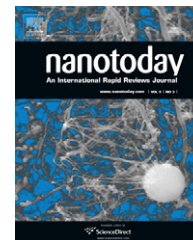


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## RAPID COMMUNICATION

# High efficiency solid-state sensitized heterojunction photovoltaic device

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Sensitizer;  
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Charge  
recombination

**Summary** The high molar extinction coefficient heteroleptic ruthenium dye, NaRu(4,4'-bis(5-(hexylthio)thiophen-2-yl)-2,2'-bipyridine) (4-carboxylic acid-4'-carboxylate-2,2'-bipyridine) (NCS)<sub>2</sub>, exhibits certified 5% electric power conversion efficiency at AM 1.5 solar irradiation (100 mW cm<sup>-2</sup>) in a solid-state dye-sensitized solar cell using 2,2',7,7'-tetrakis-(N,N-dimethoxyphenylamine)-9,9'-spirobifluorene (spiro-MeOTAD) as the organic hole-transporting material. This demonstration elucidates a class of photovoltaic devices with potential for low-cost power generation.

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The dye-sensitized nanocrystalline solar cell (DSC) offers special opportunities in the realm of renewable energy sources primarily stemming from its use of low-cost mate-

rials and its lack of stringent conditions required for device manufacturing [1]. In the quest for new sensitizers leading to an enhancement of the photon-to-electron conversion efficiency, the creation of new ruthenium dyes still remains a promising option. Indeed, recent improvements in the design and synthesis of new dyes have made it possible to obtain greater than 11% light-to-energy conversion efficiencies. The majority of these newly developed sensitizers involve polypyridyl-type complexes of ruthenium [2–4]. Advantages in terms of improved sealing properties and long-term cell stability can be realized by replacing the liquid electrolytes utilized in DSC by a solid-state

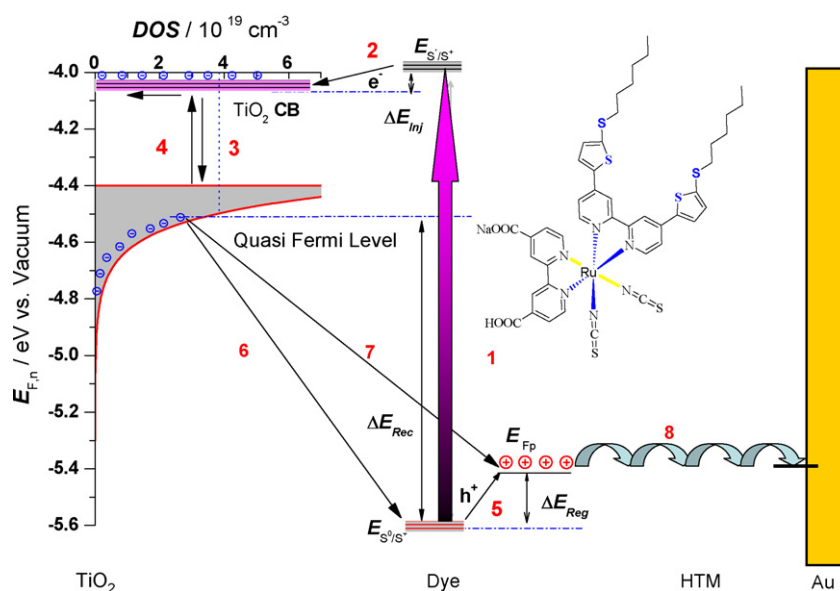
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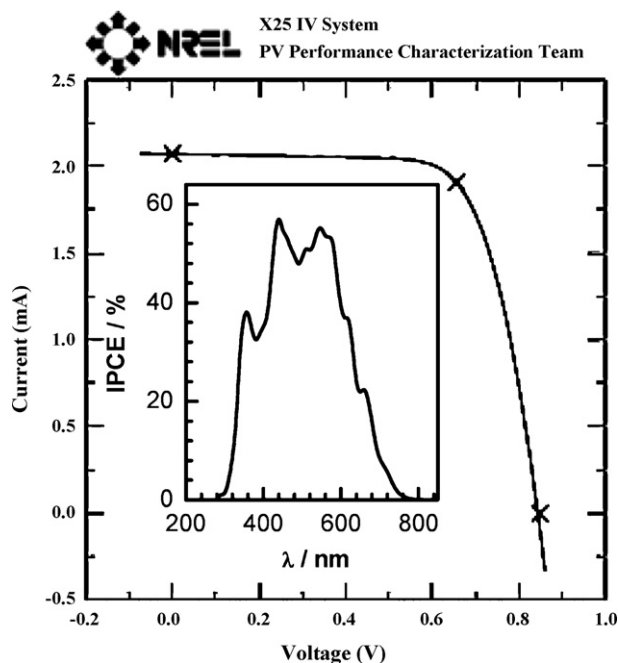


