

Hot embossed large-area plant microstructures for improved photovoltaic light harvesting

By Benjamin Fritz¹⁾, Markus Guttman²⁾, Guillaume Gomard^{1,2)} and Ruben Hünig³⁾

Within the scope of a KNMF project (KNMF = Karlsruhe Nano and Micro Facility) with the Center for Solar Energy and Hydrogen Research Baden-Wuerttemberg (ZSW) as project partner, a process routine to replicate the surface micro-/nano-textures of natural rose petals into large-area polymeric foils via hot embossing lithography has been successfully developed. Several natural petals cut into 3.5 x 2.0 cm² pieces were stitched together to achieve a total structured area of 12.5 x 10 cm². Overall, ten iterations of the shim/mold insert fabrication were carried out within this project. Using these mold inserts, a total of several hundreds of polymer foils was thus hot embossed in different polymeric thin foils. The light harvesting and self-cleaning properties of the latter were comprehensively analyzed. The fabricated structured foils were then applied onto Cu(In,Ga)Se₂ (CIGS) thin-film solar cells and 10 x 10 cm² CIGS solar modules, leading to a power output increase of around 5%, measured under outdoor conditions and over a period of 28 days. Additionally, the textured polymeric layers atop the solar modules largely reduce the amount of reflected polarized light, which is known to affect the ecology of several (polarotac-tic) insect species.

Heißgeprägte großflächige Pflanzenmikrostrukturen für eine erhöhte Lichtausbeute in der Photovoltaik

Im Rahmen eines KNMF-Projekts (KNMF = Karlsruhe Nano and Micro Facility) mit dem Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW) als Projektpartner wurde erfolgreich eine Prozessroutine zur Replikation der Mikro-/Nano-Oberflächentexturen natürlicher Rosenblütenblätter in großflächige Polymerfolien mittels Heißpräge-Lithographie entwickelt. Mehrere natürliche Blütenblätter, in Stücke von 3,5 x 2,0 cm² geschnitten, wurden zusammengesetzt um eine strukturierte Gesamtfläche von 12,5 x 10 cm² zu erhalten. Insgesamt wurden im Rahmen dieses Projekts zehn Iterationen der Shim-/Formeinsatz-Herstellung durchgeführt. Unter Verwendung dieser Formeinsätze wurden insgesamt mehrere hundert Folien aus verschiedenen Polymeren heißgeprägt. Letztere wurden umfassend auf ihre Lichteinwirk- und Selbstreinigungseigenschaften hin untersucht. Die strukturierten Folien wurden auf Cu(In,Ga)Se₂ (CIGS)-Dünnschicht-Solarzellen und 10 x 10 cm² CIGS-Solarmodule aufgebracht, was zu einer Leistungssteigerung von rund 5 % führte, gemessen im Freifeld über einen Zeitraum von 28 Tagen. Darüber hinaus verringern auf den Solarmodulen die texturierten Polymerschichten die Menge an reflektiertem polarisiertem Licht, von dem bekannt ist, dass es die Ökologie mehrerer (polarotaktischer) Insektenarten beeinflusst.

1 Introduction

The outer surfaces of animals and plants have evolved to achieve functionalities as diverse as heat, water or light management. The wealth of surface textures found in nature is a great source of inspiration for the design of (multi)functional coatings. It has been shown both experimentally and numerically, that the petal surface textures of most flowering plant species typically feature densely-packed micro-structures composed of nearly conical epidermal cells, which are further decorated by nanoscale (cuticular) folds (Fig. 1). Together, these properties foster

sunlight harvesting and trapping, as needed for photosynthesis in leaves or for achieving saturated colors in petals. The same effects can be exploited to improve the sunlight absorption capability in photovoltaics (PV), regardless of the illumination direction. One way to realize this is to replicate the plant surface textures in a transparent cover layer directly applied onto solar panels.

