Towards Efficient Photovoltaic Devices

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Key Facts and Experiments on Dye Sensitised Solar Cells

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“Make visible what, without you, might perhaps never have been seen.”

—Robert Bresson (1901-1999)

In the memory of my great-grandfather Prof. Dr Petre Andrei – scholar, sociologist, philosopher, professor, WWI fighter and minister for education – whose collection of authored books, papers, courses, studies, articles, reviews and reports inspired me to bring my own contribution to science.

“In the books that I have wrote, my attitude and philosophy are clearly shown. Check and you'll see.”

—Petre Andrei (1891-1940)

This book is dedicated to my wife Catrinel, my parents Catalina and Dan, my sister Raluca, my grandparents Bibi and Bicu, my aunt Marianne and uncle Puiu.

Dublin, March 2017

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PREFACE

Dye sensitised solar cells (DSCs) represent a novel class of photovoltaic devices that were invented by Michael Grätzel and have been brought at present to a highly competitive 15% efficiency. Among other components, the DSCs typically include a mesoporous titanium dioxide scaffold, sensitised with an adsorbed dye, as the main active element responsible for the photon absorption and charge separation functionalities.

The research conducted and presented in this book is focused on optimising the dye sensitised solar cell efficiency. The methods combine both experimental and computational studies, with two main goals: the investigation and optimisation of the DSC photoanode sintering process and secondly, the plasmon enhanced dye absorbance and charge generation for the DSC efficiency enhancement.

The plasmonic enhancement of dye sensitised solar cells was achieved herein via the chemical functionalisation of the titanium dioxide scaffold with a tailored size-distribution of gold nanoparticles, using an in-house developed chemical method. The aim was to efficiently integrate gold nanoparticles that exhibit intense absorption due to surface plasmon resonance in the visible region of the spectrum, thus increasing the DSC efficiency. Scanning electron microscopy was used to characterise the topography and dimensions of the layers, and in conjunction with a precision focused ion beam it provided access to the otherwise buried layer structure of the DSC. Raman spectroscopy and X-ray photoelectron spectroscopy were used to chemically characterise the layers of this nano-assembly, including confirmation of the presence of the gold nanoparticles on the titania layer, and to investigate their properties. Absorbance measurements and simulations, together with current-voltage efficiency tests for top performing ruthenium dyes used herein, confirmed the finding that the developed method of incorporating gold nanoparticles into photoanodes has an impressive enhancement effect. These results are additionally compared to a theoretical approximation simulation and the increase in absorbance closely matches the experimental data. The studies conclude with a substantial increase of up to 16.2% in dye sensitised solar cell efficiency.

The second goal of the research presented within this book is the study of the sintering process for dye sensitised solar cell efficiency optimisation. The sintering process employed in the active layer fabrication plays a crucial role in the formation of the nanoparticle scaffold and hence in the performance of a dye sensitised solar cell, as it allows the particles to form efficient inter-crystalline electric contacts providing high electron conductivity. Furthermore, the dye solar cell design requires a conductive transparent top electrode, which is typically made of fluorinated stannic oxide. The present research reports on a scanning electron microscopy study in conjunction with focussed ion beam milling and energy dispersive X-ray (EDX) mapping of the distribution of all relevant elements within a DSC subsequent to a classical sintering process in the range of 350°C–550°C. Additionally, this study provides quantitative results regarding the found stannum (Sn) diffusion, from the FTO onto the titania layer, and its effect on DSC efficiency is confirmed via current-voltage measurements. The effective spatial resolution of the EDX studies was calculated by Monte Carlo simulations of the electron trajectories and X-ray emission region. This permitted the construction of a model for the migration of the Sn from the transparent conductive oxide onto the titanium dioxide scaffold, resulting in alterations in the composition of the complex system, which has a direct effect on the dye solar cell performance. Current-voltage measurements conclude that the sintering temperature of 500°C is close to the optimum regarding Sn diffusion enhancement of dye solar cells. Sintering temperatures above 500°C were causing a drop in the efficiency and are therefore not recommended. In order to optimise the efficiency, the results are summarised by a model that explains how the efficiency varies with the diffusion process.