**Figure 1** Schematic illustration of the electron-transfer processes occurring in the solid-state dye-sensitized heterojunction: process 1, light-induced excitation of the chromophore; process 2, electron injection into the conduction band of the TiO<sub>2</sub> semiconductor; processes 3 and 4, trapping and detrapping of electrons in the TiO<sub>2</sub>; process 5, dye regeneration via hole transfer to spiro-MeOTAD; processes 6 and 7, the direct recombination of primarily separated charges and the secondary recombination process between the injected electron and the oxidized spiro-MeOTAD; process 8, holes hopping in the HTM. Also shown are band energies of different components. The inset shows the molecular structure of C106 dye.

hole-transporting material (HTM) to fabricate solid-state dye-sensitized solar cells (SSDSC). The initial successful demonstration of the feasibility of making SSDSC devices reported in 1998 announced a 0.74% power conversion efficiency [5]. Recent advances in the photovoltaic performance of SSDSC have augmented the power conversion efficiencies obtained to the range of 4–4.8% [6–11]. There are two key issues to address in order to increase the relatively low efficiencies found in SSDSC. One basic problem is the poorer light harvesting found in solid-state devices compared to that found in liquid-based devices, resulting from the use of a thinner nanocrystalline film (i.e.,  $\cong 2\text{--}3\ \mu\text{m}$ ) in the fabrication of the former devices. The other impediment to producing more efficient SSDSC devices is the rapid recombination dynamics found at the dye-sensitized heterojunction (see Fig. 1) [12,13]. Some approaches have been proposed to improve light harvesting and to reduce interfacial recombination, including the development of new sensitizers and coadsorbents [12–18]. It is well documented that sensitizers containing hydrophobic spacers work efficiently in DSC devices [14–18]. Tuning the length of the hydrophobic spacers is an important aspect in the design of new sensitizers. This optimization has been previously realized by incorporating a  $\pi$ -conjugation linker molecule such as a thiophene or a thienothiophene derivative into the chromophoric framework [14–18].

A promising molecular engineering approach to overcome the dilemma of low light harvesting due to the thinner titania films used in SSDSC (utilized to ensure adequate pore filling) is to increase the molar extinction coefficient of the sensitizer employed. Herein, we report on high efficiency SSDSC devices based on a ruthenium complex sensitizer, NaRu(4,4'-bis(5-(hexylthio)thiophen-2-