In a first attempt to demonstrate the potential of this bio-replication approach, the hierarchical structures of rose petals were replicated into a transparent, ultraviolet (UV)-curing resist using a polydimethyl-siloxane

(PDMS) template and applied onto the cover glass of organic solar cells. Thanks to the above mentioned optical mechanisms, the short-circuit current density (J_{sc}) of these small-area photovoltaic prototypes (active area of 3.5 x 3 mm²) improved by 13 % under normal incidence, and by up to 44 % under an incident angle of 80° [1]. A similar route, using either positive or negative templates, was tested on other solar cell technologies and resulted in a significant performance enhancement. That demonstrating the solar harvesting potential of petal texture replicas. Herein, the epidermal cells convexity was successfully preserved by replicating fresh petals into a PDMS mold at ambient conditions, hence limiting water losses and avoiding vacuum-operated processes, which promote the shrinkage of the microstructures of the natural surface texture.

In the present work, the authors review their recent advances towards the mass fabrica-

¹⁾ Karlsruhe Institute of Technology (KIT), Light Technology Institute (LTI), D-76131 Karlsruhe, Engesserstraße 13; benjamin.fritz@kit.edu & guillaume.gomard@kit.edu

²⁾ Karlsruhe Institute of Technology (KIT), Institute of Microstructure Technology (IMT), D-76344 Eggenstein-Leopoldshafen, Hermann-von-Helmholtz-Platz 1; markus.guttman@kit.edu

³⁾ Center for Solar Energy and Hydrogen Research Baden-Württemberg (ZSW), D-70563 Stuttgart, Meitnerstraße 1; ruben.huenig@gmx.de

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tion of large-area PV cover layers incorporating the rose petal surface texture, and on their subsequent lamination onto encapsulated solar modules. This is now realized by developing a hot embossing process, which, compared to the previous standard method via soft imprint replication, significantly increases the possible throughput and allows the reproducible texturing of self-standing polymer foils by using a mechanically stable and durable metallic mold. Compared to the widely used PDMS molds that are used in soft imprint replication, this hot embossing approach introduces a great flexibility in the choice of polymer material to be textured. We note that in-depth discussions of all the advances in fabrication methodology and also the PV performance enhancements which are summarized within this communication can be found in references [2, 3].

2 Development of a hot embossing routine for the large-area replication of petal textures

For the fabrication of the hot embossing mold inserts, several working steps had to be carried out, which are depicted in Figure 1 and described in detail below. Using the thus fabricated metallic mold inserts, three differ-

ent polymeric materials were hot embossed, leading to several hundred pieces of rose petal surface texture replicas with dimensions of up to 12.5 x 10 cm².

2.1 Stitching of fresh rose petals and large-area soft imprint lithography

For the preparation of a large-area PDMS stamper, fresh rose petals (variety: *Red Naomi*) were cut by using rectangular glass slides as stencils. These rectangular cut outs were manually assembled into arrays onto metallic substrates using double-sided adhesive tape and then immediately covered with PDMS (Sylgard® 184, Dow Corning) before the petals lose their structural integrity due to water evaporation from the epidermal cells. After curing the PDMS material at room temperature, the edges of the resulting soft PDMS (negative) stampers (with a thickness of about 10 mm) were cut to ensure proper planarity. This is necessary for the subsequent hot embossing step in order to achieve reasonable thermal contact over the whole area between the fabricated metallic copy of the (negative) texture and the heated substrate (massive brass sheet). After cleaning the PDMS stampers from residual plant ma-

terial, large-area soft imprint (positive) replicas were fabricated on 4", 6" or 8" silicon wafers using NOA68 (Norland Products, Inc.) as a UV-curing resist. Subsequently, these master substrates were used to carry out a nickel electroforming routine.

2.2 Fabrication of thick metallic mold inserts

First, 14 nm of chromium (adhesive layer) and 100 nm of gold (conductive plating base) were evaporated onto the resist-structured silicon wafer substrate. The textured and metal-coated wafer was subsequently fixed onto a commercial (for 4" and 6" substrates) or a homemade (for 8" substrates) plating holder and then immersed into a standard nickel electroplating system that was specially developed for thick nickel electroforming of micro- and nanostructures.