Both the Sn diffusion during the sintering process and the gold nanoparticle enhancement studies presented within this book, make a valuable contribution to dye sensitised solar cell development and efficiency optimisation. They pave the way for an enhanced efficiency dye sensitised solar cell that can benefit from the novel findings presented here.

CHAPTER ONE

INTRODUCTION

1.1 Motivation

The motivation of the present book originates from the fusion of three major factors: 1. the fact that fossil fuels are limited resources anticipated to be depleted within decades; 2. the fact that the future does not have to look "black" by employing only fossil fuels in energy production, when in fact it can look "green" thanks to present renewable energy alternatives; and 3. the fact that Solar energy is the only renewable resource capable of supplying, *per se*, today's total global energy demand. Each of the above-mentioned motivating factors is succinctly described in the following three subsections.

1.1.1 The depletion of fossil fuels

Fossil fuels have naturally formed throughout millions of years; they contain large percentages of carbon and can be divided into three main categories: coal, oil and natural gas. The biogenic theory that explains the formation of these fuels from anaerobic decomposition and the fossilization of dead organisms followed by prolonged exposure to heat and pressure within Earth's crust, was introduced in the 16^{th} century by the German scientist Georg Pawer (regarded as the founder of mineralogy). A significant milestone for what has today become a fossil fuel based industry was set by the Scottish inventor James Watt back in the 18^{th} century. Watt's steam engine proved to be a more efficient version of the Newcomen steam engine and substantially contributed to the Industrial Revolution of the 18^{th} and 19^{th} centuries. Together with growing fossil fuel explorations in the industrial development era, the CO_2 concentration levels have unfortunately also increased, and are continuing to increase accordingly with fossil fuel exploitation.

The fossil fuels have a number of acknowledged disadvantages that include: the induced increase in the concentration of greenhouse gases due to the increase in CO_2 concentration; the environmental disasters occurring

from the too-frequent oil spills; and geopolitical instabilities due to growing prices and higher production demands. One recent environmental disaster occurred in the Gulf of Mexico in 2010 because of a five-month oil spill (April to September 2010). However, even if the above-mentioned disadvantages are often ignored or sometimes hidden, the fact that fossil fuels are finite resources is well known and cannot be ignored. A study by Cho[1] published in 2010 explains why humanity should depart from using fossil fuels, starting from their finite status and the production of planet-warming greenhouse gases. Figure 1.1 reinforces this by revealing the data for the known remaining reserves (the amount of measured resources that could be expected to be exploited) and for the potential maximum resources in terms of year-production as an extrapolated approximation.

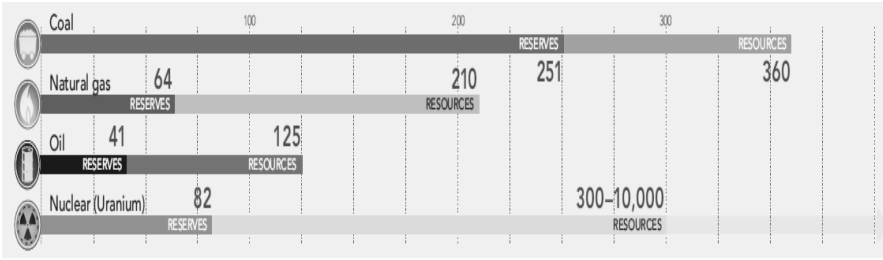

Figure 1.1: Coal, natural gas, oil and uranium resources available on Earth in 2010[1] (estimated in number of years yet available). Image adapted from [1].

With these clear arguments in mind, it can be concluded that humanity has to focus on reliable sources of alternative energy. Efforts in this direction are already being made by numerous developed countries, but this process has to be accelerated and sustainable. The advantages of using renewable energy and the challenges that have to be addressed are succinctly discussed in the following motivational subsection.

1.1.2 Renewable energy resources

Renewable energy supply is the only viable option for the future that can sustain Earth's fast growing population of 7 billion people and its expanding energy consumption. Renewable resources include hydropower, wind power, biomass, solar (photovoltaic and thermal), geothermal and ocean (tidal). The total amount of renewable energy available today accounts for less than 20% of the global energy supply. The threshold of

20% energy from renewable sources is the European Union target for 2020, and globally renewable sources are aiming to contribute 30% of the total energy supply by 2035. However, there is a continuous need for governments' policies and incentives to boost the implementation of renewable energy, and the population increase factor has to be taken into account.

