Jacob J. Lamb Bruno G. Pollet *Editors*

Micro-Optics and Energy Devices



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Jacob J. Lamb • Bruno G. Pollet Editors

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Sensors for Energy Devices



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Preface

More than 80% of our present energy consumption is chemical and non-renewable (coal, oil and gas). The most important renewable energy alternatives (wind and solar energy) produce intermittent electric energy and are also inadequate for most transportation options. The renewable transition will require a collective effort using many different types of energy conversion and storage devices and technologies to entirely remove the dependence on non-renewable fossil fuels. Many technologies have only recently entered the commercial market, with many others still not yet commercialised. This is due to the requirement for technologies to be researched further to understand how they can be improved. Sensors for the measurement and monitoring of energy conversion and storage devices are needed to improve our understanding of such technologies.

This volume intends to provide a brief research source for micro-optical sensors and energy conversion and storage devices, discussing fundamental aspects as well as cutting-edge trends. This volume provides industry professionals, researchers and students with the most updated review on modern energy conversion and storage technologies, as well as micro-optical sensors. This volume aims to help readers identify technology gaps and develop new materials and novel designs that lead to commercially viable non-fossil energy systems.

The editors and authors are grateful to the ENERSENSE programme, the ENERSENSE team and NTNU Team Hydrogen at the Norwegian University of Science and Technology (NTNU) for supporting and helping on this book volume.

Trondheim, Norway

Jacob J. Lamb Bruno G. Pollet Acknowledgements

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Jacob J. Lamb is originally from New Zealand where he got both his B.Sc. and M.Sc. in Biochemistry at the University of Otago, where he worked in a research laboratory at the University of Otago in Dunedin, New Zealand with Professor Julian Eaton-Rye. He came to Norway to undertake a Ph.D. in Biotechnology under the supervision of Associate Professor Martin Hohmann-Marriott, which he completed in June 2016. Jacob then undertook a post-doctoral position under the supervision of Professor Dag Roar Hjelme and Associate Professor Kristian Myklebust Lien looking at photonic sensor technologies for their application in biogas production. Since 2018, Jacob has been a researcher studying sensor technologies and process digitalisation for a variety of applications in biological and engineering fields related to energy. His areas of expertise include sensor technologies, optical spectroscopy, photosynthesis, microbiology, biological and biochemical techniques, electronics and programming, digitalisation, renewable energy, energy storage and process engineering. His research motivation is to improve renewable energy sources, increase sustainability within agricultural and aquacultural industries, develop technologies for climate change mitigation as well as develop ways to measure, analyse and optimise processes. He has also worked on implementing a knowledge base of Li-ion battery manufacturing at NTNU.

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Part I

Optical Properties for Sensors



Introduction

Jacob J. Lamb, Odne S. Burheim, and Dag R. Hjelme

In many recent research projects, research is conducted between several different fields, with experts working together to achieve a common goal. The development of nanostructures for use in battery technologies is one example, where the fields of electrochemistry and nanoscience have come together to achieve a common goal. In order to understand and develop research that integrates multiple fields of research, experts in the required fields must come together. Concerning modelling and simulations, the computation requires variable verification through experiments. This relies on measuring the most critical properties in sufficient detail in terms of numerical and geometrical precision. Electrochemical energy conversion and storage devices are one such case where measurements of high resolution, in regard to their geometrical precision within a device, are required.

Optical fibre-based sensors have the potential for being used on a microscopic geometric scale with very high precision. With electrochemical energy storage devices becoming thinner in order to improve their performance, the size requirements for sensor systems is in the range of $5-500 \ \mu\text{m}$. This book is intended to uncover the possibilities and requirements to integrate the fields of optical fibre-

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based sensors in electrochemical energy storage systems, allowing the improvement of variable resolution in their experimental model verification research.

Dimensions of Electrochemical Energy Storage Devices

Energy is a property defined by mass, geometry and time. The units of energy (e.g. Joule, Nm or kg m²s⁻²) illustrate this. Energy is exchanged by means of heat and work, as defined by the laws of thermodynamics. It can be stored or change form. In electrochemical (and electric) energy storage, electrical work is converted into latent work (a chemical potential) and heat. Heat, beyond reversible heat (T Δ S), is an indication of lost work from various forms of friction during energy conversion or transport processes. This heat (or loss of work) increases with the rate of energy conversion. Energy storage devices convert energy in order to store or release energy, and modern energy storage devices are often developed to convert energy more effectively (faster), rather than with higher efficiency [1].

