



## **SCALENANO FINAL REPORT**

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## DECLARATION BY THE SCIENTIFIC REPRESENTATIVE OF THE PROJECT COORDINATOR

I, as scientific representative of the coordinator of this project and in line with the obligations as stated in Article II.2.3 of the Grant Agreement declare that:

- The attached final report represents an accurate description of the work carried out in this project for the entire period;
- The project has fully achieved its objectives and technical goals for the period;
- The public website is up to date;
- To my best knowledge, the financial statements which are being submitted as part of this report are in line with the actual work carried out and are consistent with the report on the resources used for the project (section 3.4) and if applicable with the certificate on financial statement.
- All beneficiaries, in particular non-profit public bodies, secondary and higher education establishments, research organisations and SMEs, have declared to have verified their legal status. Any changes have been reported under section 3.2.3 (Project Management) in accordance with Article II.3.f of the Grant Agreement.

Name of scientific representative of the Coordinator: Alejandro Pérez-Rodríguez

Date: 29/09/2015

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Dissemination Level

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## SECTION 1- FINAL PUBLISHABLE SUMMARY REPORT

### 1.1 Executive Summary

SCALENANO ([www.scalenano.eu](http://www.scalenano.eu)) has been centred on the development and scale-up of chalcogenide based photovoltaic (PV) technologies. The project aimed to contribute to the achievement of a significant breakthrough in the competitiveness of thin film PV technologies already developed at the lab scale by their optimisation and transfer towards industrial scale-up and exploitation. The main goal has been the development and scale up of an innovative chalcogenide PV technology using environmentally friendly and sustainable technologies with lower costs and higher efficiencies in order to increase the European competitiveness of existing PV technologies, thus contributing to an increase in the share of PV electricity in Europe.

At the final stage of the project, SCALENANO has reported the achievement of several light-to-electricity power conversion efficiency world records in chalcogenide based PV. The project has led the development of environmentally friendly and vacuum free processes for the fabrication of cost-efficient CIGS ( $\text{Cu}(\text{In,Ga})(\text{S,Se})_2$ ) technologies. CIGS is the thin film photovoltaic technology with currently highest efficiency and a strong potential for reduction of manufacturing costs. The thin film approach differs from the commonly used wafer-based Si technology in the fabrication of the active materials: thin films with thicknesses in the micrometre scale are deposited onto rigid or flexible substrates while for the Si technologies wafers need to be cut out of high-grade silicon blocks. The direct deposition of thin films onto suitable substrates offers high throughput at potentially low cost-levels, low energy-payback times and a greater flexibility for choosing the substrate. This opens up new applications at low €/Wp levels, including highly aesthetic building-integrated applications on rigid or flexible, low-weight and even semi-transparent applications.

SCALENANO has made great advances in developing and optimizing thin film processes which are based on the electrodeposition of nanostructured precursors. The project has demonstrated the successful up-scaling of these technologies from the lab pilot production manufacturing with the production of  $60 \times 120 \text{ cm}^2$  modules with 13% average aperture efficiency and a best certified (independently measured by an accredited testing laboratory) 14% module efficiency. At cell level, a certified record 17.3% efficiency has been achieved, which constitutes the highest efficiency reported up to now for solution-based CIGS cells. These processes have been demonstrated at the pilot line of the company NEXCIS Photovoltaic Technology ([www.nexcis.fr](http://www.nexcis.fr)). The encapsulation structure of NEXCIS large area modules has been carefully optimised and modules have undergone a successful pre-qualification according to the relevant international standards aimed at ensuring their long-term durability in operation. The project has also succeeded in the development of alternative vacuum-free processes for the growth of transparent conductive oxide (TCO) window layers. These innovative technologies have been implemented at both cell and mini-module levels, reporting the first full vacuum-free based CIGS devices with efficiencies comparable to those achieved with reference window layers deposited with more complex and expensive sputtering tools and systems that are commonly used in the industrial production lines.

In a further step towards future mass deployment, SCALENANO has also tested the transferability of these processes to new and emerging PV technologies such as kesterites. Kesterites ( $\text{Cu}_2\text{ZnZn}(\text{S,Se})_4$ ) compounds are constituted by highly abundant chemical elements in the Earth's crust, and have been identified as the future replacement of CIGS when moving to higher scale (TeraWatt) PV deployment levels, since the scarcity of In or Ga is an issue. The extension of the electrodeposition-based processes developed at SCALENANO to these emerging materials has allowed up to now achieving cells with 9.1% efficiency, exceeding the previous world efficiency record for electrodeposited kesterite cells reported by IBM in 2014.



## 1.2 Summary Description of Project Context and Objectives

SCALENANO has been centred in the development and scale-up of an innovative chalcogenide photovoltaic (PV) technology using environmentally friendly and sustainable technologies with lower costs and higher efficiencies. The main objective of the project was to contribute to achieve a further reduction in the manufacturing costs of PV thin solar modules in order to increase the European competitiveness of existing PV technologies, favouring an increase in the share of electricity produced by PV in the next years within the European Union.

Cu(In,Ga)(S,Se)<sub>2</sub> (CIGS) PV technologies have already entered the stage of mass production. CIGS is the thin film photovoltaic technology with currently highest efficiency and a strong potential for reduction of manufacturing costs. However, current production methods typically rely on costly, difficult to control over large surfaces, vacuum-based deposition processes which require for very expensive equipment with initial high CAPEX. This compromises the potential reduction of material costs inherent to thin film technologies. At the forefront of this, SCALENANO has addressed the development of alternative environmental friendly and vacuum free processes based on the electrodeposition of nanostructured precursors. The project also included the exploration and development of alternative processes with very high potential throughput and process rate, as well as their extension to next-generation kesterite Cu<sub>2</sub>ZnSnS(Se)<sub>4</sub> (CZTS(Se)) based absorbers, which will allow the proposition of an industrial roadmap for the future generation of chalcogenide based cells and modules.

These objectives are in line with the so called 20/20/20 target for 2020, for the promotion of the use of renewable energies set by the European Commission and contained in the Directive on renewable energy (DIRECTIVE 2009/28/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 23<sup>rd</sup> April 2009 on the promotion of the use of energy from renewable sources), which sets ambitious targets for all Member States. Amongst other goals, the directive calls for a 20% share of energy from renewable sources within the EU by 2020. These requirements have become binding targets for each Member State in the EU.

Moreover, as stated in the European Strategic Energy Technology (SET) Plan, solar energy (particularly PV) is identified as one of the key technologies, in line with the goal defined by EPIA (European Photovoltaic Industry Association) to provide up to 12% of the EU electricity demand by 2020 with solar PV cells. This strongly makes necessary the availability of cost effective technologies which can easily be adapted to scaling-up towards mass production level stages.

To achieve these goals, the research program of the project included the following key objectives:

- 1) To develop a reliable low cost (30% lower than PVD process) and efficient (>95% of PVD process) electrodeposition based technology with a better lateral homogeneity (+/-5%/m<sup>2</sup>):**

At the beginning of the project, electrodeposition based processes were already available at laboratory scale and were moving towards industrial scaled-up production in different companies including NEXCIS. Improvement of the competitiveness of these technologies strongly required for a further increase in the efficiency of the devices towards values similar to those achieved by vacuum Physical Vapour Deposition (PVD) based processes (record cell efficiency 20.3%, best module efficiency 13-15%, and base lines production modules 10-11% at the beginning of the project). This required for a detailed optimisation of the processes involved in the electrochemical synthesis of the absorbers in order to achieve a better control on the composition of the CIGS alloys in the absorber and in-depth gradual composition of the layers and their lateral homogeneity on large area substrates.

- 2) To develop a new alternative scalable process compatible with high throughput (established target at pilot line 1m/min) requirements:**

Printing based processes have the highest potential to achieve very high throughput and process rate. Improvement of in cell and module efficiencies require better crystalline quality and homogeneity of the absorbers, with a more efficient control on the presence of secondary phases. Novel ink formulations and printing processes have been investigated based on the synthesis of chalcogenide ternary and quaternary compound nanoparticles with different composition, looking for simplification of the existing



processes and avoiding manipulation with toxic agents as  $\text{H}_2\text{Se}$  currently involved at this moment in industry for the selenization step. Promising alternative ink formulation approaches based on metal salts dissolved in appropriate solvents have also been investigated.

3) **To develop optimized TCO layers (transparency >80%, sheet resistance < 100 Ohm sq) by scalable non vacuum based processes:**

Development of cost efficient chalcogenide technologies also requires for scalable non vacuum processes alternative to the PVD (sputtering deposition) steps which are extensively used for the synthesis of the TCO layers in the industrial production of chalcogenide based modules. Scalable processes with strong potential for lowering of costs and compatible with the electro-optical characteristics (very good layer conductance on the order of  $10^3 \text{ ohm}^{-1}\text{cm}^{-1}$  combined with a high transparency of >80%), which are required for the window layers in high efficiency cells, have been developed. This has required for a significant effort in the optimisation of the electrical properties of the TCO layers, including the development of new TCO concepts based in the inclusion of metallic nanowires.

4) **To improve cell architectures on nanostructured TCOs for higher efficiencies cells and modules:**

Achievement of efficiencies higher than the existing record values (20.3% at cell level) strongly requires for the implementation of new concepts and cell architectures. Novel chalcogenide cell architectures as those based on the **implementation of nanostructured TCO's formed by columnar ZnO nanowire arrays** have been developed. This kind of cell structures is characterised by a very high active heterojunction surface, which allows for a significant potential improvement in the carrier collection efficiency with the minimisation of recombination losses.

5) **To develop and implement quality control and process monitoring techniques for scaled-up processes in large area substrates:**

Availability of on-line process monitoring techniques is critical to ensure a successful scale-up industrial implementation of these technologies. This includes **in-situ techniques for real-time monitoring and control of process parameters at large surface and high throughput**. Optical and electro-optical based techniques including Raman scattering, photoluminescence/ electroluminescence and photoelectrochemical based tools have been developed for the spatial mapping of the layers deposited on large area substrates. This objective includes the monitoring of the processes at different process steps along the defined process flow in pilot line, giving information directly related to the **uniformity of the optoelectronic properties of the layers and devices at different lateral scales at early process steps**. Correlation with the microstructural and compositional analysis of the layers by complementary characterisation techniques as well as with the device optoelectronic properties has allowed the identification of **key quality indicators** in terms of cell and module efficiency, obtaining relevant information related to the mechanisms of loss of efficiency and their dependence on the main process parameters. This knowledge is strongly required for device and process optimisation in terms of both cell efficiency and process uniformity.

6) **To validate and develop higher innovative processes and materials for the future generation of chalcogenide based cells and modules:**

Higher innovative approaches with significant potential for lowering of production costs and sustainable implementation at mass production levels addressed in SCALENANO included:

- Processes based on novel cost efficient deposition techniques as Electrostatic Spray Assisted Vapour Deposition (ESAVD): these are non-vacuum process strategies with potential capabilities for the synthesis of complex CIGS absorbers with graded composition across layer thickness;
- Extension of non-vacuum based processes (ED, printing, ESAVD, solution based processes) for the synthesis of CZTS(Se) layers and based device, addressing the problem which will be created by scarce materials like Indium in the future mass deployment of CIGS technologies.





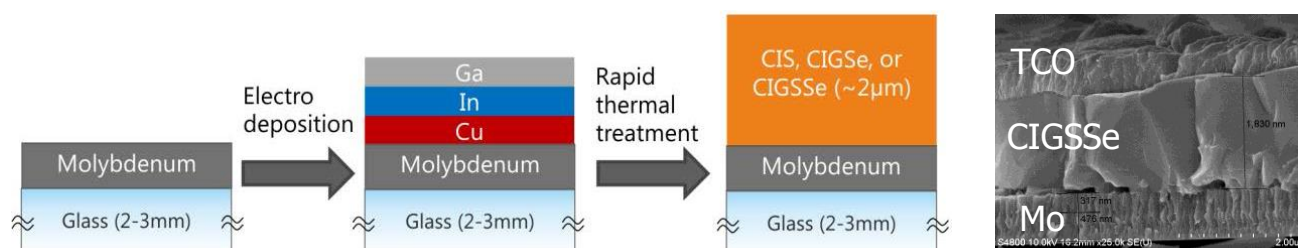
### 1.3 Description of the main S&T results/foregrounds

As described in the previous section of the report, SCALENANO is centred in the development and scale-up of an innovative chalcogenide photovoltaic (PV) technology using environmentally friendly and sustainable technologies with lower costs and higher efficiencies. The activities developed since the beginning of the project have allowed the achievement of the main objectives of the project in terms of device efficiency, up-scaling of processes, manufacturing costs and demonstration of relevant assessment methodologies and process monitoring tools, with results which are ahead from those initially planned.