The fact that the above-mentioned renewable resources are widely available, free, clean sources of energy has been highly motivating the research presented in this book. Although non-renewable, nuclear fusion (e.g. the ITER tokamak project in Cadarache, France) could provide humanity with energy by combining, for example, deuterium (a hydrogen isotope, highly naturally available from the oceans) with tritium (a hydrogen radioactive isotope obtained from lithium). Although nuclear fusion is not yet usable, it is safer than the non-renewable uranium-based nuclear fission that produces large amounts of nuclear waste, geopolitical tensions and nuclear-plant disasters (e.g. Chernobyl 1986 and Fukushima 2011). The world's nuclear energy dependency and its associated potential disasters have also contributed to the motivation of this book's research on the sun's renewable energy. The third motivation factor is in fact the highly available solar energy and it is briefly described in the following subsection.

1.1.3 Solar energy

The sun is a source of practically unlimited, pollution-free energy, from which the Earth receives about 174 PW[2] of solar radiation at the upper atmosphere. Part of this total solar radiation is reflected back by the atmosphere (6%), by the clouds (20%) and by Earth's surface (4%), while 20% is absorbed by the atmosphere and the remaining 50% is absorbed by oceans and land masses. It can therefore be calculated that the solar radiation reaching Earth annually (87 PW) is approximately 5000 times higher than the present global energy supply (15.58 TW in 2006, 16.13 TW in 2009, and 17.4 TW projected for 2012[3]). This leads to the following scenario: covering 0.2% of Earth's surface with 10% efficiency photovoltaic devices could supply the world's current energy requirements (0.2% x 10% x 87 PW = 17.4 TW). This scenario is depicted in Figure 1.2, exemplifying six potential locations distributed on desert lands in: USA, China, Australia, Africa, South America and the Middle East.

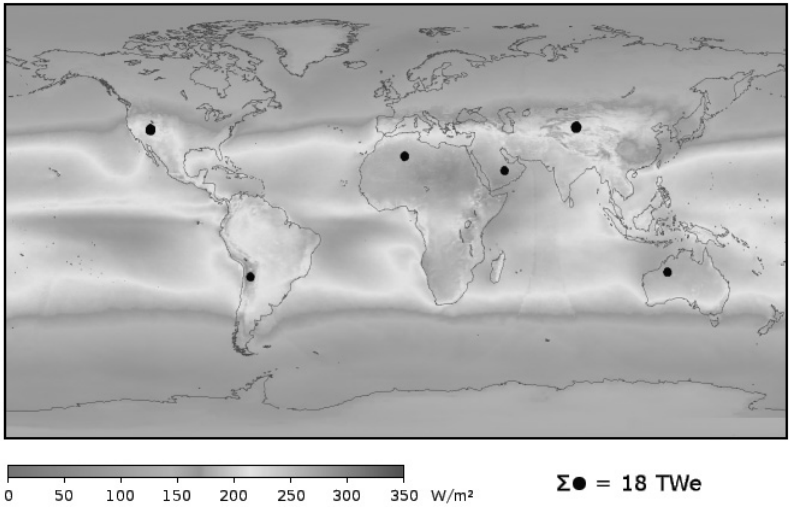

Figure 1.2: Photovoltaic panels covering the marked land areas can provide the total world energy demand[4]. Image adapted from [4].

In another motivating calculation, the solar energy absorbed by land and oceans in less than two hours (50% of 174 PW annually = 87 PW on Earth per year/365 days = 238 TW per day/24 hours = 9.9 TW per hour) provides the current annual energy consumption. While it is not possible to capture all, or even most of this solar energy, capturing less than 0.02% (0.02% x 87 PW = 17.4 TW) that falls on Earth's surface would be enough to meet current energy needs. Figure 1.3 is an example of the total resources currently available on Earth, and opposed to the depleting fossil fuels and uranium resources, the renewable annual sun energy is 5000 times higher than the present energy supply. Solar energy conversion is a growing technology, which is becoming a more and more important way of tapping into nature's renewable energy resources. However, to replace fossil fuels, most experts suggest the use of a combination of energy sources and technologies in a sustainable fashion.