Energy conversion between electric energy and chemical energy relies on electrochemical cells, such as lithium-ion batteries, supercapacitors, water electrolysis and hydrogen fuel cells [2]. These are reactors where thin layers of active and passive components are assembled in several layers. Typically, the active layers have a thickness below 200 μ m, with many as thin as 10–20 μ m [3–6].

Inside these rather thin layers, the conversion of electrical and chemical energy takes place. Increases in temperature due to irreversible reactions or friction within these materials that generally have a rather low thermal conductivity $(0.05-1 \text{ W K}^{-1} \text{ m}^{-1})$. In terms of chemical processes, degradation mechanisms begin to occur and increase as the temperature increases [7, 8]. These reactions can be traced or sensed by highly localised temperature measurements. Understanding such processes to a high resolution within such small geometric spaces requires applying sufficiently small sensors that are at the same time inert to the ongoing processes. In several cases, optical fibres can be tuned concerning size and detection variable without affecting the quality of the sensor. It is fair to say that optical fibre-based sensors are a developing tool for improved understanding and model verification within electrochemical systems. An overview of the electrochemical technologies and possible optical fibre sizes is shown in Fig. 1.1.

Electrical Versus Optical Sensors

The detection of external stimuli on electronic sensors can often be segregated into the terms active or passive sensing. By the use of an excitation signal, the active sensor will change in response to an external effect and produce the output in the form of the change in current or voltage. A thermistor is a typical example of an active temperature sensor. For passive sensing, there is no need for an excitation signal to produce the current or voltage in response to the external stimuli. Thermocouples are often used as passive temperature sensors. Sensors based on



Fig. 1.1 Overview of different electrochemical devices and possible characterisation technologies with emphasis on optical fibres

electronic circuit systems are convenient, cheap and simplified but have limitations when used for monitoring harsh environments such as oxidising (pH too) fluids, in high temperatures or high pressure, and have a size demand.

Optical fibre (OF) sensors for temperature, pressure and strain sensing have been utilised in many industrial applications in the last decade. Combined with the robustness of the sensor design for monitoring in harsh environments, OF sensors are also immune to electromagnetic interference. Active or passive sensing with OF sensors offers remote multiplexed and multipoint sensing capabilities, simplified design and real-time monitoring of temperature, strain, humidity or concentration of a specific chemical in complex mixtures. Active sensing with OFs utilises a light source such as a laser or a broadband source as the excitation signal that responds to an external stimulus measured by a photodetector. In passive sensor with OFs, the excitation signal or light source is omitted so that only the photodetector detects the background light. Active or passive OFs are organised in intrinsic or extrinsic sensor configurations. Extrinsic OF sensors monitor the medium in the exterior of the OF; whereas the intrinsic OF sensors monitor the interior medium that responds to changes in the exterior medium [9].

General Principles of Optical Fibre Sensor Systems

A sensor is often understood as the transducer alone, the part of the sensor that converts the measured quantity into an electrical signal. In an optical sensor, the transducer creates an optical signal, which then also requires an optical-to-electrical conversion. Figure 1.2 shows an illustration of the complete sensor system.



Fig. 1.2 General components needed for a sensor setup

Although the sensor sensitivity can be defined based on the transducer alone, the resolution and accuracy of the sensor are often dependent on the electronics. For chemical sensors, the required specificity introduces the need for recognition of the correct variable. These analyte molecules typically interact with binding sites or receptor molecules, which triggers a response from the transducer. Together with the transducer, this will be the focus of a significant part of this book, which will be limited to fibre optic sensors.

The transducer shown in Fig. 1.2 can modulate the optical signal in various ways (e.g. intensity, phase and polarisation), based on the source, transducer and detector type used. A common way of doing this is through refractive index (RI) sensing, where the recognition element converts the analyte response into RI changes that are detected by the transducer.

The recognition is typically realised with a stimuli-responsive polymer [10] or surface plasmon-generating layer of metal [11], and typical RI transducers include long-period gratings (LPGs) [12], Mach–Zehnder type interferometers [13] and tilted fibre Bragg gratings (TFBG) [14–16]. These sensors have factor that they excite cladding modes, of which their phase is sensitive to the surrounding RI, in common [17, 18]. The changed phase will result in an interference signal when recombined with the core mode. Sensing of pH can be realised by coating an RI-sensitive sensor with a polymer that changes its optical density based on pH [13, 18]. Linear response both in acidic and alkaline solutions has been achieved using these techniques [13]. Although these sensors are temperature dependent, the sensitivity is an order of magnitude lower than that in LPGs.