Next sections describe the main results that have been obtained during the development of SCALENANO, in relation to the key objectives and targets that were initially defined in the project.

- **Key objective 1: To develop a reliable low cost (30% lower than PVD process) and efficient (>95% of PVD process) electrodeposition (ED) based technology with a better lateral homogeneity (+/-5%/m<sup>2</sup>):**

SCALENANO has achieved strong advances in the optimisation of electrodeposition (ED) based processes for the achievement of higher efficiency low cost Cu(In,Ga)(S,Se)<sub>2</sub> (CIGS) PV devices and their successful up-scaling to large areas with very high uniformity. In these processes, the CIGS absorber layers are synthesised following two steps: i) sequential electrodeposition of Cu/In/Ga metal precursors, and ii) atmospheric pressure Rapid Thermal Process (RTP) chalcogenisation with elemental S and Se, according to the schematic diagram shown in Figure 1.



**Figure 1:** Main process steps involved in the synthesis of the CIGS absorbers. Left: SEM cross section image of a typical CIGS cell.

The successful development of these processes has led to a strong improvement in the performance of devices at both cell and module level. Improvements achieved since the beginning of the project are summarised in Table 1 in terms of cell and module efficiency for three device sizes, 60x120cm<sup>2</sup> modules, 30x60cm<sup>2</sup> modules and test cells 0.5cm<sup>2</sup>. As shown in the Table, initial processes (up to Baseline v3) were based on Cu(In,Ga)Se<sub>2</sub> (CIGSe) absorbers, and since month 30 faster significant advances in the performance of the devices were achieved by the inclusion of sulphur (CIGSSe: Cu(In,Ga)(S,Se)<sub>2</sub>), leading to the consecutive development of the Baseline v4 and Baseline v5 processes.

Up to month 22, prototypes at NEXCIS pilot line were only finished as 30x60 cm<sup>2</sup> modules coming from 60x120cm<sup>2</sup> hybrid processes for ED and thermal treatment steps. However, from month 22 a complete 60x120cm<sup>2</sup> fabrication line, including the CdS and ZnO deposition steps in full industrial size, has been set up. In month 24 we reported the first 60x120cm<sup>2</sup> module with a record efficiency of 12% (AA). From the month 24 to month 30 period, several modules 60x120cm<sup>2</sup> have been produced with an average efficiency of 12.4% (AA) and a best certified module at 13.2%. Finally, in month 36 the average efficiency of 60x120cm<sup>2</sup> modules was raised up to 13.3% with a certified best module at 14.0% (aperture area) yielding a 92.8W power output under the standard testing condition.

These results are already comparable to the average module efficiency in production using vacuum-based processes (13% - 14%). At cell level, highest efficiency achieved is 17.3%, which constitutes a world efficiency record for devices fabricated by solution based processes.





	6 <sup>th</sup> Month report Baseline v1 & v2	12 <sup>th</sup> Month report Baseline v2 CIGSe	18 <sup>th</sup> Month report Baseline v3 CIGSe	24 <sup>th</sup> Month report Baseline v4 CIGSSe	30 <sup>th</sup> Month report Baseline v4 CIGSSe	36 <sup>th</sup> Month report Baseline v5 CIGSSe
Average modules 60x120cm <sup>2</sup> (aperture area) – internal	-	-	-	-	12.4	<b>13.3</b>
Best module 60x120cm <sup>2</sup> (aperture area) – certified	-	-	-	12.0	13.2	<b>14.0</b>
Average modules 30x60cm <sup>2</sup> (aperture area) – internal	10.5	11.2	11.7	13.5	13.8	<b>14.1</b>
Best module 30x60cm <sup>2</sup> (aperture area) – certified	12.3	12.3	13.1	14.2	14.5	<b>14.8</b>
Average of 99 0.5cm <sup>2</sup> cells on 30x60cm <sup>2</sup> sample (internal measurements – active area w/out ARC)	<i>12.6</i>	<i>12.9</i>	<i>13.6</i>	<i>14.9</i>	<i>15.1</i>	<b><i>15.3</i></b>
Best certified cell aperture area with ARC	14.9 (0.5cm <sup>2</sup> )	14.9 (0.5cm <sup>2</sup> )	15.4 (0.5cm <sup>2</sup> )	15.8 (1cm <sup>2</sup> )	16.0 (1cm <sup>2</sup> )	<b>17.3 (0.5cm<sup>2</sup>)</b>

**Table 1:** Efficiency improvements at cell- and module level achieved since the beginning of SCALENANO

These advances have allowed the demonstration of the industrial transferability of a CIGS ED pilot line for the fabrication of large area (60x120 cm<sup>2</sup>) modules with the fulfilment of all the criteria that were defined in the project, including:

i) An efficiency higher than 95% of the average efficiency of vacuum based industrial modules:

The comparison with the efficiencies of commercial available modules that are manufactured by the main CIGS companies (Solibro-Hanergy, TSMC, Avancis, Solar Frontier, Stion) shows that the large area modules developed in SCALENANO have achieved an average efficiency of 96% of that of the reference vacuum based modules.

ii) An improved (lower than 5%) lateral homogeneity:

Upscaling of the ED and RTP processes to large area substrates has been developed with a very large uniformity, including:

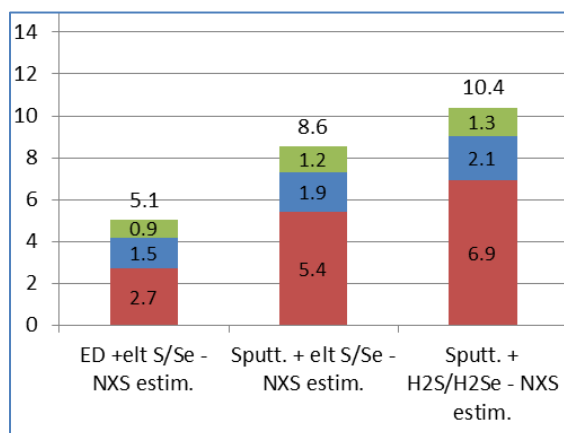
- ✓ Electrodeposited Cu, In, Ga and overall thickness with deviation < 4%
- ✓ Composition uniformity of Cu/(Ga+In) relative content  $\pm 0.02$
- ✓ Composition uniformity of Ga/(Ga+In) relative content  $\pm 0.007$
- ✓ Chalcogen content after thermal treatment with deviation < 4%
- ✓ No segregation of detrimental secondary phases on the absorber

These criteria have been achieved on a 60x120 cm<sup>2</sup> panel size, which is greater than the initially targeted in the project in terms of substrate size (30x60 cm<sup>2</sup>).



iii) Low fabrication costs (30% lower than for equivalent PVD technology):

Figure 2 shows the costs estimated for the fabrication of the CIGS absorbers according to the model developed in SCALENANO. The figure compares the costs calculated for the SCALENANO process (ED + chalcogenisation with elemental S and Se) with those of equivalent 2-step vacuum based processes (precursor deposited by sputtering and annealing with elemental S/Se chalcogens, precursor deposited by sputtering and annealing with hydride gases). The last process is representative for the most common process for producing commercial CIGS modules (Solar Frontier, TSMC) while the first and second bars in the figure are representative of the cost reduction when modifying the precursor deposition step and the annealing step, respectively.



**Figure 2:** CIGS absorber production cost in c€/Wp (calculations based on a module efficiency of 14% according to the cost model developed by NEXCIS). The columns include the costs estimate for Facility and Maintenance (green), Depreciation costs (blue) and Maintenance costs (red)

As shown in the figure, replacement of the all-sputtered precursor by an electrodeposition step enables a 40% cost reduction in CIGS production, with a cost dropping from 8.6c€/Wp to 5.1c€/Wp. The reduction is sensible especially on the material costs dropping by 50% which is due to higher utilization rate of expensive In and Ga metals and lower cost of baths compared to sputtered targets. The cost estimation of the standard 2-step PVD process agrees with already published values. The 2-step process developed at NEXCIS and reported in the SCALENANO project shows that a 5 c€/Wp cost is reachable for a process based on ED and elemental S/Se chalcogenation, which corresponds to a 50% cost reduction when comparing against standard vacuum processes and already a 40% reduction (8.6 → 5.1 c€/Wp) when using electrodeposition for the precursor step.

According to these results, **all the quantitative targets and the Performance Research Indicators that were defined in the project in relation to Key Objective 1 have been fully achieved.** The Performance Research Indicators for Key objective 1 were the following:

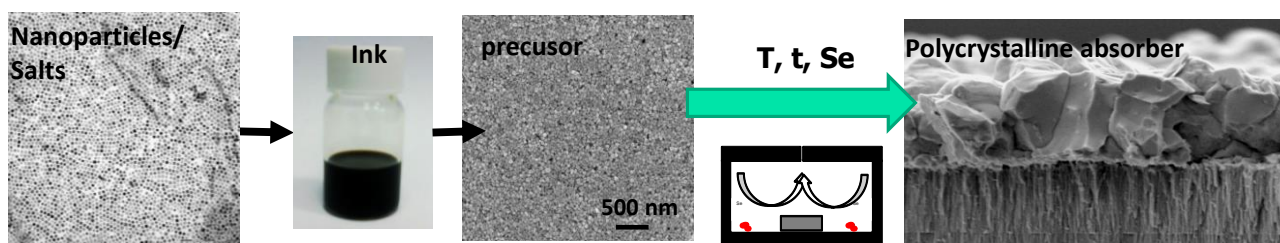
- ✓ CIGS ED layer on an area of 15x15 cm<sup>2</sup> without segregation of secondary phases (D1.1, M18): **achieved on an area of 60x120 cm<sup>2</sup> (instead of 15x15 cm<sup>2</sup>).**
- ✓ CIGS electrodeposited device with efficiency ≥ 12% on an area of 15x15 cm<sup>2</sup> (MS2, M30): **achieved with a certified 14.0% efficiency on 60x120cm<sup>2</sup> and a certified 14.8% efficiency on 30x60cm<sup>2</sup> (instead of 15x15cm<sup>2</sup>).**
- ✓ CIGS electrodeposited layer on an area of 30x60 cm<sup>2</sup> with <5% of deviation in the compositional and thickness uniformity (D1.4, Month 36): **achieved with relative standard deviation values lower than 4% (from 60x120cm<sup>2</sup> mapping on CIS, CIGS & CIGS/CdS material with XRF and PL measurements).**



- ✓ CIGS cell with efficiency  $\geq 95\%$  of reference PVD cells on  $30 \times 60 \text{ cm}^2$  substrates with improved (better than 5%) lateral homogeneity and low fabrication costs (30% lower than for equivalent PVD processes (D1.6/MS4, Month 42):  
**achieved with efficiency  $\geq 96\%$  of reference PVD cells on  $60 \times 120 \text{ cm}^2$  substrates with lateral homogeneity  $< 4\%$  and fabrication costs 40% lower than for equivalent PVD technology.**

- **Key objective 2: To develop a new alternative scalable process compatible with high throughput (established target at pilot line 1m/min) requirements:**

SCALENANO has also involved a significant activity in the development of printing based processes alternative to the ED ones for the synthesis of the absorbers with very high throughput and low cost. Printing based processes have the highest potential to achieve very high throughput and process rate. This activity has included the development of CIGS and CZTS absorbers that were prepared by several technologies being the use of inks the central point in common (Figure 3). The main deposition technologies used were doctor blading, spin coating and pulsed spray deposition. The inks used were based either on nanoparticles or on the relevant metal salts.