The system included boric acid containing nickel sulfamate electrolyte at a temperature of $T = 52\text{ }^{\circ}\text{C}$ and a pH in the range of 3.4–3.6. By gradually ramping up the current density from 0.1 A/dm² to 1.0 A/dm² from start to finish of the electroplating process, defect-free nickel deposition into the micro-/nanotextured areas was assured. With this routine, nickel mold inserts with a thickness of up to 3 mm were fabricated. After a first wire electrical discharge machining (wire-EDM) step (to planarize the backside) and a consecutive removal of the silicon substrates by wet chemical dissolution (using KOH solution), the nickel mold inserts were thereby slightly reduced in thickness. The residual NOA68 was removed by treatment with dichloromethane. By employing a further wire-EDM step, the final outer dimensions of the nickel mold inserts were realized. The first finished mold inserts had the shape of a small rectangle of 2.5 x 7.0 cm² or circular configurations (with diameters of the textured area between 78 and 136 mm) but finally, textured areas of up to 12.5 x 10.0 cm² could be achieved (with outer dimensions of the mold inserts reaching up to 15.5 x 13.0 cm²).

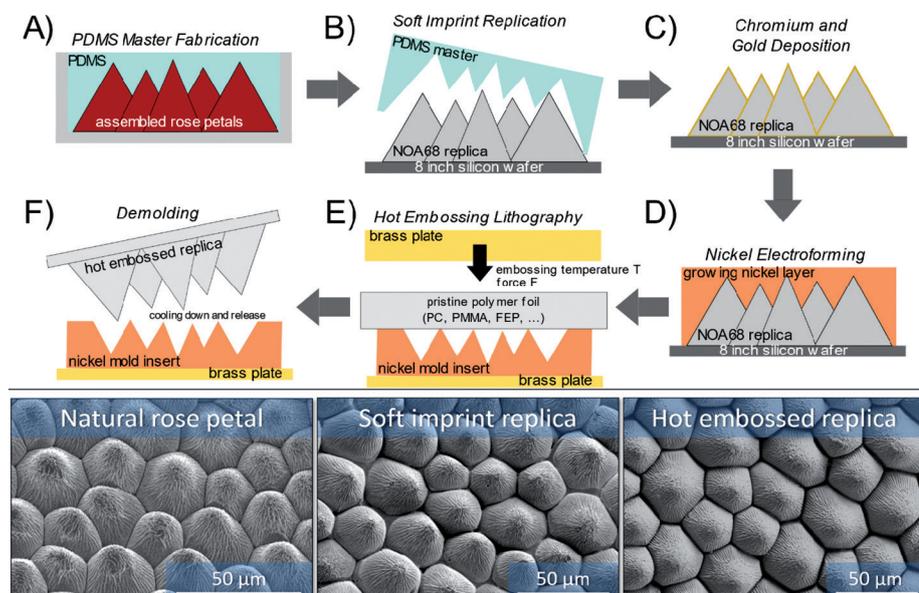


Fig. 1: Top - Schematic depiction of the fabrication routine of rose petal textured polymeric foils via hot embossing [3]. A) and B) At first, a soft imprint lithography process is carried out to transfer the petal surface texture via PDMS into a UV-curing polymer on top of a silicon wafer. C) Subsequently, the wafer with the structured polymer are covered by thin layers of chromium and gold to ensure a good surface conductivity for D) the nickel electroforming. E) and F) The finished nickel mold insert is then used to structure a polymer foil by applying heat and pressure. Bottom - SEM images (top view) of a natural rose petal, a soft imprint replica and a hot embossed replica [4]. The typical rose petal surface texture is kept intact during the double-replication process depicted in A) - F).

2.3 Hot embossing the (up-scaled) rose petal texture into polymeric foils

Large-area polymeric replicas of the petal surface texture were fabricated from three different polymer foils, namely poly(methylmethacrylate) (PMMA, upag AG), polycarbonate (PC, Cadillac Plastic GmbH) and fluorinated ethylene propylene (FEP, RCT Reichelt Chemietechnik GmbH + Co. KG) foils, by us-

ing hot embossing. The pristine polymeric foils were 1 mm (PMMA), 0.3 mm (PC) and 0.25 mm (FEP) thick, with refractive indices of 1.49 (PMMA), 1.58 (PC) and 1.34 (FEP), all measured at a wavelength of 588 nm. The foils were first placed on a polytetrafluoroethylene (PTFE) sheet on top of a polished steel plate to ensure a smooth back surface of the replica.