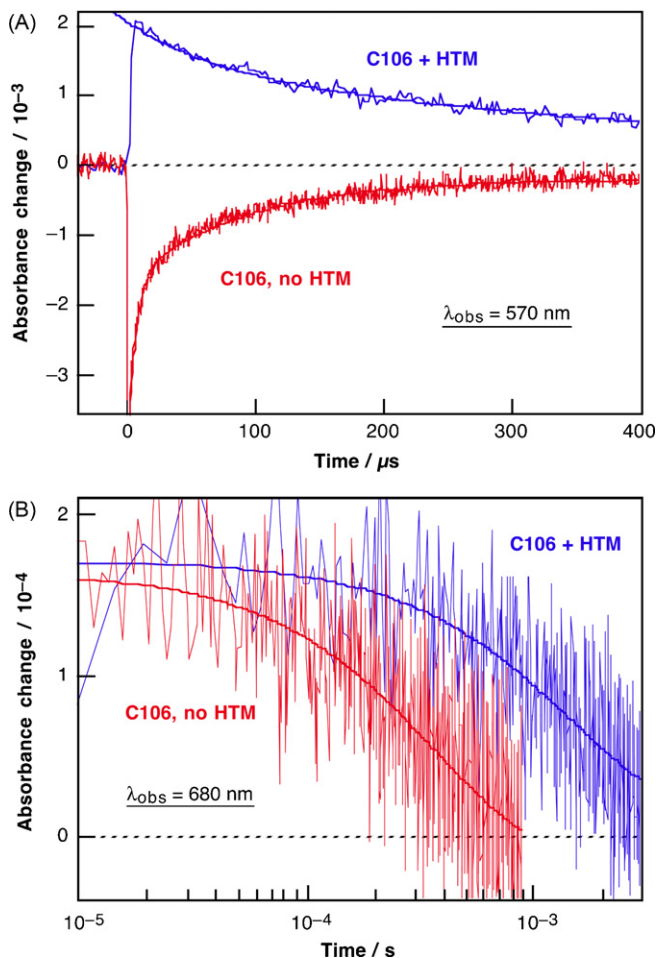
yl)-2,2'-bipyridine) (4-carboxylic acid-4'-carboxylate-2,2'-bipyridine) (NCS)<sub>2</sub>, coded as C106 [16] (see Fig. 1). Photovoltaic parameters were measured for C106 derivatized SSDSC and compared to devices fabricated using the analogous Z907Na dye. Laser transient and photoinduced absorption (PIA) techniques have also been used to analyze the kinetics of SSDSC devices employing C106 sensitizer. The insertion of thiophene units in-between the alkyl chain and the pyridine units augments the molar extinction coefficient of the sensitizer by increasing the  $\pi$  conjugation. The dye's hydrophobicity likewise augments due to its alkyl chains, rendering better pore filling by the HTM, resulting in more efficient dye regeneration and a lower recombination rate. The molar extinction coefficient ( $\epsilon$ ) of C106 at 550 nm is  $18,700\ \text{M}^{-1}\ \text{cm}^{-1}$ . Under identical conditions, the widely used analogous Z907Na dye exhibits an absorption band at 521 nm with an extinction coefficient of  $\epsilon = 12,200\ \text{M}^{-1}\ \text{cm}^{-1}$ . Sensitization of the thin nanocrystalline titania film by C106 dye leads to the expected enhancement of the device's performance in comparison to cells sensitized by Z907Na. This enhanced performance is attributed in part to the improvement of C106's light harvesting properties relative to Z907Na. In Fig. 2 the  $J$ - $V$  characteristics of a SSDSC device fabricated using the C106 dye and a  $1.9\ \mu\text{m}$  thick transparent titania film (consisting of  $\cong 20\ \text{nm}$  sized TiO<sub>2</sub> particles) and spiro-MeOTAD as HTM exhibits a  $J_{\text{sc}}$  of  $8.27\ \text{mA}\ \text{cm}^{-2}$ , a  $V_{\text{oc}}$  of 848 mV and a FF of 0.71, yielding an impressive 5% efficient SSDSC. This is the first time that a certified efficiency of 5% (measured at the National Renewable Energy Laboratory (USA)) under standard global AM1.5 solar conditions ( $100\ \text{mW}\ \text{cm}^{-2}$ ) is being reported for a solid-state DSC device. The increase in open circuit voltage of the C106 SSDSC device compared to that of the analogous Z907Na device (749 mV)



**Figure 2**  $J$ - $V$  characteristics of a SSDSC sensitized by C106 dye measure by the NREL photovoltaic calibration laboratory under standard reporting conditions, i.e. illumination with AM 1.5G sunlight (intensity  $100 \text{ mW cm}^{-2}$ ) and 298 K temperature. The inset exhibits its photocurrent action spectrum. Cell active area tested (with a mask):  $0.2505 \text{ cm}^2$ .