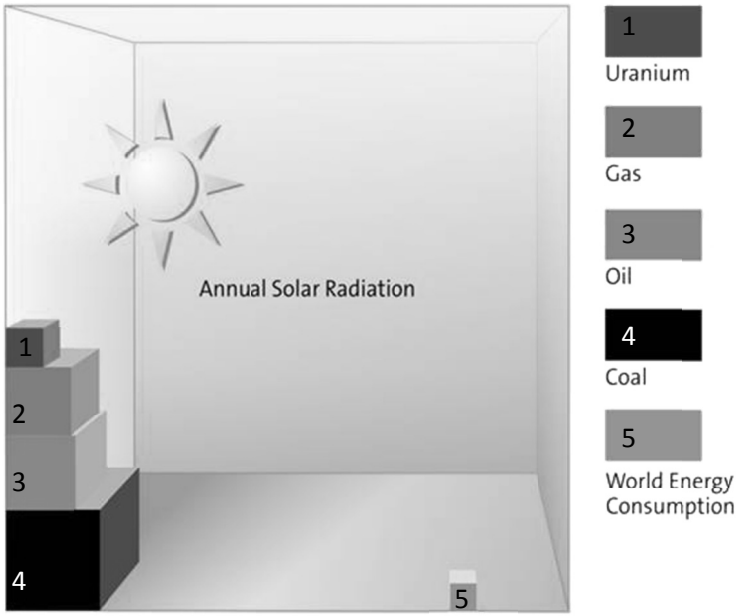

Figure 1.3: Relative dimensions of resources available on Earth[5]. Image adapted from [5].

1.2 Book outline

The book is structured in six chapters. Chapter 1 introduces the book by describing the motivation and the outline. In Chapter 2, the fundamentals of the dye sensitised solar cells (DSC) are considered and presented. The chapter starts with a short historical background and efficiency timeline, followed by some DSC commercial applications. The road from single solar cells to solar panels is discussed together with the costs, stability and present applications, development status and challenges. Special interest is given to the dye sensitised solar cell's key components that are individually reviewed in this chapter. The dye sensitised solar cell principles of operation are then presented and conclude the chapter.

Chapter 3 describes the experiments and methods used in the present research. The choices of materials, multiple setups and devices used in the investigations are presented and described. The description of the analysis

devices includes: the atomic force microscope, the scanning electron microscope, the focussed ion beam, energy dispersive X-ray spectroscopy, Raman and X-Ray spectroscopes, and the vacuum thermal evaporator, together with optical and electrical efficiency testing setups.

Chapter 4 demonstrates a substantial and stable increase in the efficiency of ruthenium based dye sensitised solar cells via plasmonic enhancement. This was achieved by developing a chemical procedure of functionalising the titanium dioxide (TiO_2) film with gold nanoparticles (AuNPs) via short, stable sulfur linkers. Scanning electron microscopy (SEM) was used to describe the topography and in conjunction with a precision focused ion beam (FIB) it provides access to the otherwise buried layer structure of the DSC. Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) are used to chemically characterise the layers of this nano-assembly, including the confirmation of the presence of the AuNPs and to investigate their properties. Absorbance measurements for the top performing dye Ru N719 confirm the finding that the passivated AuNPs have an impressive enhancement effect that is additionally compared to and confirmed by the theoretical discrete dipole approximation simulation. In summary, the presented system shows an increased performance compared to non plasmonic enhanced ruthenium dye solar cells as will be described in this chapter.

Chapter 5 presents a spatially resolved scanning electron microscopy study in conjunction with focussed ion beam (FIB) milling and energy dispersive X-ray (EDX) mapping of the distribution of all relevant elements within a DSC subsequent to a classical sintering process in the range of 350°C–550°C. It is shown here that the Sn of the transparent conductive oxide (TCO) migrates into the TiO_2 scaffold, resulting in alterations in the composition of the complex scaffold, which has a direct effect on the DSC performance. The experiments confirmed that up to a certain sintering temperature, the rate of Sn diffusion can be efficiently used to optimise DSC efficiency. This is described in a model that explains how the Sn diffusion process was used herein to optimise DSC efficiency.

In Chapter 6, the major facts of each chapter together with the research results achieved in this book are summarised and concluded.