The nickel mold was then heated and embossed into the three different polymer materials utilizing the following parameters:

- For PMMA, hot embossing was performed at 150 °C with a force of 90 kN and a demolding temperature of 115 °C.
- For PC, embossing with 60 kN at 170 °C and demolding at 130 °C was performed.
- For FEP, which has a higher glass transition temperature, a force of 37 kN at 275 °C with demolding at 235 °C was used.

These parameters were utilized for hot embossing using a nickel mold insert with a circularly shaped textured area of 78 mm in diameter. It was noted that, in case of using a mold insert with a larger textured area, the forces necessary to achieve a proper hot embossing replication need to be proportionally increased as well (to keep the force per area constant).

2.4 Lamination of hot embossed rose petal replicas onto solar cells and modules

The hot embossed rose petal replicas were integrated into highly efficient, single Cu(In,Ga)Se₂ (CIGS) thin film solar cells (0.5 cm²) by using the UV-curing resist NOA88 (Norland Optical Adhesives), and on CIGS mini modules (10 x 10 cm²) by lamination. For the latter, a polyolefin foil (PO8110) was placed on top of the module and a laminator (SPI Laminator 240, Spire) was used to glue the cover layers onto the modules. Mechanical pressure was applied normally to the layer stack at a temperature of 140 °C under vacuum (10 min at 10 mbar, then another 20 min at 0.5 bar) to achieve homogeneous and mechanically robust coupling. Lamination of planar glass cover layers and planar PMMA cover layers was achieved analogously (Fig. 2). Scanning electron microscopy (SEM) micrographs of a PMMA rose petal replica after this lamination process and photographs of CIGS modules equipped with planar and textured cover layers can be seen in Figure 2. Here a high replication fidelity from the (natural) original structure template to the hot embossed

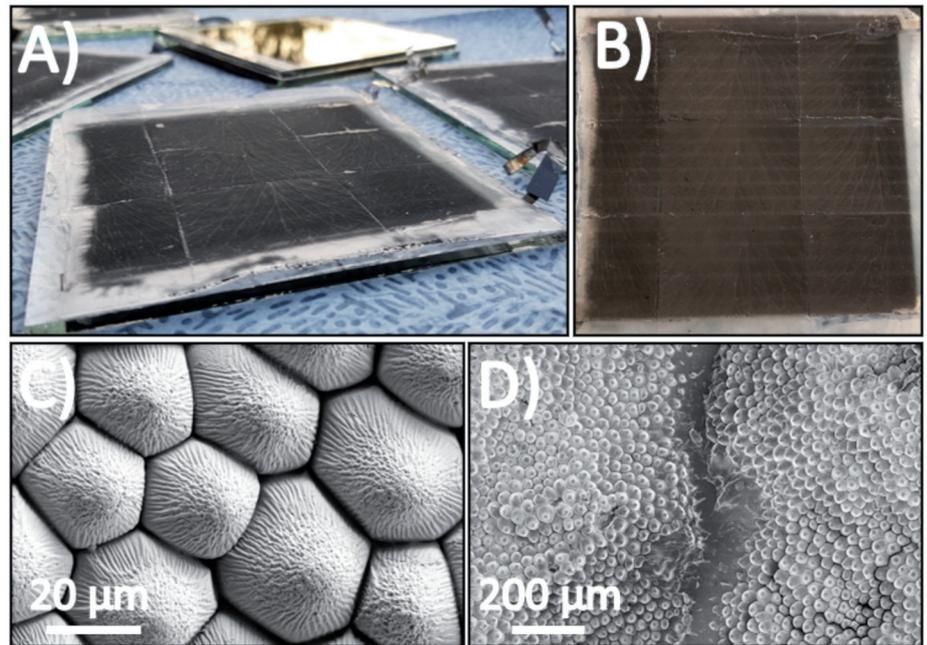


Fig. 2: A) Photograph of several 10 x 10 cm² CIGS modules with rose petal textured PMMA laminated directly onto the cover glass. In the background, an identical CIGS module with planar glass as its uppermost layer exhibits a much brighter appearance with a strong mirroring effect. The photos were taken under direct sunlight illumination in the evening hours. B) Top-view photo of a CIGS mini module equipped with a large-area PMMA rose petal replica. C) Shows an SEM image of a defect-free part in the center of one of the rose petal patches of a large-area PMMA replica after lamination onto a CIGS module, while D) depicts an average stitching line with an unpatterned area of about 100 µm in width [3].