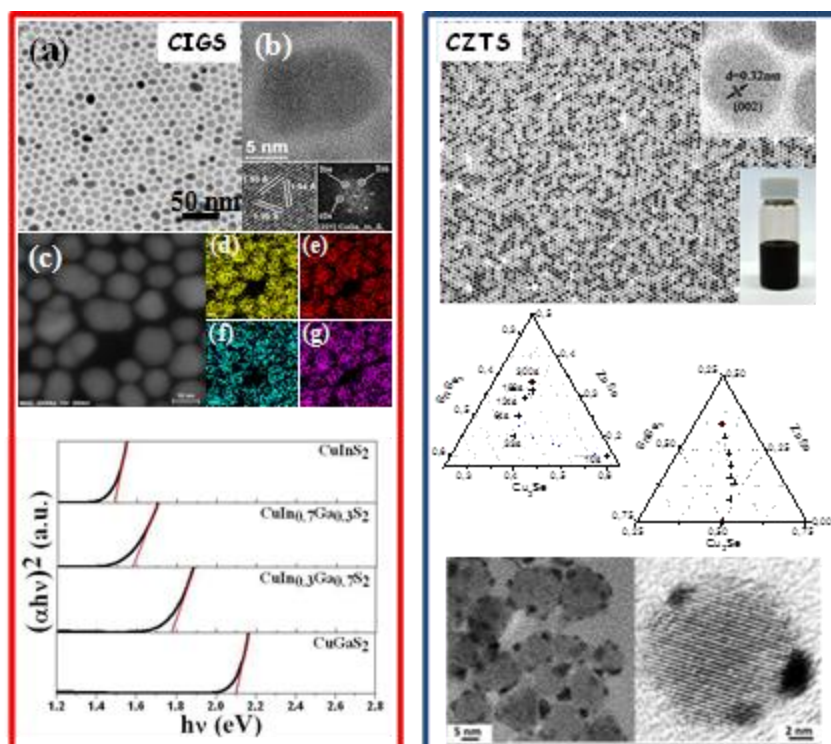


**Figure 3:** Main steps involved in the synthesis of the absorbers by printing based techniques.

The main achievements obtained in relation to this key objective are:

- **Definition of environmental friendly synthetic routes for the preparation of CIGS and CZTS nanocrystals with controlled composition and phase:**

IIT and IREC groups have developed several synthesis protocols for the production of CIGS (IIT) and CZTS (IREC) nanoparticles (NPs) at the gram scale and with controlled phase and metal ratios. Figure 4 shows representative TEM micrographs of the CIGS and CZTS nanoparticles obtained. EELS and EDX mapping were used in both cases to demonstrate that all particles contained the different elements and that nanoparticles were characterized by a narrow composition distribution. Both for CIGS and CZTS, composition could be tuned by adjusting the precursor concentrations as shown by the UV-Vis CIGS measurements and on the CZTS ternary diagrams in the figure.



**Figure 4:** Representative TEM and HRTEM micrographs of CIGS and CZTS NPs. In the left panel, elemental mapping of the CIGS nanoparticles and the UV-vis spectra of nanoparticles with different compositions are displayed. In the right panel, ternary diagrams with the composition of different CZTS batches and a TEM and a HRTEM image of a CZTS-metal nanoparticle are shown.

- Development of NP and ionic/molecular ink formulations and printing procedures for the preparation of solar-cell grade CIGS and CZTS layers (high crystallinity and uniformity, control over composition and secondary phases) on small (2x2 cm<sup>2</sup>) and large (20x20 cm<sup>2</sup>) substrates:

Specific nanoparticle-based and metal salts-based inks were formulated for the different ink-deposition techniques. Using these inks, the deposition techniques were optimized to produce highly uniform CIGS and CZTS layers. Furthermore, layers were crystallized under controlled atmosphere to produce solar-cell grade absorber layers on small and large area substrates. To avoid contamination with carbon when preparing absorber layers from nanoparticle-based inks, procedures to exchange the organic ligands used to control the nanoparticle synthesis by shorted inorganic/organic ions/molecules were developed. We also took advantage of this ligand exchange procedure to introduce crystallization promoters such as Na or Sb.

A potentially large-scale pulsed spray deposition system was developed and optimised to produce small (2x2 cm<sup>2</sup>) and large (20x20 cm<sup>2</sup>) precursors free of cracks and with acceptable uniformity. Recrystallization processes were developed either in conventional tubular furnaces (2x2 cm<sup>2</sup> precursors) or in RTP systems (20x20 cm<sup>2</sup> precursors) using in all cases elemental Se for chalcogenisation, and different selenisation strategies (open reactor, close box, deposition of Se on precursor surface...) and Mo substrate configurations were investigated in order to optimise their crystalline quality while preventing excess selenisation of the Mo back contact region.

- Development of solar cell prototypes:

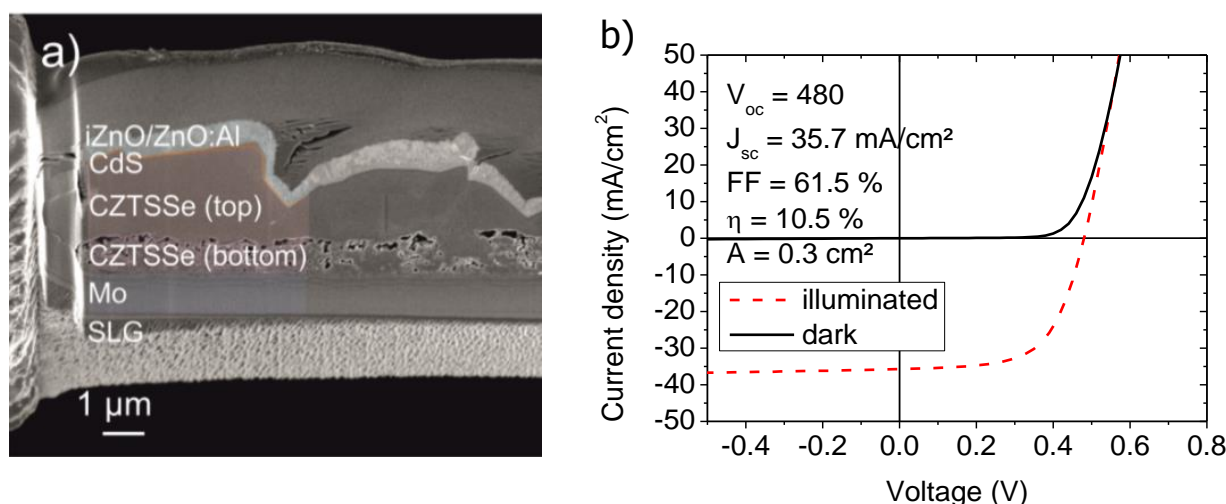
CIGS and CZTS solar cell prototypes were fabricated using the described high throughput processes. While we succeeded to produce highly crystalline CIGS and CZTS layers, best efficiencies were obtained using a





metal salt-based ink and a crystallization process resulting in a double layer CZTS structure with a crystalline CZTS on the top and small crystals on the bottom (Figure 5(a)). The use of chemical bath deposition to introduce the top CdS layer allowed CdS to penetrate through the porous structure and extract charges even from the not perfectly crystalline material as demonstrated by electron beam induced current (EBIC) analysis.

Best efficiencies achieved were obtained for the  $2 \times 2 \text{ cm}^2$  precursors:  $\eta = 4.9\%$  (CIGS, NP based inks);  $\eta = 10.3\%$  (CZTS (salt-based inks). Figure 5(b) shows the IV characteristics of the best cell achieved by these processes. According to these data, the efficiency target defined in this key objective (80% efficiency than that of equivalent PVD processes) has been successfully achieved for the CZTS printed processes on  $2 \times 2 \text{ cm}^2$  substrates: in this case the efficiency obtained is comparable to the best efficiency achieved in CZTS devices using PVD based processes at the SCALENANO consortium (10.6% at IREC), and close to the world record CZTS efficiency reported for coevaporation processes (11.6% by coevaporation processes at IBM) [Advanced Energy Materials (2015) DOI: 10.1002/aenm.201401372].



**Figure 5:** Cross section SEM Image (a) and I-V characteristics (b) of best CZTS printed cell

These results confirm the higher potential of strategies based on printing processes using salt-based inks instead of the nanoparticle based inks. On the other hand, the low efficiency values obtained on  $20 \times 20 \text{ cm}^2$  precursors (up to 2.9%) indicate that the extension of these processes to larger area substrates still requires for a significant process optimization to increase the device efficiency.

These results demonstrate the **fulfillment of main quantitative targets and Performance Research Indicators that were defined in the Project in relation to Key Objective 2**. The Performance Research Indicators defined for Key objective 2 were:

- ✓ A synthetic procedure for the preparation of  $\text{Cu(In,Ga)(S,Se)}_2$  and  $\text{Cu}_2\text{ZnSn(S,Se)}_4$  nanoparticles (MS6, Month 12):  
**achieved for both CIGS and CZTS systems with controlled composition, crystalline structure and size.**
- ✓ MS7: An ink formulation and an established procedure to obtain homogeneous solar-grade layers (MS7, Month 26):  
**achieved, including new formulations of nanoparticle based inks and metal salt based inks.**
- ✓ MS11:  $20 \times 20 \text{ cm}^2$  CIGS and CZTS solar cell prototypes with 80% efficiency compared with equivalent cells produced by evaporation techniques (MS11, Month 40):  
**partially achieved. Efficiency target only achieved for CZTS printed prototypes (small area).**



- ✓ Analysis of viability of developed processes for low cost (30% lower than PVD processes) and high throughput (1 m/min at pilot one) large scale production of solar cells (D2.7, Month 42): **achieved. This analysis demonstrates that the preparation of precursor inks, especially those formed from metal salts, and their spray deposition are a technologically viable process for the fabrication of solar cells in a large scale production scenario. However, processes need to be further optimized to achieve sufficient efficiencies. From the cost point of view, the estimations developed in the project show a significant reduction for CZTS printed processes in relation to the PVD ones. Nevertheless, lowest estimated costs are still achieved for the electrodeposition based processes.**

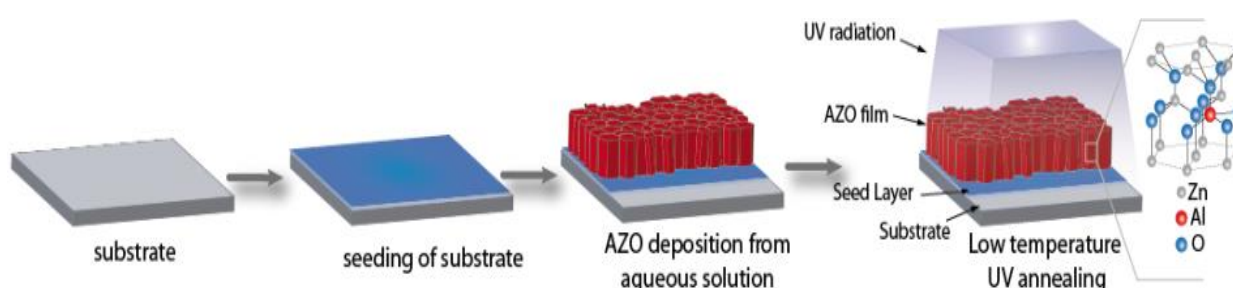
- **Key objective 3: To develop optimized TCO layers (transparency >80%, sheet resistance < 100 Ohm sq) by scalable non vacuum based processes:**

The main goal within this key objective is to demonstrate a transparent front contact based on i-ZnO and TCO (Al-doped ZnO or another metal oxide), suitable for high efficiency CIGS/CZTS solar cells (transparency >80%, sheet resistance <100 Ohm sq). Importantly, the maximum processing temperature for a non-vacuum deposition should not exceed 200-300°C, because higher temperatures are detrimental for other semiconductor layers.