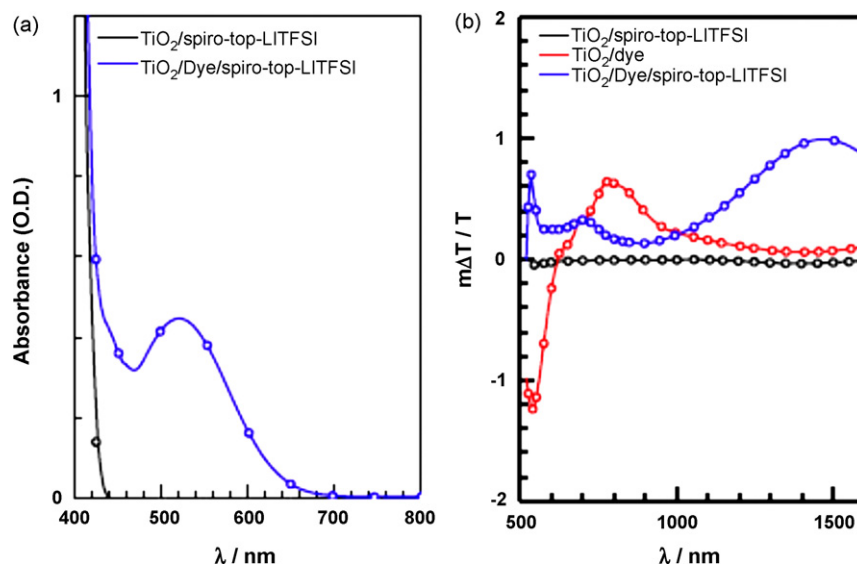
can be attributed to a longer comparative recombination lifetime (see supporting information, Fig. S1), contributing to the overall enhanced efficiency of C106 devices [9,10]. The incident photon-to-current conversion efficiency (IPCE) spectrum exceeds 40% over the spectral range from 420 to 580 nm reaching a maximum of approximately 60% at 500 nm (see the inset in Fig. 2).

Laser transient absorbance measurements were performed to monitor the dynamics of the recombination of electrons injected in the conduction band of  $\text{TiO}_2$  ( $e_{\text{cb}}^-$ ) with the oxidized dye ( $S^+$ ), of hole injection from  $S^+$  into the HTM, and of the indirect recombination process taking place between the oxidized HTM and  $e_{\text{cb}}^-$ . Fig. 3A displays the temporal evolution of the absorbance measured at  $\lambda = 570 \text{ nm}$  of a transparent C106 dye-sensitized  $\text{TiO}_2$  film upon pulsed laser excitation at 510 nm. In the absence of HTM, photo-excitation and ultrafast electron injection leads to the bleaching of the dye's ground state (negative transient signal, Fig. 3A, red curve). From the magnitude of the bleaching signal and the absorbed number of laser photons it can be inferred that charge injection occurs with a quantum yield close to 1. When dye stained titania film pores were filled with the HTM the dye is regenerated by hole injection in the organic solid resulting in the appearance of the typical absorption of spiro-MeOTAD cation radicals (Fig. 3A, blue trace). This hole transfer is completed within a few nanoseconds and hence occurs within the laser excitation pulse [19,20]. In this case the laser excitation energy fluence was quite high ( $200 \mu\text{J cm}^{-2}$  per pulse) and resulted on average in the injection of much more than one electron per  $\text{TiO}_2$  particle and in the fast decay of the observed



**Figure 3** Temporal profiles of the transient absorbance measured at  $\lambda = 570 \text{ nm}$  (A) and at  $\lambda = 680 \text{ nm}$  (B) upon pulsed laser excitation ( $\lambda = 510 \text{ nm}$  wavelength, 5 ns full width half-maximum pulse duration, 30 Hz repetition rate) on samples comprised of C106 dye adsorbed on nanocrystalline  $\text{TiO}_2$  films in the presence (blue traces) and in the absence (red traces) of spiro-MeOTAD hole transport material. Excitation pulse energy fluence was  $200 \mu\text{J cm}^{-2}$  (A) and  $20 \mu\text{J cm}^{-2}$  (B). Smooth solid lines are double exponential fits of experimental data.