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CHAPTER TWO

DYE SENSITISED SOLAR CELL FUNDAMENTALS

2.1 Introduction

Since the discovery of the photovoltaic effect in 1839[1] by the French scientist Alexandre Edmond Becquerel, researchers have been working on creating and improving photovoltaic devices that can efficiently harvest the 174 petawatt[2] of solar energy available. Becquerel's observation that a current passes between two platinum electrodes immersed in an electrolyte in the presence of sunlight, has paved the way for photography,[3] and for photovoltaic devices that were originally based on selenium[4, 5] and later on silicon[6, 7] solar cells.

Invented by the Swiss scientist Michael Grätzel[8, 9] from the École Polytechnique Fédérale de Lausanne (EPFL), the dye sensitised solar cell (DSC) became the prototype of a new generation of thin film solar cell devices. The DSC is based on a nanoparticle photoelectrode functionalised with a light-absorbing dye, and offers a promising alternative to silicon solar cells, due to advantages that include: low fabrication costs in high production volumes and widely available inexpensive materials;[10] the choice of both rigid and flexible designs, various shapes and transparency levels to suit domestic devices or architectural/decorative applications;[10] a conversion efficiency that does not decrease with rising environmental temperatures up to 60^{o}C;[11] and a low level of angular dependency and thus the ability to efficiently harvest diffuse light.[9] This thin film solar cell consists of a mesoporous, semiconducting nanoparticle film onto which a monolayer of dye is adsorbed, an electrolyte, a catalytic layer and two electrodes, one of which should be transparent (Figure 2.1).

In the process of switching from fossil fuels to renewable energy, as motivated in Chapter 1, there is a need to develop new materials and improved concepts and methods for solar energy conversion. Among other types of solar cells, the dye sensitised solar cell is one of the most promising technologies for the twenty-first century due to the above-

mentioned unique advantages. However, there are a number of challenges that need to be addressed which leave room for more improvement as will be described in the present book.

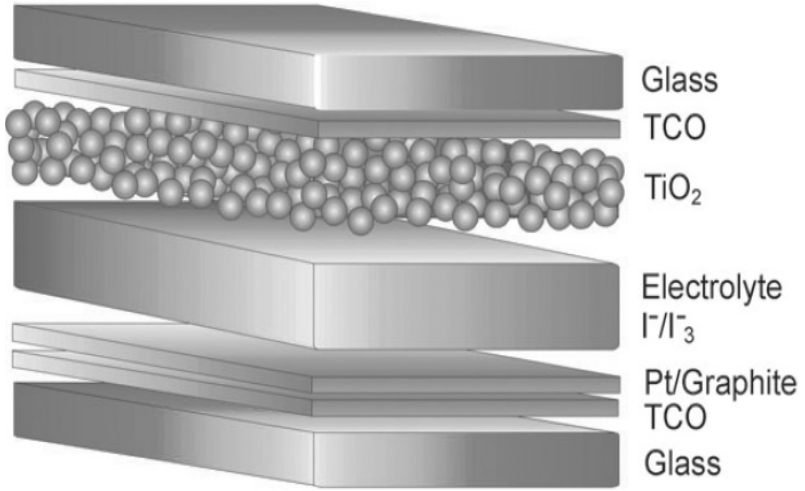

Figure 2.1: The schematic of the dye sensitised solar cell design.

The present chapter starts by exposing the key events in the DSC history, together with some commercial applications and future prospects. The key components of DSCs are reviewed and are the focus of this chapter that concludes with the DSC's principles of operation.

2.2 Historical background and efficiency timeline

The DSC efficiency reached a record value of 15%[12] that was confirmed under simulated air mass 1.5 sunlight in July 2013 by the EPFL group of Switzerland. The DSC origins date back in the 1960s, when it was shown by Gerischer et al.[13] that photo excited dye molecules can inject electrons into the conduction band of semiconductor substrates. In the 1970s and 80s, followed the idea to adsorb the dye on the surface of the semiconductor,[14, 15] and the use of dispersed particles[16, 17] came as another important feature in 1984-1985. Although semiconductors like ZnO sensitised with dyes like cyanine[18] have been tested by Namba et al.

since 1965, TiO_2 has become the main semiconductor[19] employed in the DSC.