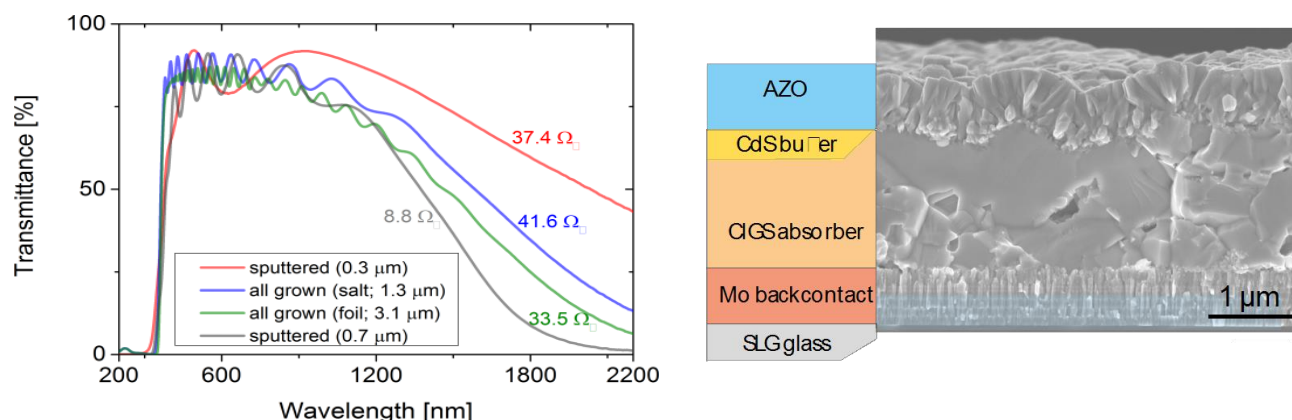
Two main strategies have been identified for the fulfilment of this objective: i) Chemical Bath Deposition (CBD) based processes for the growth of Al-doped ZnO (AZO) and ii) ESAVD based processes for the synthesis of Ag nanowires based TCO's. In both cases, the viability of the developed processes for the achievement of the transparency and conductivity requirements has been demonstrated. In addition, the compatibility of the developed processes with their implementation in CIGS based solar cells has also been validated, fabricating solar cell prototypes with CIGS absorbers synthesized by either electrodeposition (ED) and PVD processes with efficiencies comparable to those achieved with reference sputtered TCO layers. This includes very promising results on fully solution based CIGS solar cells which constitute first steps towards the development of fully solution based CIGS PV technologies.

- **Low temperature aqueous solution process for transparent and conductive AZO layers:**

A novel low-temperature (<100 °C) process using aqueous solutions has been developed to deposit doped ZnO layers on any kind of temperature-sensitive substrates (Figure 6). Using the innovative process transparent and conducting AZO layers with sheet resistance of < 50  $\Omega/\square$  and optical transmission  $T > 80\%$  could be obtained (Figure 7). The AZO layers have been implemented as transparent electrical contacts in CIGS solar cells, yielding up to 14.4% (with sputtered i-ZnO interlayer) or 13.8% (all-solution processed solar cell with ED CIGS absorber).



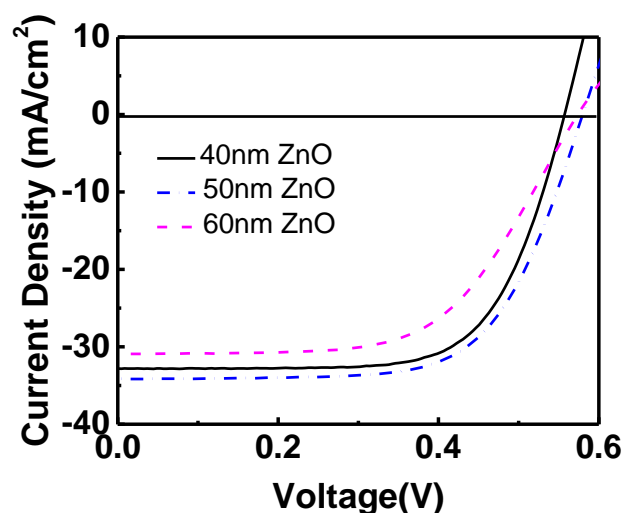
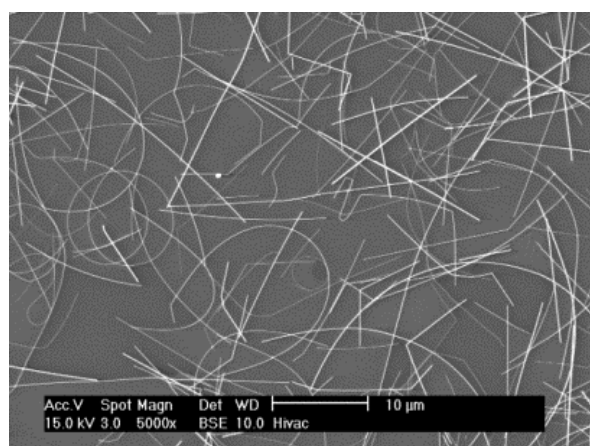
**Figure 6:** Schematic representation of main process steps involved in the CBD growth of AZO layers.



**Figure 7:** Left: Optical transmission of CBD and sputtered AZO layers with different sheet resistances. Right: Cross section SEM image of a 13.8% efficiency solution processed CIGS solar cell fabricated with the CBD AZO layer.

- Electrostatically spray assisted vapour deposition (ESAVD) of Ag-ZnO hybrid layers:

The proprietary ESAVD process has been developed to obtain new TCO layers that are based on Ag nanowires (NWs) embedded in a ZnO matrix (Figure 8 left). Optimisation of the processes and the density of nanowires gives layers with optical transmission  $T > 80\%$  and sheet resistance of  $< 50 \Omega/\square$  micron. These conductive layers have also been implemented as front electrical contact in electrodeposited CIGS solar cells yielding up to 13.8% efficiency for full solution based devices (including a 50 nm thick intrinsic ZnO layer deposited by a non vacuum chemical based deposition method) (Figure 8 right).



**Figure 8:** Left: SEM image of an Ag NW-ZnO hybrid layer (sheet resistance 50 Ω/micron). Right: I-V curves of full solution CIGS cells with Ag NW-ZnO hybrid TCO, including i-ZnO layers deposited with different thickness. The highest efficiency of 13.8% is achieved for a 50 nm thick i-ZnO layer.

- Zn(O,OH,S) buffer layers by CBD:

An additional objective of SCALENANO was to define an optimum TCO-buffer combination for CIGS or CZTS(Se) absorber layers using alternative Cd-free buffer layers. In the project, a CBD process has been developed for the growth of Zn(O,OH,S) buffer layers and it has been demonstrated on CZTSSe solar cells,





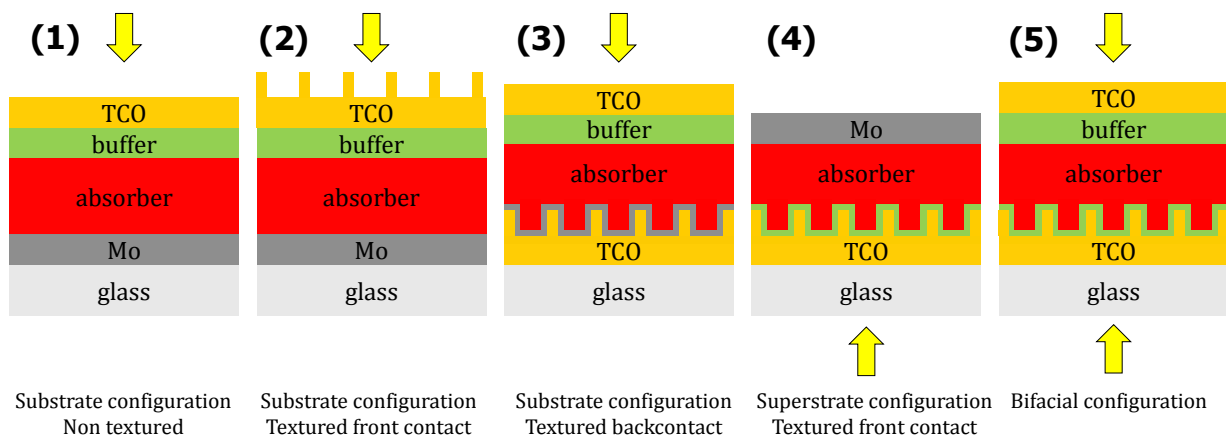
obtaining up to 5.8% cell efficiency as compared to 7% that was achieved for equivalent cells with reference CdS buffers.

According to these results, **all the quantitative targets and the Performance Research Indicators that were defined in the project in relation to Key Objective 3 have been fully achieved.** The Performance Research Indicators for Key objective 3 were the following:

- ✓ Established viability of developed strategies (CBD, ESAVD) for achieving TCO layers with contact sheet resistance < 100 Ohm sq and optical transparency > 80% (MS12/MS13, Month 24):  
***achieved using two different strategies (CBD of AZO layers/ ESAVD of Ag NW-ZnO hybrid layers, including also the demonstration of full solution based cells using both kinds of non-vacuum TCOs with efficiencies similar to those achieved with reference sputtered AZO.***
- ✓ Analysis of the scalability of developed non vacuum deposition processes to module surfaces (D3.5, Month 40):  
***achieved. The scalability analysis shows that both processes (CBD of AZO layers/ ESAVD of Ag NW-ZnO hybrid layers) are suitable for large area deposition, even if deposition area is still low (up to 15x15 cm<sup>2</sup> for CBD AZO) and further development is required (including material issues as the stability of the deposited layers) for their successful implementation in large area modules.***

• **Key objective 4: To improve cell architectures on nanostructured TCOs for higher efficiencies cells and modules:**

Novel chalcogenide cell architectures as those based on the implementation of nanostructured TCO's formed by columnar ZnO nanorod arrays (NRAs) have been investigated for the development of higher efficiency devices. This has involved the development of a new 2-D model for the simulation of the optical properties of the different device configurations. The ZnO NRAs were synthesised by different techniques (electrochemical processes, ESAVD), and they were implemented in the different cell configurations, according to the results of the simulation studies, for their experimental validation. Figure 9 shows the device configurations that have been studied. The non-textured substrate configuration is the classical CIGS cell architecture that has served as reference in this study. Also in substrate configuration the ZnO nanorods can be implemented on top of the finished solar cell, providing a textured front contact, or serve as textured substrate providing a nanostructured back contact. In a superstrate configuration, either the fabrication of a complete cell (including metallic back-contact) on top of the ZnO nanorod or a cell working in bifacial configuration can be envisaged.



**Figure 9: ZnO NRA's based cell configurations investigated in SCALENANO**

The main achievements obtained in relation to this key objective are:



- Development of an optical model for the simulation of the different device configurations

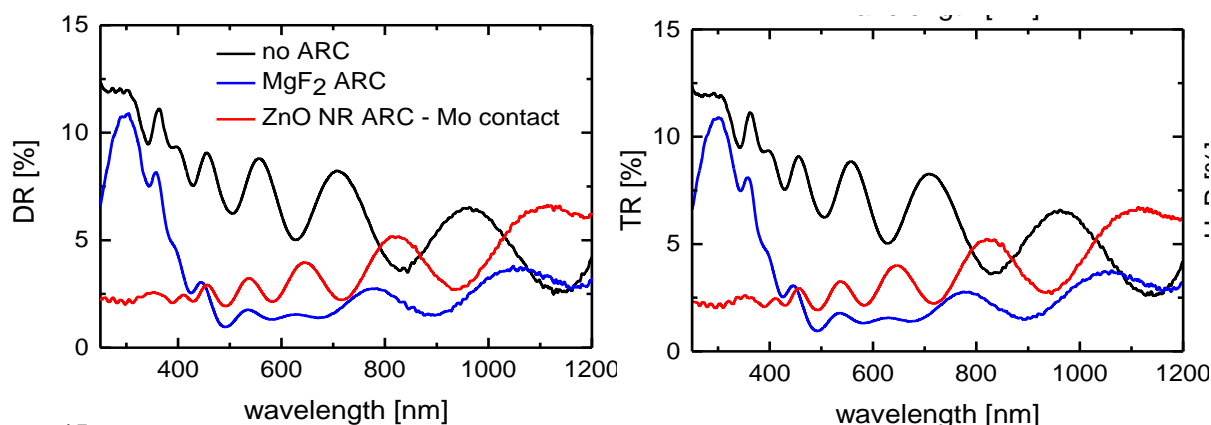
A new 2-D optical model has been developed at CEA. The model has been calibrated with experimental data from reference cells produced with different textured TCOs, and it has been applied to the analysis of the different architectures proposed in the project. The optical simulations have shown that it is possible to achieve an increased absorption in the CIGS layer by implementing ZnO nanorods (NRs) in the cell. The main effect occurring in the presence of the NRs is an antireflection effect, thus the configurations 2, 4 and 5 are the most advantageous. The results on the textured back contact, and the similar - bifacial configuration with front illumination - showed that, since the NRs are placed at the bottom of the structure and are too small with respect to the wavelengths, they are not appropriate to produce increased absorption in the CIGS layer by photon scattering. The simulations also showed the importance of the positioning of the NRs in the cell. To obtain significant antireflection effect, the refraction index difference between the ZnO NRs and the surrounding material should be high enough. In configuration 2, less gain is observed when the NRs are encapsulated because the refraction indices of the materials are similar. In this context also, the simulations showed that when the NRs are inserted in the cell (like the superstrate or the bifacial configurations) enhanced absorption is obtained when the absorber layer penetrates in between the NRs.