Sensor Integration

Interrogation methods and components applied in OF sensor systems are often specially designed for the parameters used for the monitoring. To make the sensor system compatible with existing regulation techniques in fuel cells and electrolysers, the components used for signal excitation, modulation and acquisition should be designed without disrupting and reducing the efficiency of the energy storage system. For an OF embedded in a cathode catalyst layer of a proton exchange membrane fuel cell (PEMFC) for multipoint temperature measurements, the fibre itself will disrupt the thickness of the layer by occupying a space equal to $125 \,\mu\text{m}$ in diameter of a cylinder. With a large surface area of the layers of a PEMFC, an OF embedded in the layer will result in small disruption of the gas diffusion or the transportation of heat.

By monitoring the temperature along the OF (i.e. along a one-dimensional line in the PEMFC), the regulation of the temperature can be controlled by cooling channels along the same line. Therefore, for OFs in a mesh, the temperature of the cooling channels can be controlled along with the same mesh. The inputs and outputs of the signal have to be centralised by a control unit depending on if it is a reflection- or transmission-based OF sensor system. Sensor fusion can be applied in such a mesh system based on a model calibrated for that particular fuel cell in response to controlled temperature changes.

This book intends to give an overview of selected energy conversion and storage devices as well as describing essential optical properties required to be considered when coupling OF-based sensors for parameter detection. Examples of integration of OF sensors into energy conversion and storage devices are also detailed to display the ability afforded with such sensor networks.

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Light Properties and Sensors

Markus S. Wahl, Jacob J. Lamb, Harald I. Muri, Rolf K. Snilsberg, and Dag R. Hjelme

Light as Electromagnetic Waves

Light has the properties of both particles and waves, depending on the situation. In Young's double-slit interferometer, light behaves like waves, creating a pattern similar to what one could make with water ripples in a pond.

On the other hand, the photoelectric effect proves that light consists of particles each with an energy of E = hv, where h is Planck's constant and v is the light frequency. The energy of light with the specific frequency v is quantised as a photon that represents the "particle." The wave–particle duality can be observed in the Youngs double slit and the photoelectric effect.

Light as a wave contains frequency, amplitude and phase; whereas, light as a photon contains quantised energies and different momentum for different frequencies. Light can also be described in the so-called Ray model, where only simple geometrical laws are used to determine the refraction of light in an optical imaging system. Further in this chapter, we will talk about the light as electromagnetic (EM) radiation, where the EM term describes the light containing an electric and a magnetic component in a propagating wave.

The EM wave creates charges and currents when propagating in a dielectric or metal media (e.g. glass or gold), and carries energy and momentum that is dependent

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Fig. 2.1 Electromagnetic spectrum

on the wavelength. The light interacting with charges and currents in materials are also often referred to as EM optics. In EM optics, Maxwell's equations are used to solve the electric and magnetic field vectors for position and time, where the material or the medium properties serve as the input values. The light–matter interactions in this model are explained as an EM wave interacting with charges and currents in the conducting materials.

The electromagnetic spectrum ranges from wavelengths 0.003 nm (gamma rays) to 10^8 m (long radio waves). The wavelength range used for optical sensors can be divided into categories of ultraviolet, visible light that our eye can see, near-infrared (NIR) and mid-infrared (MIR) (Fig. 2.1).

The short and long light wavelengths may interact with materials in different ways. In the MIR or NIR range, the light may interact with the vibrational states of molecules; whereas, in the UV or VIS range the light may interact with the electron clouds of atoms or molecules. For some wavelengths (e.g. in the NIR range), the light can be confined and guided over long distances with small losses in silica-based waveguides. To explain other effects such as interference or diffraction, we can have a look at the electric component of the EM wave expressed as a function of time *t* and space \vec{z} :

$$\vec{E}(\vec{z},t) = \operatorname{Re}\left\{\vec{A} \exp\left[j(k\vec{z}-\omega t)\right]\right\} = \vec{a} \cos\left(k\vec{z}-(\omega t+\varphi)\right)$$
(2.1)