PMMA (second) replicas after lamination onto a working PV device, and a significant reduction of reflected light could be realized.

3 Characterization of large-area hot embossed rose petal replicas

The quality of the structured foils and their suitability as photovoltaic (PV) light harvesting layers were investigated using various methods. First of all, SEM analyses were carried out to evaluate the fidelity of the bio-replication process at the micro- and nanometer scale. Secondly, the optical properties of the foils were measured by the means of reflectance spectroscopy, and after integration into photovoltaic devices, optoelectrical characterizations were carried out under controlled conditions. Finally, the solar modules were long-term tested under outdoor conditions in the city of Karlsruhe, Germany. Furthermore, another (side-)study making use of the up-scaled hot embossed replicas to investigate the (so far often overlooked) topic of polarized light pollution and its negative ramifications for many insect species, was conducted last year and some of the resulting achievements are also briefly summarized below.

3.1 Topographical analysis

The investigation of structured polymer foils and nickel shims by SEM analyses shows, that the texture of the rose petals was transferred with very high structural fidelity (not only from the original biotemplate to the NOA68 replica on a silicon wafer substrate, but also from this soft imprint replica to the hot embossed self-standing polymer replica, see SEM images in Figures 1 and 2). Even the nano-wrinkles atop the microstructures were truthfully transferred. Although they are exposed to both heat and pressure during the lamination process, the CIGS modules still exhibit a perfectly intact surface (Fig. 2) afterwards.

3.2 Optical characterization

Before testing the performance of the bio-replicated foils onto PV devices, their optical properties were first characterized. The angle-dependent reflectance spectra were acquired for hot embossed rose petal replicas on black absorbers, but also on the complete layer stack corresponding to the real CIGS PV devices (only without the necessary electrical contacts). For this purpose, small samples (2.5 x 2.5 cm²) were analyzed using a spectrophotometer (Lambda 1050 UV/

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Vis/NIR, Perkin Elmer) equipped with an integrating sphere. To test different illumination angles, the samples were fixed on a rotatable center mount. Thus, the reflectance over a broad range of angles (up to 80°) could be studied. From the three materials investigated herein, PMMA was found to exhibit the most efficient anti-reflection properties (Fig. 3).

3.3 Opto-electrical characterization of replicas on CIGS solar cells under lab conditions

To check if the reduced reflection is translated into a higher photocurrent generation of the photovoltaic devices, external quantum efficiency (EQE) measurements on small CIGS cells with rose-PMMA layer on top were carried out. Thus, one can see for each wavelength of the relevant light spectrum how efficiently the light is converted into a usable current. Due to restrictions of the EQE setup, only normally incident light could be investigated. Nevertheless, the PMMA foils performed as well as the highly optimized, state-

of-the-art anti-reflection coating of 105 nm MgF_2 (Fig. 4A). I-V-measurements under standard testing conditions on the same cells confirmed the finding, that the hot embossed PMMA foil is at least equivalent or better than the MgF_2 coating.

The CIGS modules (10 x 10 cm² total area) were evaluated by angle-resolved I-V-measurements. Here, the hot embossed PMMA foil could show its full potential by almost doubling the photocurrent output at high angles of incidence, clearly outperforming the uncoated devices (Fig. 4B).

3.4 Outdoor tests of replica-covered solar modules

To make sure the improvements measured under lab conditions due to the implementation of hot embossed PMMA foils still holds true under realistic operating conditions, CIGS modules were tested outside for 28 days (Fig. 4C). The average gain of generated photocurrent was around 5 %, comparing a solar module with glass encapsulation and hot embossed PMMA foil with a

solar module with glass encapsulation and flat PMMA foil. During this time, no significant soiling or degradation of the structured foils could be observed.

3.5 Reduced polarized light pollution of replica-covered solar modules

An important characteristic of the rose petal surface texture, namely that the very small amount of light that is still being reflected is mostly depolarized light, is of great relevance in view of the current mass extinction of insects. Conventional, glass-covered solar modules reflect light with a predominantly horizontal polarization direction, which can trigger misleading behavioral reflexes in polarotactic insects (*polarotaxis* = orientation of an organism in line with the plane of polarization of polarized light). The surfaces of solar modules can actually be confused with water surfaces or the bodies of host animals, which can lead to (erroneous) egg deposition or foraging. Both types of such misguided behavior can cause massive damage to such insect populations.