- Development of electrochemical and ALD based processes for fabrication of ZnO NRAs

A strong effort has been devoted to the development of electrochemical and ALD based processes for the growth of ZnO NRAs with well controlled characteristics, including the dimension of the nanowires (width, length), and their density and orientation. By tuning the deposition parameters, the optical properties (total reflectance and transmittance and scattering properties) of the nanorods can be adjusted. Moreover, with the electrodeposition technique, highly homogeneous nanostructures ( $\pm 5\%$ ) were successfully achieved on large substrates ( $30 \times 30 \text{ cm}^2$ ), demonstrating the scalability of these processes to the development of large area devices.

- Implementation of ZnO NRs on a substrate configuration with textured front contact: demonstration of antireflection effect of the ZnO NRs

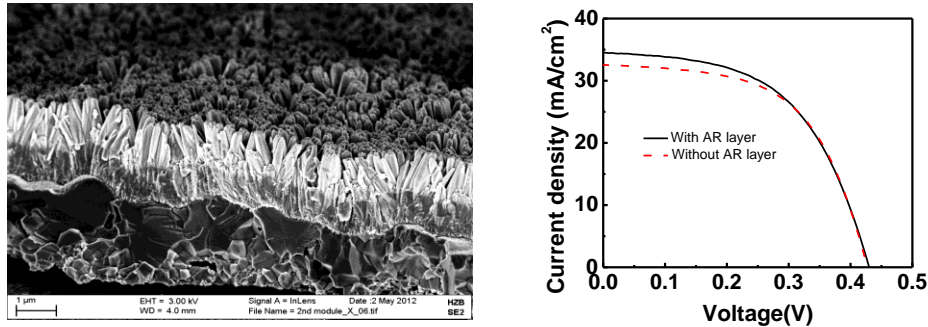
The optical characterisation of the deposited ZnO NRAs has confirmed their antireflection behaviour as predicted by the simulations. These NRAs exhibit enhanced diffuse transmittance and antireflection properties, as shown in Figure 10. These are important for maximizing the amount of light delivered to the solar cell absorber in configuration 2, where the ZnO NRA is acting as antireflective coating (ARC) in a standard CIGS cell architecture.



**Figure 10:** Diffuse reflectance (DR) and total reflectance (TR) of CIGS cells with a) no antireflective coating (ARC), b) standard  $\text{MgF}_2$  ARC, and c) ZnO NRA ARC. In the short wavelength range ( $< 450 \text{ nm}$ ) ZnO NRA show higher anti-reflection performance than  $\text{MgF}_2$ .



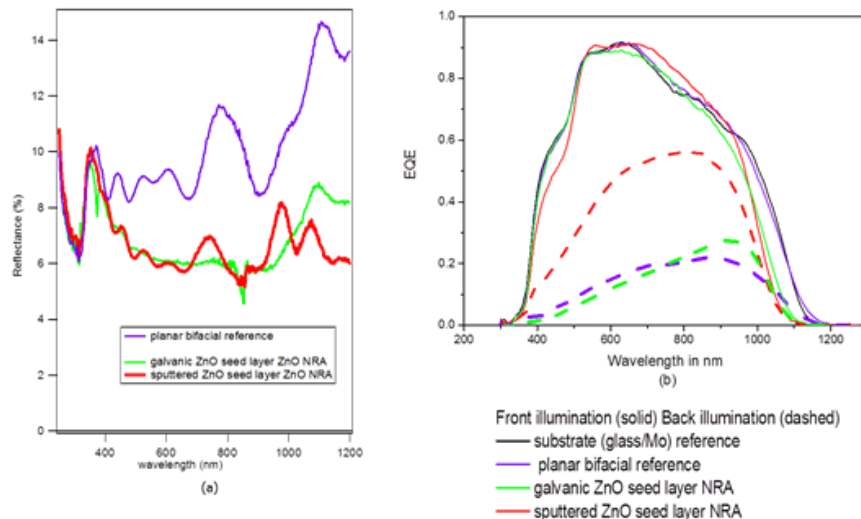
The optoelectronic characterisation of the CIGS cells developed with configuration 2 has allowed to experimentally demonstrate the ARC effect of the NRs on the characteristics of the devices, observing an enhancement of the EQE and  $J_{sc}$  without deterioration of the  $V_{oc}$  and FF when using short (100-200 nm) and low packing density NRs. These effects are more relevant for PVD based cells (that have a lower surface roughness) and decrease when the cells are encapsulated, in agreement with the simulation results.



**Figure 11:** Left: SEM image of a cell with ZnO nanorods deposited as ARC on the surface of the TCO. Right: I-V characteristics of equivalent cells with and without ZnO NRA ARC, showing the increase in the short circuit current with the ZnO NRs.

- Implementation of ZnO NRs on superstrate and bifacial cell configurations

The bifacial cell in rear illumination is another example of the successful implementation of ZnO NRs in CIGS solar cells. In this case, the consequence of the NRs presence is double: increased absorption is due to antireflection effect and also enhanced carrier collection. These effects are determining of the increase of the EQE that has been experimentally observed in bifacial cells fabricated with a 0.7 μm thick absorber for back illumination, as shown in Figure 12 (right). As pointed out by the simulations, the nature of the buffer layer should be controlled, as the gain due to the NRs could be lost if an absorbing material is formed. Spray pyrolysis deposition of MoO<sub>3</sub> seems a promising technique to avoid this.



**Figure 12:** Reflectance for rear illumination (left) and EQE (for both front (solid line) and rear (dashed line) illumination) (right) as a function of wavelength for ZnO NR solar cell bifacial devices and equivalent reference devices (absorber thickness 0.7 μm).



- Identification of technological challenges for the exploitation of the efficiency gain potential of ZnO NRA cell configurations

The intense activities developed in the simulation and technological implementation of the different device configurations has allowed identifying the main technological challenges that have to be solved to exploit their potential to achieve higher device efficiencies. The main challenges are related to the following points:

- i) Need for an improved control of the buffer layer located between the NRs and the absorber, as the gain due to the NRs can be compromised by parasitic absorption in the buffer layer;
- ii) Adaptation of the absorber deposition process so that the ZnO NRs retain their morphology during the subsequent processing steps. The standard PVD technique destroys the NRs because of the corrosive selenium atmosphere at temperatures up to 525°C. A lower temperature PVD process was developed and other processes (nano-ink printing, electrodeposition or ESAVD) have been tested. However, the selenisation step remains a critical issue.
- iii) An additional key point in these processes is the filling of the NRs array with absorber. The optical simulation results showed that when the CIGS does not fill the NR array, the optical enhancement is much reduced. Initial tests by ESAVD showed good covering of the NRs and they seem to be intact even after selenization at 400°C and 425°C.

According to these results, **all the quantitative targets and the Performance Research Indicators that were defined in the project in relation to Key Objective 4 have been fully achieved**. The Performance Research Indicators for Key objective 4 were the following:

- ✓ Dimensioning of non-vacuum solar cell by optical simulation based on calibration of reference cells (D4.2, Month 21):  
***achieved, with the development of a new 2-D model for the simulation of the optical properties of the different device configurations.***
- ✓ Choice of new cell architectures based on ED, printing and ESAVD processes (MS16, Month 24):  
***achieved, with the identification (from both the simulation and the experimental characterization of the implemented layers and devices) of the cell structures with highest potential for performance improvement***
- ✓ New high efficient cell architectures with electrodeposition processes (D4.4, Month 36):  
***achieved, with the demonstration of the improved ARC effect in substrate cell configurations with textured front contact and of the increased absorption and carrier collection in bifacial cells.***

- **Key objective 5: To develop and implement quality control and process monitoring techniques for scaled-up processes in large area substrate:**

Both the process monitoring during the growth of a single layer and the non-destructive characterisation of a complete device are relevant issues for the improvement of the yield and reliability of the processes which is critical for the development of cost efficient PV technologies. This implies the need for the availability of non-destructive techniques and methodologies suitable for the monitoring at both real time (in-situ) and on-line levels of the different process steps involved in the fabrication of the devices, with the determination of quality control parameters relevant for device efficiency, including the detection of layer and device inhomogeneities in the processes up-scaled for the production of large area modules.

In relation to this key objective, a significant effort has been made in SCALENANO in the development, implementation and validation of non-destructive techniques and methodologies for process monitoring at in-situ (real time) and ex-situ levels at both laboratory and pilot line levels. With this goal, different strategies have been developed based in 3 kinds of techniques: Light scattering based techniques, Photoelectrochemical (PEC) based techniques, and Electroluminescence (EL) / Photoluminescence (PL) based techniques.



In the frame of this key objective the following process methodologies have been developed and validated in SCALENANO:

- Light scattering based techniques: In-situ elastic light scattering analysis of the electrodeposited layers during the electrochemical growth of the metallic precursors

IREC has developed and validated a new methodology that is based on Elastic Light Backscattering (ELBS) measurements for the real time in-situ monitoring of the electrodeposition (ED) process steps in the fabrication of the metallic Cu/In/Ga precursors. This has included the design and implementation of several optical probes that were optimised for the real time monitoring of the following ED process steps: i) In ED on Cu/Mo/SLG substrates and ii) Ga ED on In/Cu/Mo/SLG substrates. Each of these processes gives an ELBS pattern that is characteristic of the process and is directly related to the changes in the morphology and structure of the surface of the sample during the layer growth. The correlation of the ELBS signal with the detailed ex-situ electron microscopy and EELS analysis of samples that were growth during different times corresponding to the different process stages has allowed identifying in the ELBS pattern the main steps taking place during the process, including the initial nucleation, growth and coalescence of the deposited grains.

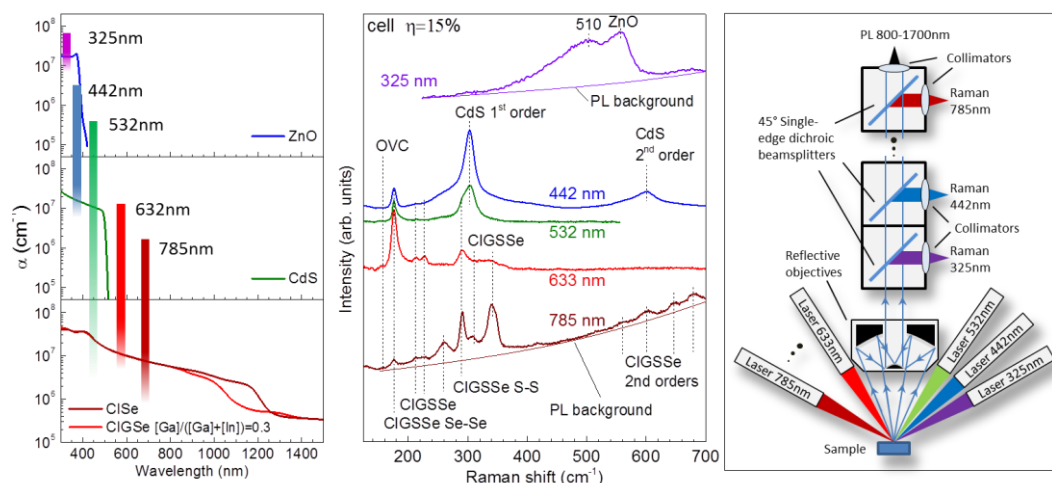
The sensitivity of the ELBS pattern to changes of different relevant process parameters has demonstrated the viability of the ELBS tool for the real-time assessment of the ED processes. For the In ED process step, the parameters included in the study have been the nominal deposition current, the pH of the electrolytic bath and the state of the surface of the Cu/Mo/SLG substrates. In the case of the Ga ED process step, the parameters analysed have been the nominal deposition current and the content of Ga in the electrolytic bath. In all these cases, methodologies suitable for the quantitative assessment of the different process parameters have been developed. These methodologies allow for the detection of relative changes in the analysed parameters that are in the range of  $\pm$  (1% - 10%) of the nominal values corresponding to the baseline processes developed at NEXCIS.