signals. Fig. 3B shows transient absorbance traces recorded at  $\lambda_{\text{obs}} = 680 \text{ nm}$ . This time, energy fluence was attenuated down to  $20 \mu\text{J cm}^{-2}$  per pulse, so as to ensure that at most one electron was injected per  $\text{TiO}_2$  particle during each excitation pulse. In such conditions, recombination kinetics is expected to be identical to those characterizing functional systems under solar irradiation. In the absence of HTM (Fig. 3B, red trace), absorbance at 680 nm was attributed mainly to the dye oxidized state species  $S^+$ , a small contribution to the signal being due to conduction band electrons. Decay of the absorbance resulted from the direct recombination process ( $S^+ - e_{\text{cb}}^-$ ), with a half-reaction time of  $t_{1/2} = 200 \mu\text{s}$ , significantly larger than what was observed upon excitation by higher fluence laser pulses (Fig. 3A). In the presence of spiro-MeOTAD (Fig. 3B, blue trace), the signal is dominated by the absorbance of oxidized spiro-MeOTAD. The decay time constant  $t_{1/2} = 1 \text{ ms}$  measured in this case thus reflects the kinetics of the recom-



**Figure 4** (a) The visible absorption spectra of the thin  $\text{TiO}_2$  film filled with spiro-MeOTAD (black line) and in presence of C106 dye (blue line). (b) The photo-induced absorption (PIA) spectra of nano-structured  $\text{TiO}_2$  films ( $1.9 \mu\text{m}$  thick): red line, spectra for a C106 dye-sensitized  $\text{TiO}_2$  film in air; blue line, for a dye-sensitized  $\text{TiO}_2$  film in the presence of spiro-MeOTAD, and black line, for a bare  $\text{TiO}_2$  (no dye) infiltrated with solid hole conductor.

bination between holes in the HTM and conduction band electrons. The indirect recombination observed here with the spiro-MeOTAD solid-state hole conductor suggests this process is responsible for the performance loss compared to electrolyte-based DSC where electron lifetime is on the order of three to hundreds of ms range [17]. Since the electron injection in the SSDSC device with C106 dye is fast, the IPCE value is limited by the light harvesting efficiency  $\eta_{\text{LH}}$  (process 1) which inferred from Fig. 4a ( $\eta_{\text{LH}} = 1 - 10^A$ ,  $A$  being the absorbance) to be 65% for a single passage of light through the film.

Unraveling of the details of spectral and kinetic information of the devices was likewise undertaken by employing PIA techniques. Fig. 4b shows the PIA spectra using the dye adsorbed titania film, deposited on FTO glass covered with a compact  $\text{TiO}_2$  layer, in the presence or absence of the HTM. In the absence of the HTM, the PIA spectrum (red line) shows clearly the differential spectrum of C106 upon formation of the dye oxidized state, with a bleaching of the main absorption band at 538 nm (the ground state MLCT absorption) and an absorption peak at 807 nm (the oxidized state dye,  $\text{C106}^+$ ) [21–23]. Upon addition of the solid-state hole conductor, the PIA spectrum changes significantly. The absorption peak at 712 nm arises from the formation of the oxidized spiro-MeOTAD (spiro-MeOTAD $^+$ , Fig. S2) in addition to a small contribution from  $\text{C106}^+$ . From the known extinction coefficients of the three species (spiro-MeOTAD $^+$ , C106 and  $\text{C106}^+$ ), which are responsible for the optical changes [24–26], one derives the spiro-MeOTAD $^+$ /C106 $^+$  ratio, from the two curves, to be close to  $0.85 \pm 0.05$  indicating a very efficient hole-transfer process. The dye-sensitized nanocrystalline film without spiro-MeOTAD exhibits a shorter estimated PIA signal lifetime than that found in presence of HTM over the spectra range from 500 to 1500 nm (Fig. S3), due to a fast dye regeneration.