The dye sensitised solar cell foundations can be attributed in particular to the research produced by Gerischer and Willig,[20] Tributsch,[21, 22, 23] Spitler,[24] Calvin,[22, 24] Fillinger and Parkinson[25, 26, 27] and their co-workers. Gerischer, Tributsch and Calvin pioneered the field in the 70s with their studies on photosynthesis mimicking by using ZnO sensitised with chlorophyll.

Figure 2.2: Left: the first DSC embodiment (1988). Right: SEM picture of the TiO_2 film used in the first DSC. Image adapted from [19].

Figure 2.2 presents the first DSC embodiment, created by Vlachopoulos, Liska, Augustynski and Grätzel in 1988. They were using TiO_2 sensitised with the "yellow" ruthenium dye (RuL_3) and a platinum wire mesh within a beaker containing the electrolyte. The open circuit voltage was 1V and the dye absorption maximum was 470 nm, yielding a 1% efficiency DSC at the time. The efficiency remained limited to this value up to 1991, when O'Regan and Grätzel reported a 7.1% DSC efficiency record.[9] They achieved this major contribution to DSC history by introducing nanoporous TiO_2 electrodes that had a roughness factor (defined as the ratio of the actual area of a rough surface to the geometric projected area) higher by 1000 times compared to the value used up to that date. Thus, the TiO_2 area available for the dye adsorption was highly increased, and therefore the light harvesting efficiency was highly enhanced. An increasing number of inorganic dyes[28-30] have been synthesised and multiple organic dyes[31-33] with high efficiencies have been embedded into DSCs. The electrolyte, mainly based on the iodide/triiodide mediator, has also been continuously optimised.[34, 35] Titanium dioxide has remained the most used semiconductor of the DSC technology and has been continuously

researched and improved over the last two decades (e.g. the $TiCl_4$ pre~ and post~treatments[36]).

Research on the DSC has grown steadily since its invention, and after the major leap in 1991, it received some growth boosts as described in the following. Figure 2.3 displays the number of published articles worldwide each year between 1985 and July 2013, containing simultaneously the keywords "dye", "solar" and "cell" in the title. From less than 10 articles in 1985 to more than 1250 peer-reviewed articles in 2012, the number of DSC articles has grown steadily. Although this is not an exact count of the number of DSC papers (around 7114 articles found in total), it is a good indicator of its growth to date.

Another relevant example of the growth in DSC research interest is the exponential increase in the number of total citations for each year presented in Figure 2.4. The data were collected in July 2013 from the ISI database[37] by searching for articles with the keywords "dye", "solar" and "cell" in the title. For these articles it was found that the sum of the times cited is approx. 157612, out of which, excluding the self-citations (74856), 82756 citations are non-self-cited.

One major growth boost came in 1993, when Nazeeruddin et al. reported a 10.3%[28] DSC efficiency by using the so-called ruthenium N3 dye. This remained the record in DSC efficiency until 2001, when Nazeeruddin et al. embedded the ruthenium "black dye" (also known as N749) and reported a value of 10.4%.[38] Wang et al.[39] reported four years later a 10.5% efficiency record, achieved in black dye sensitised cells when using an acid (HCl) TiO_2 pre~treatment. Further improving the DSC based on the N749, Chiba et al.[34] introduced larger particles (400 nm in diameter) within the TiO_2 and reported an 11.1% efficiency. In 2005, Nazeeruddin et al.[40] reported that a new ruthenium dye, N719, derived from the ruthenium N3 dye achieved a validated 11.18% efficiency record. After 6 years of DSC research worldwide, a breakthrough[41] achieved by Grätzel's EPFL group in the fall of 2011, set a new world record in efficiency: 12.3% for a single DSC. This record was achieved by using a cobalt based redox electrolyte in conjunction with a zinc porphyrin dye (YD2-o-C8) as the sensitiser. Another record on DSC efficiency was set in the summer of 2013 at 15% due to a new fabrication process developed by the team in EPFL. The new solid-state DSC uses a perovskite material as photon antennae and an organic hole transport material to replace the cell's electrolyte.