- Light scattering based techniques: Multi-excitation wavelength Raman /PL methodology for on-line assessment of the CIGS cells and modules

A multi-excitation wavelength Raman/PL methodology has been developed at IREC for the on-line non-destructive selective assessment of the different layers in the cell heterostructure. The methodology is based in the existence of a decreasing band gap from the top (Al-ZnO) layer to the bottom (absorber) layer of the Al-ZnO/buffer/absorber layers stack in the solar cell. This device structure allows the use of different excitation wavelengths coupled with the band gap energy of each layer, as shown in Figure 13 (left). This coupling induces a resonant excitation mechanism that increases significantly the efficiency of the Raman scattering process, allowing the selective measurement of quality control indicators that are strongly relevant for device efficiency. This includes

- ✓ The electrical conductivity of the Al doped ZnO window layer in the devices (from the analysis of the relative intensity of a Raman defect induced band from UV resonant Raman measurements);
- ✓ The thickness uniformity of the CdS buffer layer (from resonant Raman scattering measurements performed with 442 nm or 532 nm excitation wavelengths);
- ✓ The content of Ordered Vacancy Compound (OVC) phases at the surface region of the absorber (from resonant Raman scattering measurements performed with 785 nm excitation wavelength);
- ✓ The relative Ga/(In+Ga) content at the surface region of the CIGSe absorbers (from PL measurements performed with 532 nm, 633 nm, or 785 nm); and
- ✓ The relative S/(S+Se) content at the surface region of the CIGSSe absorbers (from Raman scattering measurements performed with 633 nm).



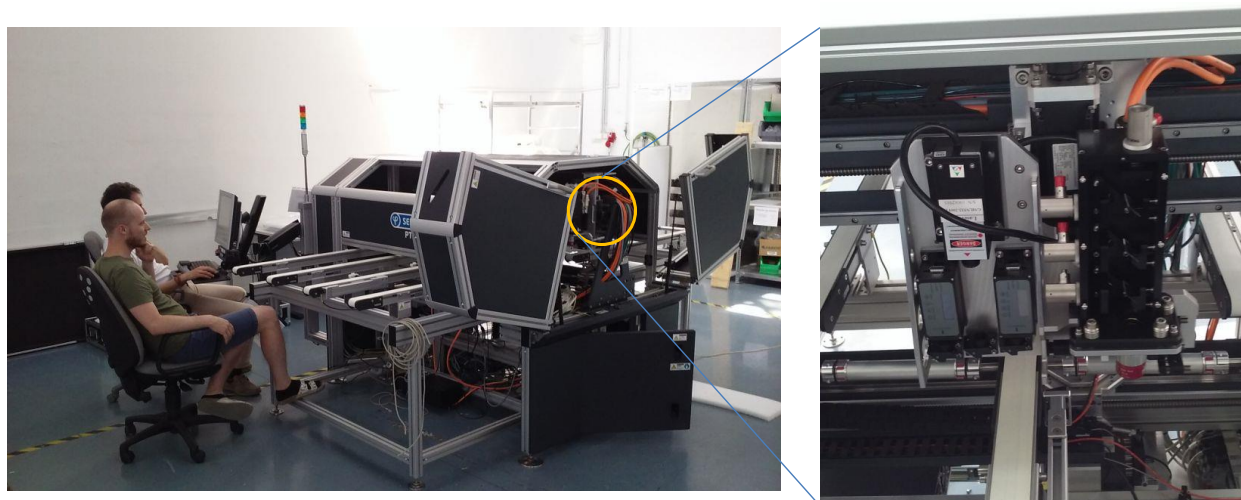


**Figure 13:** Left: Optical absorption of the ZnO, CdS and CIGS layers. Vertical lines show the excitation wavelengths suitable for selective layer assessment. Mid: Typical Raman spectra measured from the different layers in a CIGSSe cell. Right: Schematic diagram of a multi-wavelength excitation probe.

This methodology has been systematically applied for the analysis of the uniformity of the CIGSe and CIGSSe absorbers and cells developed at NEXCIS corresponding to the different baseline processes indicated in Table 1. The correlation of these data with the optoelectronic characterisation of the cells has corroborated the strong impact of the Raman/PL quality control indicators on the efficiency of the devices, demonstrating the viability of the developed methodology for the detection of efficiency losses related to presence of different kinds of inhomogeneties in the devices. These can be related to i) the electrical conductivity of the AZO window layer, ii) the thickness of the CdS buffer layer, and iii) the chemical composition and OVC content at the surface region of the absorber layer. For these measurements, a modular/portable Raman/PL setup has been implemented and tested at laboratory scale. This includes a first proof of concept of a multi-excitation optical probe that allows the simultaneous measurement of Raman and/or PL spectra with different excitation wavelengths, minimising the measuring time required for the assessment of the different layers in the device, according to the optical diagram shown in Fig. 13 (right).

For the implementation of the multi-excitation wavelength Raman/PL combined measurement technique at pilot line level, an up-scaled Raman/PL mapper system has been manufactured at Semilab and tested on 60x120 cm<sup>2</sup> NEXCIS modules. This system follows the same methodology that has been demonstrated at lab-scale in IREC, and was mounted on a Semilab PT5 large area mapping scanner, for mapping of commercial size modules. Figure 14 shows a photographic image of the whole system and the optical probe that has been developed.

As in the case of the setup developed in IREC at laboratory scale, the up-scaled system uses specific spectrometers and detectors for each excitation wavelength, which are optimised in order to improve the resolution and the speed of the measurements. Raman/PL mapping measurements performed on the large area NEXCIS modules have allowed to validate the system for the analysis of the uniformity of the CdS buffer layer and the CIGSSe surface absorber region, with measuring times (after system optimisation) that are below 5 minutes for the realisation of mappings with 180 points, compatible with the implementation of the technique as on-line process monitoring tool.



**Figure 14:** Left: Upscaled Raman/PL system developed at Semilab for mapping of large area modules. Right: Optical probe developed for the system

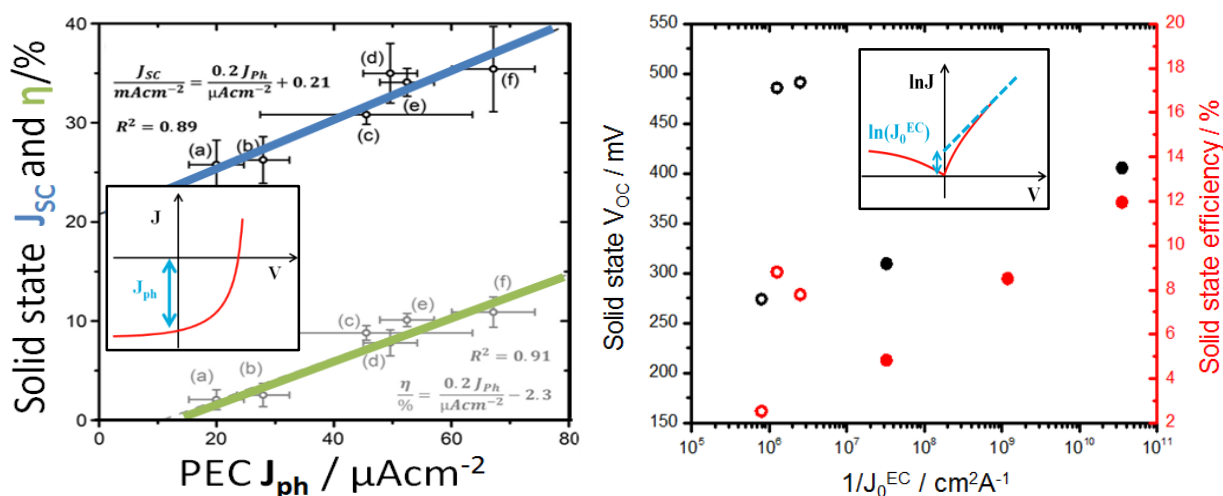
- Photoelectrochemical (PEC) based techniques

A new PEC tool for on-line characterisation of the absorber layers and the absorber/buffer heterostructures has been developed by the UL group. The PEC technique works by forming an electrolyte Schottky diode contact to the semiconductor allowing the separation of excited charge carriers. When the light shines carriers are generated in the absorber, collected by the electrolyte and recorded with a potentiostat. The suitability of PEC has been systematically tested as an analytical technique for characterization of CIGSe absorbers for solar cell performance prediction. Two main quality control indicators have been established and validated ex-situ (Table 2, Figure 15), as described in [D. Colombara et al, Electrochem. Comm. 48, 99-102 (2014)] and [D. Colombara et al, ECS Trans. 66 (9) 19-25 (2015)].

PEC quality control indicator	Solar cell parameter predicted	Acquisition conditions
Electrochemical photocurrent density ( $J_{Ph}$ )	Short circuit current density ( $J_{SC}$ )	Reverse bias under chopped illumination in the presence of $Eu^{3+}$ 0.2 mol/l (inset Fig. 15 left)
Electrochemical reverse saturation current density ( $J_0^{EC}$ )	Open circuit voltage ( $V_{OC}$ )	Forward bias under dark in the presence of $Eu^{2+/3+}$ 0.2 mol/l (inset Fig. 15 right)

**Table 2:** PEC quality control indicators identified for the assessment of different device parameters





**Figure 15:** Left: Short circuit current density and efficiency of the full solid state solar cells (y axis) versus PEC photocurrent density measured in solution (x axis). Right: open circuit voltage (black y axis) and solar cell efficiency (red y axis) of the solid state devices versus reverse saturation current density estimated electrochemically on CIGSe (full dots) and CIGSe (hollow dots) layers in solution (x axis).

An automated PEC mapping tool has been developed comprising (i) a PEC probe prototype with a tip composed of polyacrylamide gel acting as interface between the liquid electrolyte and the solid semiconductor (ii) an x-y-z stage (capable of hosting up to  $30 \times 30 \text{ cm}^2$  samples) supporting the PEC probe and a light emitting diode source (Fig. 16a), (iv) a software interface for intuitive PEC mapping acquisition and data display (Fig. 16b), and (v) an interfaced potentiostat connected to the sample and to the computer for data collection.



**Figure 16:** (a) View of the PEC mapping tool hosting a CIGSe/CdS sample being analysed. (b) Software interface. (c) Results of a PEC mapping test on a CIGSe sample with graded CdS thickness to assess measurement reproducibility: full and hollow black dots correspond to first and second set of measurements, respectively.

The probe has been validated at laboratory scale, using CIGSe/CdS and CIGSSe/CdS large area samples that were subjected to a round robin characterisation experiment involving all techniques developed in the project, and the measurements performed have demonstrated the potential of the probe for the prediction of the short circuit current, open circuit voltage and device efficiency. Further optimisations of this promising new monitoring tool will allow improving the PEC probe reproducibility.

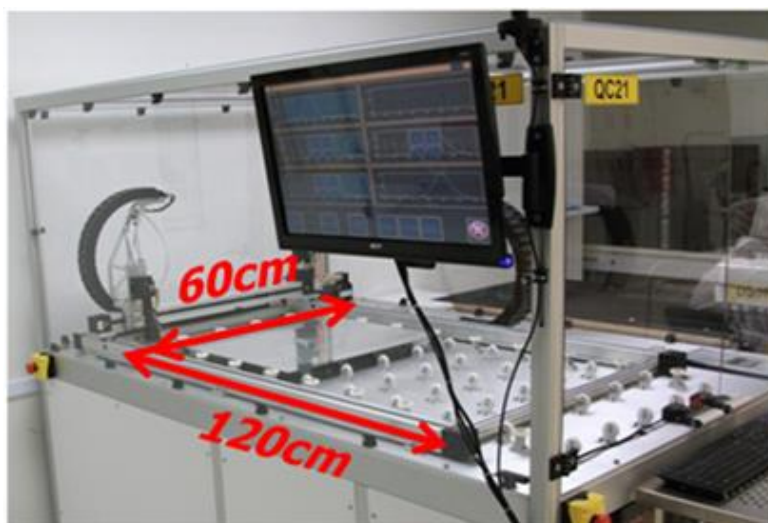
- PL/EL based techniques



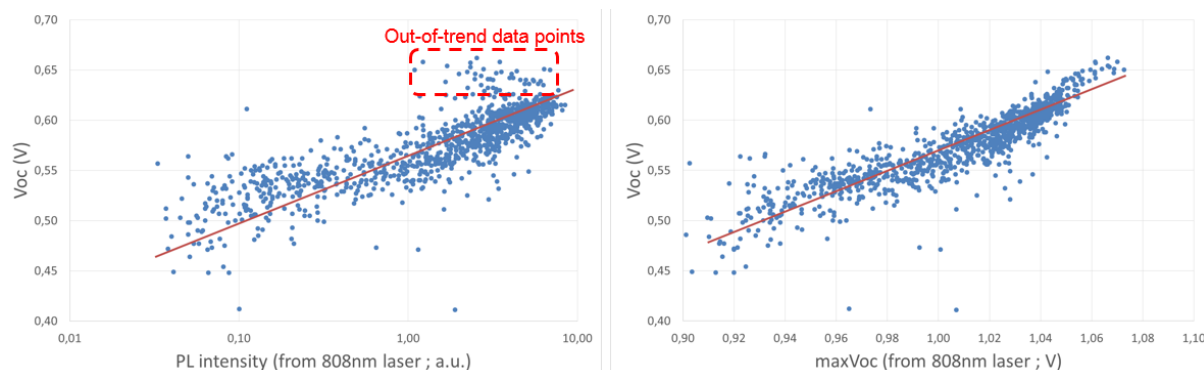
PL/EL based techniques are powerful tools providing direct access to the optoelectronic properties of the absorber layers. Whereas EL is performed using complete devices by injecting current in analogy to the operation of a LED, PL allows the characterisation of bare absorber layers without need for any functional or contacting layers.