Thanks to the hot embossing route discussed herein, we were able to fabricate 50 x 50 cm² rose petal textured test surfaces by stitching together several replicas (12.5 x 10.0 cm² textured area per up-scaled replica). In order to investigate the effect of rose petal textured surfaces on insect behavior, those 50 x 50 cm² test surfaces were sent to a research group in Hungary (Prof. Dr. Gábor Horváth et al., Environmental Optics Laboratory, Department of Biological Physics, Eötvös Loránd University, Budapest). The structures were examined by means of imaging polarimetry and additionally, they were used in field experiments, which were designed to directly assess and compare the maladaptive attraction that different solar cell cover layers cause for polarotactic mayflies and horseflies. With respect to planar cover layers or commercial solutions and for most illumination and observation configurations, the rose petal replicas showed almost no horizontally polarized reflected light, and as stated above, a much lower total surface reflectance. Therefore, almost no attraction of the hot embossed replicas to both of the studied species of polarotactic insects could be observed. Thus, we demonstrated that our design and prototypes can be deployed over large areas to promote light harvesting, while simultaneously minimizing their (possible) negative impact on the ecology of such insects [4, 5].

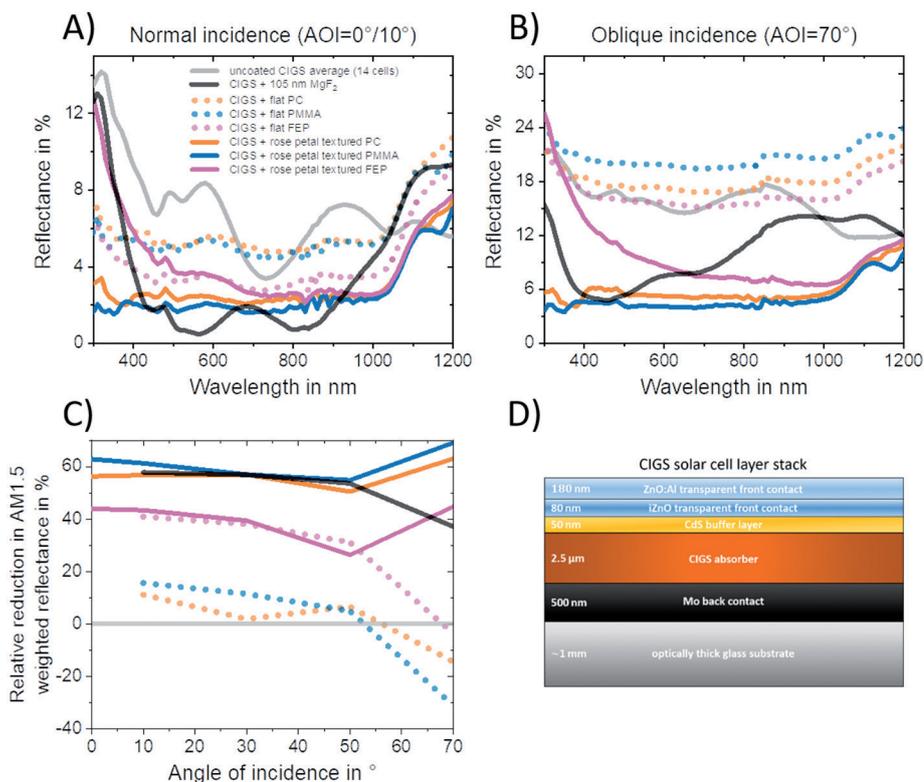


Fig. 3: Light harvesting properties of CIGS thin-film solar cells coated by the flat cover layer and by the hot embossed (rose replicated) one; for three selected polymers [2]. A) Spectrally resolved reflectance at (close to) normal incidence and at B) an oblique incidence angle of 70°. For comparison, reflectance spectra were also acquired for the uncoated CIGS solar cell layer stack and for the same device with an additional 105 nm MgF_2 antireflective layer on top. C) By weighting the measured reflectance spectra with the AM1.5G solar spectrum, the relative reduction in weighted (solar) reflectance (relative to uncoated devices) was computed. Positive values denote a reduction in weighted reflectance. The layout of the CIGS solar cell stack is indicated in D).