In conclusion, we have presented a highly efficient solid-state DSC sensitized by a heteroleptic polypyridyl ruthenium complex coded as C106 in which (hexylthio)-thiophene units have been inserted between the pyridine moieties and the alkyl chains leading to higher molar extinction coefficient and an increase in the charge recombination lifetime relative to SSDSC devices employing the analogous Z907Na dye. Through judicious molecular design in the development of the C106 dye, an encouraging certified 5% power conversion efficiency was achieved. It has been demonstrated that increasing the molar extinction coefficient of sensitizers and reducing the indirect recombination of the oxidized spiro-MeOTAD species with the photo-injected electron is an elegant strategy to improve the photovoltaic performance of solid-state dye-sensitized solar cells.

## Experimental

In the present work, C106 was used as an efficient sensitizer for solid-state DSCs, following synthetic procedures reported earlier [16]. Solar cell fabrication and characterizations were performed according to prior literature [10]. The PIA spectra of the various cells were recorded over a wavelength range of approximately 500–1500 nm following a (on/off) photo-modulation using a 9 Hz square wave emanating from a blue LED. White probe light from a halogen lamp was used as an illumination source. The recombination rate constants were determined by employing transient photovoltage decay measurement techniques [10,12].  $\text{TiO}_2|\text{C106}|$ HTM samples used in the laser transient absorbance measurements were obtained by peeling off the gold cathode layer of solar cells identical to those used in photo-electrochemical measurements. Samples were excited by nanosecond laser pulses produced by a

broad-band optical parametric oscillator (OPO GWU-355), pumped by a frequency-tripled Q-switched Nd:YAG laser (Continuum Powerlite 7030). Excitation pulses ( $\lambda = 510$  nm wavelength, 30 Hz repetition rate, pulse width at half-height of 5 ns) were attenuated by neutral density filters to reduce pulse fluence on the sample. The probe light from a Xe arc lamp was passed through an interference filter, various optical elements, the sample, and a grating monochromator before being detected by a fast photomultiplier tube, only 3 dynodes of which were used.

## Acknowledgements

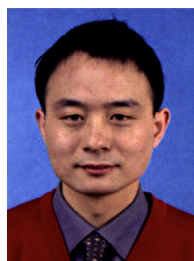
This publication is based on work supported by the Center for Advanced Molecular Photovoltaics (Award No KUS-C1-015-21), made by King Abdullah University of Science and Technology (KAUST). Financial support from the Swiss National Science Foundation is also gratefully acknowledged. PW and JL thank the National Key Scientific Program (No. 2007CB936700) for the financial support. We are grateful to Mr. P. Comte for TiO<sub>2</sub> nanoparticles preparations and Mr. Jean-David Decoppet for helping in performing the NREL measurements.

## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.nantod.2010.04.001](https://doi.org/10.1016/j.nantod.2010.04.001).

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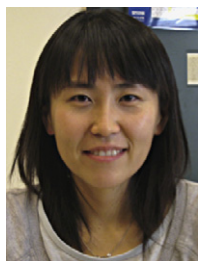
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He has published more than 150 peer-reviewed scientific papers and is the inventor of 20 patents.



**Prof. Michael Grätzel** is a professor at the Ecole Polytechnique de Lausanne where he directs the laboratory of photonics and interfaces, pioneered studies of electron and energy transfer reactions in mesoscopic systems, a field where he has an outstanding track record and has maintained leadership on the global scale through several decades. Grätzel's infatuation with the mesoscopic dimension started early on in his career.

Investigating photo-induced energy and electron transfer reactions in micellar systems, he discovered the unique quenching kinetics of solubilized fluorophores and used the local electrostatic field present at the micellar surface to control the dynamics of photo-induced electron transfer reactions. His publications include over 800 research papers in peer-reviewed scientific journals and 60 reviews or invited book chapters. He is the inventor or co-inventor of over 50 patents and editor, author or co-author of three books. His publications have received close to 60,000 citations (*h*-index 116) ranking him amongst the most highly cited scientists in the world. He has received numerous prestigious awards, including the Balzan prize, the Harbey Prize, the Faraday Medal of the British Royal Society of Chemistry, the Dutch Havinga award, the ENI-Italgas prize and the Gerischer award. He received a doctor's degree in natural science from the Technical University Berlin and honorary doctors degrees from the Universities of Delft, Uppsala and Turin.