In SCALENANO, a combined PL/EL imaging system has been setup and tested by HZB for the assessment of CIGSe and CIGSSe based devices, analysing the correlation of the luminescence yield with the device efficiency and band gap corrected Voc of the solar cells. A special care has been given to the analysis of inhomogeneities in the PL/EL images with the definition of a suitable homogeneity parameter and the development of a model including lateral homogeneities for the homogeneity correction of the PL/EL imaging data. At the final stage of the project, the PL/EL system has been enhanced to incorporate hyperspectral imaging measurement, and the determination of the bandgap distribution and PL efficiency by hyperspectral imaging has been demonstrated on NEXCIS CIGSSe solar cells. These measurements also allow for a very fast spatially resolved prediction of the quasi-Fermi level splitting for the fast evaluation of the open circuit voltage of the devices, being possible the realisation of PL spectral images within times of the order of 5 minutes (much shorter than the times that are required for equivalent PL spectral mapping measurements). This gives a strong interest for the implementation of these techniques as on-line process monitoring tools at the up-scaled pilot line.

PL/EL based techniques have also been implemented as process monitoring tools at the NEXCIS pilot line. A spectral PL mapping system has been installed (shown in Fig. 17), and the system has been tested in real manufacturing conditions on the NEXCIS pilot line. A methodology suitable for the assessment of the quasi-Fermi level splitting and open circuit voltage has been established, following an approach similar to that developed at lab level at HZB (Fig. 18).



**Figure 17:** Spectral PL mapper used at NEXCIS for large area Quality control of the absorbers



**Figure 18:** Correlation between open circuit voltage of  $0.5\text{cm}^2$  test cells manufactured on  $30\times 60\text{cm}^2$  samples and different PL quality indicators: Left:  $A \cdot \ln(\text{IPL}) + B$ ; Right:  $A \cdot [kT(\ln(\text{IPL}) - 3 \ln(\text{PeakPos})) + \text{PeakPos}]$ , where  $A$  is a calibration factor. As shown, the second quality indicator allows for an improved assessment of  $V_{oc}$ .

However, spectral PL mapping has one drawback because only a small portion of the overall area of the module can be characterized during a time compatible with in-line characterization. On this respect, PL imaging remains a more powerful technique. Indeed, one localised defect can strongly impact the performance of a full size module. There is no guaranty that such defect is measured by the spectral PL mapper while it would appear obvious on a PL image. This gives a strong interest to hyperspectral PL imaging which has already been demonstrated by the HZB group at lab scale for the fast quantitative determination of the open circuit voltage of the solar cells. Table 3 summarises the main Quality control indicators that have been identified for the different methodologies that have been implemented and validated for monitoring of the processes developed in SCALENANO:

According to these results, **all the quantitative targets and the Performance Research Indicators that were defined in the project in relation to Key Objective 6 have been fully achieved.** The Performance Research Indicators for Key objective 6 were the following:

- ✓ Identification of quality control indicators for on-line process monitoring (D6.1, Month 18): **achieved, with the identification of the quality indicators summarized in Table 3.**
- ✓ Implementation of in-situ and on-line characterization systems at laboratory scale (MS21, Month 30): **achieved, including the assessment at laboratory scale of the proposed methodologies for in-situ and on-line characterization.**
- ✓ Developed quality control and process monitoring techniques at pilot line level (MS22, Month 42): **achieved, including the development of an up-scaled Raman/PL multiwavelength excitation system for mapping of large area substrates and the implementation of EL/PL based techniques at the pilot line.**



Technique	Indicator	Process/device parameter
ELBS Light elastic scattering	Real time scattered light pattern	Changes during electrodeposition in process parameters (pH, current density, bath concentration species, substrate)
Raman scattering	Relative intensity ZnO defect induced band	AZO conductivity (impact on device efficiency)
	Relative intensity CdS Raman peak	CdS thickness/uniformity (impact on FF, efficiency)
	OVC spectral contribution	$V_{oc}$ , efficiency
	Ratio of intensities of Se-like & S-like Raman peaks	S/(S+Se) content in surface of absorber (impact mainly on $V_{oc}$ and efficiency)
Spectral PL	PL peak energy	Ga/(In+Ga) content in surface of absorber (impact mainly on $V_{oc}$ and efficiency)
	PL intensity & peak position	$V_{oc}$ , Efficiency
PL/EL imaging	Average counts; Spatial distribution of luminescence signal strength; Spatial distribution of luminescence peak position	$V_{oc}$ , $J_{sc}$ , efficiency, minority carrier lifetime * doping density Band gap, distribution and inhomogeneities
DLCP	Net doping density	Efficiency, fill factor
Photoelectrochemical (PEC) measurements	Dark current at different voltages $\rightarrow$ ( $\Delta J_D$ ); Photocurrent/Dark current ratio	Shunt resistance $R_{sh}$ $J_{sc}$ , $E_g$ $V_{oc}$

**Table 3:** Main Quality control indicators identified for the different process monitoring methodologies developed and validated in SCALENANO

- **Key objective 6.1 To validate and develop higher innovative processes and materials for the future generation of chalcogenide based cells and modules: ESAVD**

ESAVD processes have also been analysed as alternative to ED for the synthesis of the absorbers with very high throughput and low cost. ESAVD processes have been successfully implemented for the synthesis of device grade CIGS and CZTS absorbers at small area ( $2 \times 2 \text{ cm}^2$ ) scale, and solar cell prototypes have been developed and characterised. The best device efficiencies achieved with these processes are

- ✓ CIGS devices: efficiency = 10.7%. Even if this value is lower than the efficiency target proposed in this key objective (80% of efficiency of equivalent PVD processes), it is interesting to remark that this value is about 62% of the best efficiency reported for solution based processes (17.3% achieved in SCALENANO for Electrodeposition processes).
- ✓ CZTS devices: Efficiency = 6.35%. This efficiency is about 55% of the world record efficiency achieved in CZTS cells by PVD based processes (coevaporation: 11.6%) and is 60% of best PVD CZTS cell developed in the SCALENANO consortium (10.6%, at IREC).

These data demonstrate the **partial fulfilment of the main quantitative targets and Performance Research Indicators that were defined in the Project in relation to Key Objective 6.1**. The Performance Research Indicators defined for Key objective 2 were:

- ✓ CIGS and CZTS layers by ESAVD method at small scale ( $2 \times 2 \text{ cm}^2$ ) with high uniformity ( $\leq \pm 8\%$  deviation in lateral homogeneity) and no critical secondary phases (D2.3, Month 33):  
**fully achieved, with the synthesis of device grade layers.**





- ✓ Small scale ( $2 \times 2 \text{ cm}^2$  substrate) ESAVD based prototype with 80% of efficiency of best cell based on evaporation material (D2.6, Month 42); ***partially achieved. Efficiency target only achieved at < 50% (CIGS) and 55% (CZTS) in relation to efficiency world records for CZTS printed prototypes (small area) (fulfilment of 60%- 70% of proposed efficiency targets). These are promising results, taking into account the high novelty of the ESAVD based processes.***

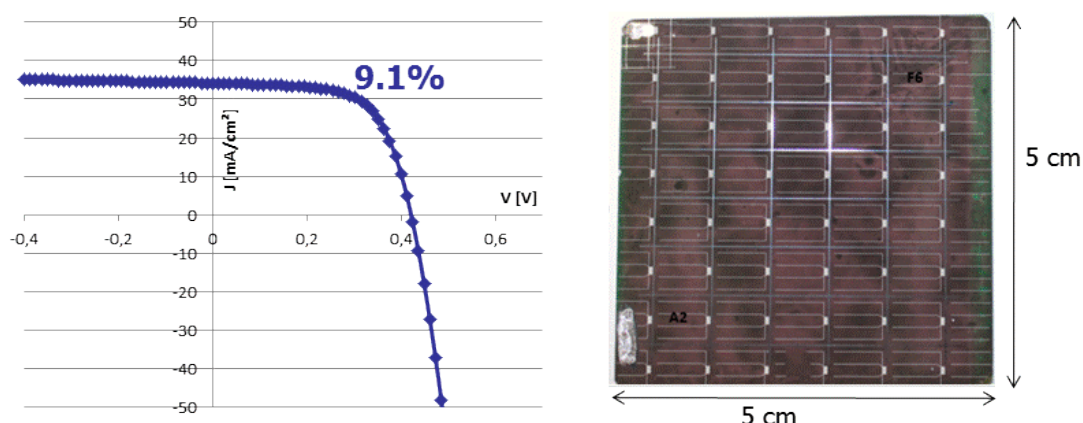
• **Key objective 6.2 To validate and develop higher innovative processes and materials for the future generation of chalcogenide based cells and modules: Extension of ED processes to kesterites**

Extension of these processes to kesterite based solar cells has led to a significant advance in the understanding of the mechanisms of formation of the kesterite. In this case, the developed processes have allowed achieving numerous solar cell efficiencies beyond 8%, including a very promising efficiency of 9.1%, which constitutes a new world record efficiency for ED kesterite solar cells.

The analysis of the processes developed for the synthesis of the kesterite absorbers has allowed identifying the optimal strategy to achieve an improvement of the device efficiency. This involves:

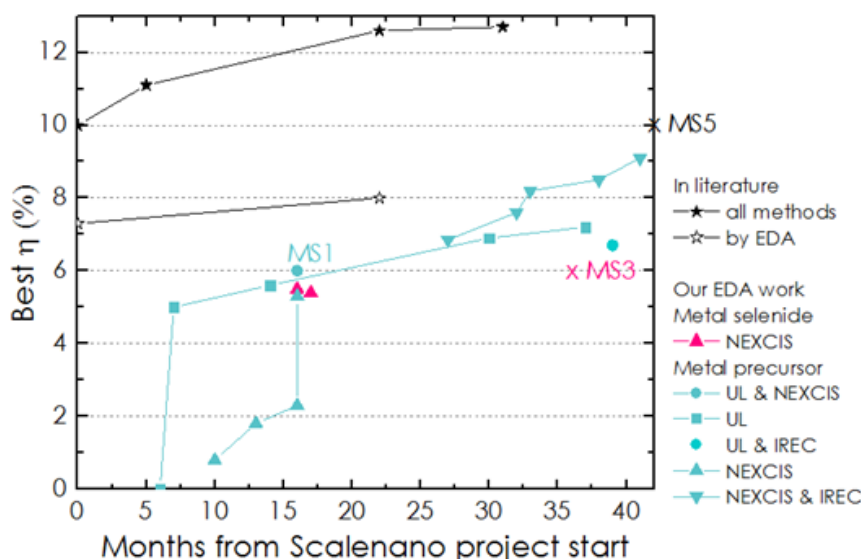
- ✓ Use of metal precursors (instead of metal selenide);
- ✓ Selection of Zn-rich / Cu-poor absorber compositions limiting the amount of secondary phases (ZnSe, Sn-Se) and promoting their segregation at surface region (corresponding to compositions ratios  $\text{Cu}/(\text{Zn}+\text{Sn}) = 0.73 - 0.86$ ;  $\text{Zn}/\text{Zn} = 1.02-1.20$ );
- ✓ Removal of secondary phases using selective removal processes developed at IREC
- ✓ Optimisation of CdS CBD process: replacement of  $\text{CdSO}_4$  by  $\text{Cd}(\text{NO}_3)_2$  as Cd precursor allows slower CdS chemical bath deposition and better control of CdS thickness, allowing the synthesis of thinner CdS layers leading to higher short circuit current.