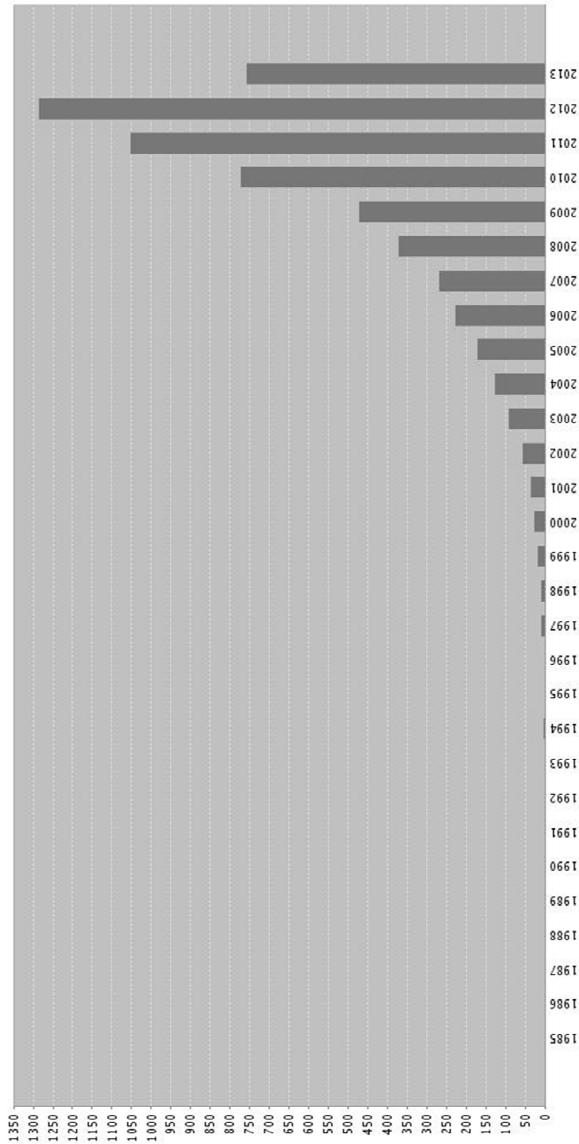

Figure 2.3: The number of publications (approx. 7114) on DSCs in each year from 1985 to July 2013. The data were obtained by searching the Institute for Scientific Information (ISI) database. [37] The keywords used were “dye”, “solar” and “cell”, and the document type was limited to “article”.

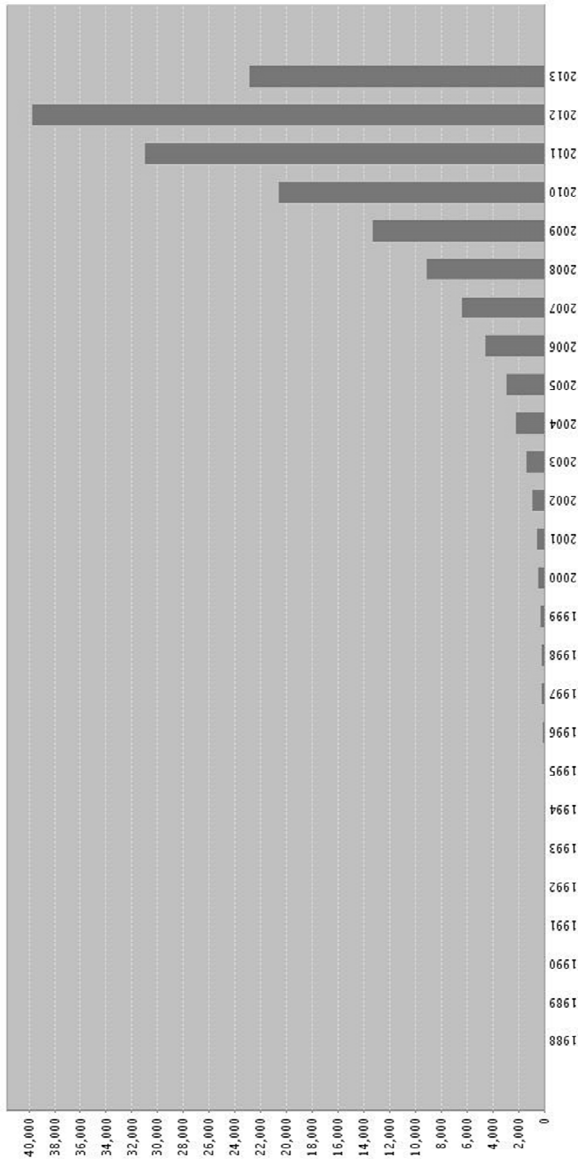

Figure 2.4: The number of total DSC article citations, in each year, from 1985 to July 2013. The data were obtained by searching the ISI database [37].