Here $A = \vec{a} \exp (j\varphi)$ is the complex envelope, ω is the angular frequency of the light, $k = 2\pi/\lambda$ is the wavenumber and φ is the phase. The electric wave may be understood well by observing it for *t* and *k* individually. With t = 0 an immobilised electric cosine wave is observed and distributed along \vec{z} with a period determined by the propagation constant *k*. When the value $k\Delta\vec{z} = (2\pi/\lambda) \Delta\vec{z} = 2\pi$, then $\Delta\vec{z}$ represents the distance equal to the wavelength period. The value of 1/k can then be understood as the distance in radians equal to the period of the wave. With $\vec{z} = 0$, we are observing the cosine wave for a fixed point in space with a frequency determined by ω . With $\omega\Delta t = 2\pi v\Delta t = 2\pi$, then Δt represents the time it takes for the wave to complete one cycle. Therefore, ω represents the frequency of the cosine wave in radians.

Given the EM wave, we can now understand the effect of interference by first noting the phase travelled by the waves with the same frequency as $\varphi = kz$. One wave with phase $\varphi_1 = kz_1$ may have constructive interference with another wave with the same frequency but different phase (e.g. $\varphi_2 = kz_2$), so that the difference is $\varphi_2 - \varphi_1 = q2\pi$, where q = 0, 1, 2 are integers. For destructive interference, the difference in phase needs to be half the period of the wave so that $\varphi_2 - \varphi_1 = q\pi$.

The light diffraction effect can be explained given the interference of waves with the same wavelength but a different phase. When EM waves enter a slit or an obstacle with normal incidence, the waves are refracted for many angular degrees θ . Here, the phase for each wave can be represented by phase $\varphi = k \sin \theta z$, where θ ranges from 0 to 90 degrees. The difference in propagation lengths z and in angular degrees θ creates phase differences $\varphi_2 - \varphi_1$, with $q2\pi$ as constructive interference and $q\pi$ as destructive interference. For EM waves with different wavelengths, there are now three variables changing the phase $\varphi = k \sin \theta z = (2\pi/\lambda) \sin \theta z$, and the interference will not only vary with the θ and z, but also with the colour of the light. One example of polychromatic light diffraction is the reflection of light by a CD or DVD that appears as a rainbow to the eye.

Despite this, light refraction by a prism is due to another effect that is different from diffraction. Each EM wave with different wavelengths will propagate in the prism with a different RI so that phase $\varphi_1 = k_1 \sin \theta_1 n_1 z = (2\pi/\lambda) \sin \theta_1 n_1 z$, is different to $\varphi_2 = k_2 \sin \theta_2 n_2 z = (2\pi/\lambda) \sin \theta_2 n_2 z$. When white light is incident on the prism, the light will refract with different angles for different wavelengths. A wave with a normal incidence on the prism will have the phase at the air-glass interface as $\varphi_{air} = \varphi_{prism}$, so that $(2\pi/\lambda) \sin 90 n_{air} z = (2\pi/\lambda) \sin \theta_{refract} n (\lambda) z$. It can be observed from $\theta_{refract} = n_{air}/n (\lambda)$ that the refraction angle $\theta_{refract}$ will with be dependent on wavelength due to $n (\lambda)$, which will change with different wavelengths.

Absorption, scattering, refraction or interference of light can be measured by detecting its intensity or energy for one or several wavelengths. There are several definitions of intensity or energy of light. Radiant flux expresses the energy emitted per unit time (W), and spectral flux expresses radiant flux per frequency or wavelength (W/Hz). To include the angle of incidence of light we can express radiant or spectral intensity, which is flux per steradian (W/sr) or flux per steradian and wavelength (W/(sr Hz)). Lastly, we have radiance or spectral radiance that takes into account the radiant or spectral intensity reflected, transmitted or received by a surface area of m^2 (W/(sr m²)) and (W/(sr Hz m²)), respectively.

Mathematical Formalism

The following section will discuss the mathematical formalism of EM fields. The main areas of focus will include field strength and intensity, wave interference and polarisation.

Field Strength and Intensity

As described in Eq. (2.2), the electric field of a light wave can be expressed as:

$$\vec{E}(\vec{z},t) = \operatorname{Re}\left\{\overrightarrow{E_m}(z)\exp\left(i\omega t\right)\right\}$$
(2.2)