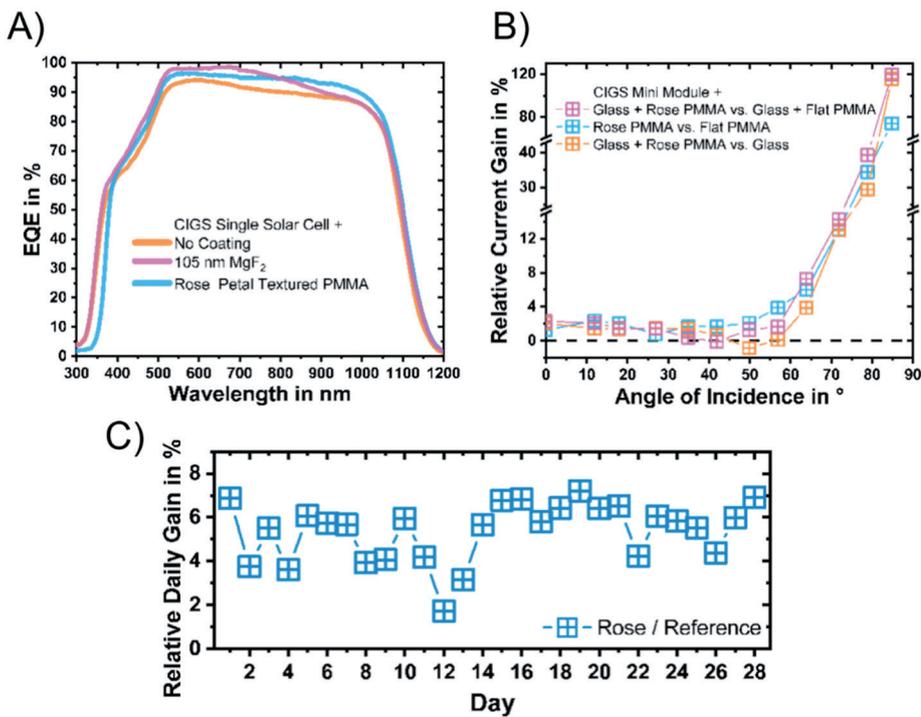


Fig. 4: Opto-electrical properties of CIGS single solar cells and mini modules, both under laboratory and under outdoor conditions [3]. A) EQE spectra of single CIGS solar cells with different cover layers. B) Relative gain in short circuit current for CIGS mini modules (10 cm x 10 cm) as a function of the light incidence angle and for different top layer configurations and reference devices. C) Results of outdoor performance monitoring of a CIGS mini module equipped with a PMMA rose petal replica in Karlsruhe, Germany. The daily overall photocurrent gain was calculated with respect to modules only encapsulated by a planar glass cover layer.

4 Conclusions & Outlook

Within this KNMF project, we successfully up-scaled the fabrication routine of polymeric petal texture replicas, incorporating the hierarchical surface micro-/nanotexture of rose petals over areas of up to 125 cm², which has, to the best of our knowledge, never been realized so far. The hot embossing routine allowed us to produce several hundred large-area replicas and to fabricate functional photovoltaic prototypes after laminating these replicas onto 100 cm² CIGS solar modules. Outdoor measurements of such modules equipped with rose petal light harvesting layers demonstrated a power output increase of more than 5%. This project also opened new perspectives for these coatings that might be relevant in terms of insect-conservation, owing to their reduced amount of polarized light pollution produced. However, the potential of the bioreplication approach, as discussed herein, has not yet been fully exploited. Therefore, the authors had to overcome a number of challenges: Most notably, the manual stitching of several textured patches leads to undesired planar regions that are visible by naked eye. These defects both affect the aesthetic appeal of

our previously developed prototypes and result in an incomplete anti-reflection effect. In addition, the macroscopic waviness of the individual petals requires a relatively high polymer layer thickness for a proper replication via hot embossing, which leads to high material costs and increased parasitic absorption in the foils. The solution to these problems is the subject of a patent application initiated by the authors and enables the large-scale and cost-effective production of plant-textured films for use in commercial solar modules and many other applications.

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Karlsruhe Nano and Micro Facility (KNMF)

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