientists and organised as part of the European Optical Society Annual Meeting (EOSAM) in Berlin in September 2016.

- Four training sessions on novel experimental methodologies for synchrotron radiation to the scientific community were attended by members of the scientific community from higher education and public research organisations.

Twenty four peer-review papers were published and 42 conference presentations were delivered to conferences such as the EU Photovoltaic Science and Engineering Conference (PVSEC) Solar Cell Conference, European Conference on X-ray Spectrometry (EXRS), the International Congress of Metrology and the European Conference on Applications of Surface and Interface Analysis (ECASIA).

In order to facilitate update of know-how developed in the project, the consortium published the following 4 Good Practice Guides:

1. Grazing incidence X-ray fluorescence analysis
2. Photoelectron spectroscopy in the VUV spectral range
3. Calibration of IR photothermal radiometry
4. Ellipsometry measurements on solar cells

These Good Practice Guides are available from the project website, through the technical website ResearchGate and through societies websites, such as the European X-ray Spectrometry Association (EXSA). They have also been presented at key conferences and will be used as teaching material in universities such as the Danish Technical University.

Impact on standards and policy

The consortium actively engaged with standardisation bodies, international metrology committees and policy makers. Results from this project were presented and discussed with the International Bureau of Weights and Measures (BIPM), DIN, ISO and the Versailles Project on Advanced Materials and Standards (VAMAS).

- Part 1 of draft standard (DN 50989-1) on data analysis in spectroscopic ellipsometry was published with significant input from this project and is now available online. Input to other parts of this standard are onoing and discussions with ISO are in place to bring this work into the international level (objective 2).
- The consortium contributed to an ISO inter-laboratory study on elllipsometry measurements of thin films that will inform standards development in the field (objective 2).
- Presentations were delivered to the BIPM's Consultative Committee Task Group for Thermophysical Quantities and to the steering committee of VAMAS to inform future activities in the international arena.
- The consortium contributed to the successful completion of the BIPM CCQM (Surface Analysis) Pilot Study P140 on Quantitative Surface Analysis of Multi-Element Alloy Films and to the BIPM KC125 CCQM Key Comparison on amount of substance, these studies are crucial to ensure international agreement in the measurement of thin films.
- A new Calibration and Measurement Capability (CMC) for the quantification of thin film CIGS layers was registered with and approved by BIPM, the highest international authority in metrology (objective 5).

Results from the project have also informed Government policy. Members of the consortium gave a presentation about metrology strategy to the Board of Experts for the Ministry of Economic Affairs in the Netherlands, and contributed as invited experts in a round-table discussion with the Government Office for Science in the UK on policy for materials for energy technologies.

Early impact

- A new measurement facility that allows correlation of the effect of temperature and irradiation on parameters of thin films energy materials and is now available for users at the SOLEIL synchrotron, in France (objective 3).
- Multiple parameter characterisation under the same stress conditions is crucial for the determination of reliability of performance of new thin film energy materials and is now available at NPL (objective 4).
- Increased solar cell efficiency was demonstrated when compared with current technology in the production line of a German Solar Cell Manufacturer by applying the passivated Si high efficient solar cells thin films developed in this project.

- A patent application for high efficiency Si thin film technology has been submitted and based on this the spin-off company ElFys Inc. has been established and started operating.
- New instrumentation has been made available to end users via license agreements. For instance, a portable environmental chamber that can be coupled to existing instrumentation for on-site characterisation, was developed at NPL and licensed to a company and is now commercially available. It allows in-situ electrical and optical measurements under well controlled environmental conditions and can be coupled to existing measurement instrumentation (objective 4).
- Several NMIs are providing consultancy and other services to large EU companies using know-how developed in this project, this will help with increase competitiveness. For instance, one large European equipment manufacturer has signed a long term consultancy agreement with VSL to develop new instrumentation for thin film characterisation.
- A new guide for calibration of scatterometer measurement is being used to deliver commercial projects (e.g. measurement service) to companies in the EU (objective 5).
- The new CMC entry within BIPM for quantification of thin CIGS layers is now available for European companies (objective 3).
- The project attracted additional collaborations leading to follow-on projects i.e. 16ENG03 HyMET and H2020 CORNET that will further develop results from this project into a wider range of applications. This includes companies contributing to standards development and to development of new instrumentation as well as application of methods developed in this project to new energy materials systems.

Potential future impact

The know-how, new facilities and instrumentation developed in this project will facilitate the development of new technologies and increase competitiveness of European energy technologies. By extending Europe's leadership in energy technology and innovation, this project has helped to ensure economic growth and an energy efficient future for Europe.

List of Publications

[1]. ***Elemental depth profiling in TCO thin film by XRR-GIXRF combined analysis***
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[4]. ***CASTOR, a new instrument for combined XRR-GIXRF analysis at SOLEIL***
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