Variations to the original DSC setup, together with multiple optimisations, are the focus of recent research developments: the use of flexible electrodes,[42] polymers,[43] hole conductors,[44] gel electrolytes,[45] and multi-junction devices as well as multiple exciton generation in quantum dots,[10] shows promising results for future applications. Tandem DSCs have currently exceeded 16% efficiency.[46] DSC modules of 8.4%[47] efficiency have been reported by Sony Corporation, which has also predicted a 9.9% module efficiency. The DSC efficiency and stability have been continuously improved over the last decades, but there is room for much more improvement. A relevant and motivating example was given by Polman and Atwater in 2012,[48] who reported that the Shockley-Queisser thermodynamic limit of energy conversion efficiency of 33.7%[49] in single junction solar cells can be surpassed by using plasmonic enhancement in photovoltaic conversion. The Shockley-Queisser limit is calculated by analysing the amount of electrical energy that is extracted per incident photon. It takes into account three considerations: blackbody radiation (the blackbody radiation from a solar cell at room temperature cannot be captured and represents approx. 7% of available incoming energy), recombination (these processes reduce efficiency under STC with an extra 10%), and spectrum losses (approx. 49% as not all of the incident photons are converted).[49] A plasmonic DSC efficiency enhancement approach that can pave the way in this direction, is presented in Chapter 4 of the present book. While the 15% DSC efficiency is at only 44.5% of the Shockley-Queisser limit for a single junction, new advancements could bring the DSC efficiency closer to the higher efficiency photovoltaics (e.g. 25%[50] for crystalline Si and 28.3%[50] for thin film GaAs, and both for a non-concentrated single junction under STC). From this point, the performance/price ratio for DSCs would exceed the other photovoltaic devices.

From 1% before 1991, to 15% in 2013, the DSC has become efficient enough to emerge on the market. Although higher efficiencies are more often achieved in laboratories, some companies worldwide can produce DSCs with an efficiency above 10%. Therefore, the first DSC applications and end-products have recently emerged on the market. Some of the DSC commercial applications and challenges that need to be addressed are discussed in subsection 2.3.

2.3 DSC commercial applications

Since 1991 when Grätzel and O'Regan published the 7.1% efficiency breakthrough, the DSC has been continuously researched and interest has

steadily increased as shown in Figure 2.4. However, the design and materials employed in DSC manufacturing remained practically the same: substrates, a semiconductor (nominally TiO_2), dyes, a catalyst and an electrolyte. Nevertheless, DSC efficiency is the key factor that has increased and thus allowed the first DSC applications to be commercially envisaged and finally produced.

A major factor for DSC development and commercial availability is long-term stability. Based on DSC kinetics studies, M. Grätzel concluded in 2006[51] that a 20-year DSC lifetime is achievable based on laboratory tests. However, in order to achieve long-term stability while maintaining high efficiency in an "outside-of-the-lab" environment, there is a clear need to select stable dyes and electrolytes and robust sealants. Based on this and on recent advancements, DSC technology has found a number of applications in the photovoltaic industry.

The photovoltaic industry contains five major market segments: grid connected solar farms, consumer products, building integrated photovoltaics (BIPV), remote industrial applications and remote communities. Out of these five segments, consumer products and building integrated photovoltaics are the two foremost DSC developed sectors. In the consumer products market, the available DSC applications include: a solar charger, either as a stand-alone (e.g. for mobile phones or laptops) or part of other products (e.g. backpack, bicycle panniers), a wireless keyboard and windows powered shade. The UK based G24 company is a major player in this area and is currently developing a number of applications addressing both indoor and outdoor power solutions. Their commercial products were the first DSC end-products worldwide and were shipped in October 2009. Figure 2.5 shows some of the previous G24 products: a DSC solar charger backpack and a wireless DSC powered keyboard.

Figure 2.5: Two examples of DSC products: a solar backpack (left) and a wireless keyboard (right).[52]