Figure 19 shows the I-V characteristics and a photographic image of the cells with highest efficiency developed in the project. These devices have been fabricated on  $15 \times 15 \text{ cm}^2$  Cu/Sn/Zn electrodeposited precursors and include a deposited grid for collection of the current. These devices do not have antireflective coating.



**Figure 19:** I-V curve (left) and photographic image (right) of best efficiency CZTSe cells (9.1%) from electrodeposited precursors with a deposited grid and without antireflective coating.

Figure 20 shows the evolution of the efficiency of the ED kesterite devices since the beginning of SCALENANO and their comparison with the best efficiency devices reported in the literature. As shown in this figure, since month 33 the devices developed in SCALENANO have efficiencies higher than the highest ones reported for the kesterite ED cells.



**Figure 20:** Evolution of kesterite devices efficiency since the beginning of the SCALENANO project. In black are represented the best device efficiencies reported in literature for CZT(S,Se) devices (full stars) independently of the deposition method and (open stars) by electrodeposition. In pink and blue are represented the best devices efficiencies obtained in SCALENANO for kesterite absorbers made by metal selenide precursor route (pink) and metal precursor route (blue), respectively. Target efficiency values proposed in Milestones MS1, MS3 and MS5 are also represented in the graph.

These data demonstrate the **partial fulfilment of the main quantitative targets and Performance Research Indicators that were defined in the Project in relation to Key Objective 6.2.** The Performance Research Indicators defined for this key objective were:

- ✓ 6% efficient kesterite device from metal precursor on 0,5 cm<sup>2</sup> area (MS1, Month18):  
**fully Achieved**
- ✓ 6% efficient kesterite device from metal selenide precursor on 0.5 cm<sup>2</sup> area (MS3, Month 36):  
**5.4% achieved (best pixel) on metal selenide precursor & 8.2% achieved (best pixel) on metal precursor: this lead to the decision to focus only on metal precursors for the last period on the project**
- ✓ Kesterite based ED cell with efficiency 10% from optimal precursor and sulphur/selenium composition (D 1.7, Month 42):  
**Achieved with an efficiency of 9.1% (without ARC) (world efficiency record for ED kesterite cells) and with the identification of the routes required to improve the device efficiency to the 10% target.**

• **Demonstration of up-scaling of the processes up to module level and validation at pilot line level**

In addition to the Key Objectives 1 to 6 described in section 2 of the Final Report, SCALENANO has developed a significant activity in the demonstration of the scalability of the processes for the industrial manufacturing of large area PV modules. The main results achieved in these activities include:

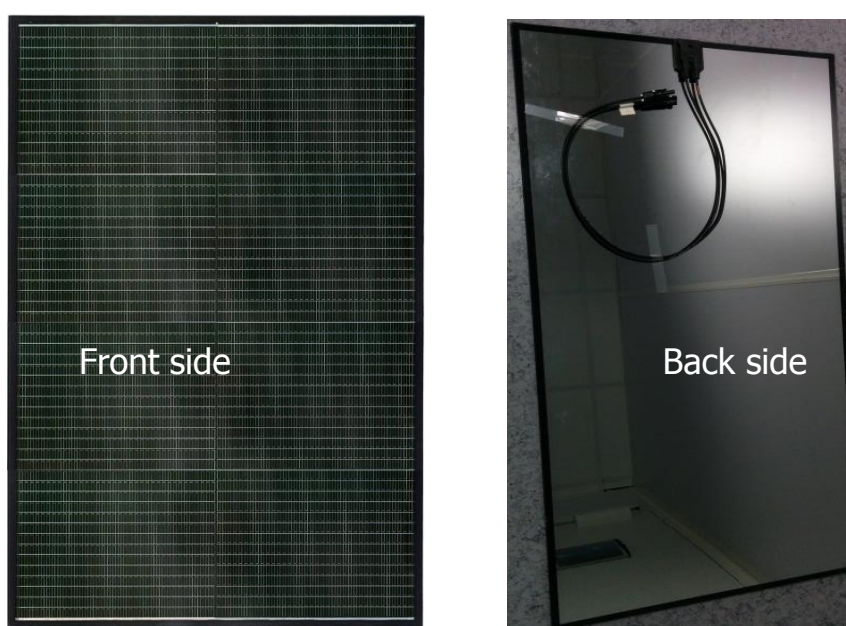
• **Development of module prototypes**

The transfer of the knowledge developed in the project from a cell level to a module level has been successfully performed. In the first period of the project, a design of large area modules consistent with the



optimisation of the output power of the modules was developed, including the definition of proper patterning processes for the interconnection of the cells.

Activities performed have included the development and manufacturing of medium area ( $30 \times 60 \text{ cm}^2$ ) and large area ( $60 \times 120 \text{ cm}^2$ ) modules with ED based absorbers, achieving the module efficiency values already indicated in Table 1. Since month 19, activities have also been developed on the lamination/encapsulation process of CIGS modules and the life-time testing, mainly as a quality feedback to the encapsulation process. At the end of the process, lamination and encapsulation studies have allowed determining the best encapsulation material exhibiting longest resistance to moisture ingress. In parallel, life-time testing has also included the full pre-qualification of the NEXCIS  $60 \times 120 \text{ cm}^2$  modules according to IEC 61646, and the development of a proper electrical characterisation of the modules has allowed drafting proper pre-conditioning procedures in order to perform a correct power estimation (Wp) of the modules.



**Figure 21:** Front side and back side of the  $60 \times 120 \text{ cm}^2$  large-area NEXCIS module prototype developed at SCALENANO.

In addition, the scalability of alternative solution based processes developed in SCALENANO –as CDB based processes for growth of AZO layers and printing based processes for synthesis of kesterite absorbers - has also been studied, obtaining first mini-module prototypes integrating these processes. This has included the fabrication of  $10 \times 10 \text{ cm}^2$  CBD AZO mini-modules and  $5 \times 5 \text{ cm}^2$  solution-based kesterite minimodules. In the last case a promising module efficiency of 5.5% -comparable to that obtained using vacuum-based processes- has been achieved. The results obtained have allowed demonstrating the viability of these processes for their upscaling into large area modules, although more work is required for their further development and optimisation.

- Demonstration of industrial viability of developed processes

The demonstration activities developed in the project have included the detailed analysis of the industrial viability of the most mature developed process which is the electrodeposition based CIGS modules produced on NEXCIS pilot line. The module structure has been defined, and the results for electrodeposition-based modules are in line with the expected cost reduction and the 14.0% ApE record module and 13.3% ApE on average achieved the target of obtaining 95% of the efficiency of PVD-based modules that was established in the project.





Other non-vacuum processes such as the CIGS by ESAVD, CZTS by chemical bath deposition as well as the vacuum free TCO showed promising results at small scale and up to 15x15cm<sup>2</sup> with efficiencies in the 10 to 13% range but did not reach sufficient maturity yet to be installed on the pilot line for manufacturing full 30x60cm<sup>2</sup> modules. Further work is necessary to reach a TRL of 5-6 for these processes but no major technological obstacle is foreseen. The cost analysis performed in the project confirms that there is a cost incentive to use vacuum free processes and abundant materials. However with an estimated price as low as 44.5c€/Wp for CIGS modules, one must keep in mind that the cost cannot be easily reduced much further and that each process improvement cannot pull the cost down by more than a few euro cent at the most.

According to these results, **all the quantitative targets and the Performance Research Indicators that were defined in the project in relation to the demonstration of the scalability of the processes for the industrial manufacturing of large area PV modules have been fully achieved.** The Performance Research Indicators for these activities were the following:

- ✓ 30x60 cm<sup>2</sup> medium-area module with ED absorbers (NEXCIS) (laboratory test device) (MS17, Month 18):  
**achieved at month 18 with a best module efficiency of 13.1% and an average module efficiency of 11.7%.**
- ✓ 60x120 cm<sup>2</sup> large-area module with ED absorbers (NEXCIS) (laboratory test device) (MS19, Month 30):  
**achieved since month 24, with a best module efficiency of 13.2% and an average module efficiency of 12.4% at month 30.**
- ✓ 10x10 cm<sup>2</sup> minimodule with CBD TCO (laboratory test device) (D5.9, Month 42):  
**achieved. Minimodules produced at the final stage of the project have a cell efficiency of 3.5% for a 12.44 cm<sup>2</sup> cell and a module efficiency of 2.6%. These results demonstrate that a working PV module can be fabricated with a TCO CBD process, even if further work is necessary to improve the efficiencies at module level.**
- ✓ Electrical performance of manufactured devices and validated quantitative performance targets: efficiency higher than 95% of equivalent reference PVD modules with costs 30% lower than PVD processes (D5.8, Month 42);  
**achieved. Validation of the quantitative performance targets corroborates the fulfillment of the efficiency and costs targets that were defined in the project. This includes the economic analysis of the processes that has been performed according to the costs model developed by NEXCIS.**
- ✓ Sensibility analysis of the main factors affecting the industrial take up (D7.1, Month 32):  
**achieved. The sensibility analysis was based on the results obtained from the pre-industrial line installed at NEXCIS and producing 60x120cm<sup>2</sup> modules on glass substrates**
- ✓ Report on the design, implementation and cost analysis of a 500 MW plant (MS25, Month 42):  
**achieved. The industrial production line has been designed using multiple smaller volume production units of 100 MW.**
- ✓ 60x120 cm<sup>2</sup> module prototype with optimal encapsulating and anti-reflective coating configuration (D7.5, Month 42)  
**achieved. The PV module structure with EVA encapsulation has been proved to be the most satisfying one based on the studies performed at SUPSI.**



#### 1.4 Potential Impact

The main impact of SCALENANO is related to the contribution of the project to the improvement of the competitiveness of thin film CIGS PV technologies, with the demonstration of the industrial transferability of electrodeposition based processes for the fabrication of large area PV modules with efficiencies comparable to those from commercially available modules that are produced with higher cost vacuum-based processes.

Improvement of the competitiveness of PV technologies in Europe will allow consolidating an industrial strategic sector with significant technology added value, with a potential impact in the creation of new jobs and in the further development of industrial niche activities with potential market growth as those related to building and residential PV applications. This will also contribute to achieve a significant increase in the share of electricity produced by PV in the next years. Replacement of conventional energy sources by alternative cost-efficient renewable technologies with very low environmental impact is critical to achieve a significant reduction in greenhouse emissions, mitigating the impact of these emissions on the climatic change and reducing the dependence of Europe on energy sources, therefore contributing to transition of the society towards a sustainable socio-economical model based in the use of low carbon technologies.

The following table summarises the main contributions of SCALENANO on the expected impacts that were listed in the work programme corresponding to the ENERGY.2011.2.1-2 Call of the project:

Expected Impact	Main contributions of the project
Solutions going well beyond the state-of-the-art in terms of cost (target of far below 1 €/Wp) and efficiency	<ul style="list-style-type: none"> <li>• Development of a reliable low cost (40% lower than PVD processes for the fabrication of the CIGS absorbers) and efficient (&gt;95% than PVD processes) electrodeposition (ED) based technology with better lateral homogeneity (<math>\pm 5\%/m^2</math>) on large area substrates.</li> <li>• New cell architecture configurations for higher efficiency devices based on the use of nanostructured TCO layers and compatible with low cost processes have also been developed, identifying the most optimal architecture configurations and determining the technological challenges required for their implementation.</li> </ul>



Stimulation and acceleration of the industrial take-up of promising results beyond laboratory scale	<ul style="list-style-type: none"> <li>• SCALENANO has demonstrated the industrial transferability of a CIGS ED pilot line for the fabrication of large area (60x120 cm<sup>2</sup>) modules with the fulfilment of the uniformity, efficiency and cost criteria that were defined in the project</li> <li>• Analysis of the implementation of vacuum-free processes developed in SCALENANO (including alternative printing based processes for synthesis of the absorbers and vacuum-free processes for growth of TCO layers) in the pre-industrial pilot line available at NEXCIS</li> <li>• Design and simulation of an economically viable 500 MW industrial pilot plant</li> </ul>
New competitive industrial processes.	<ul style="list-style-type: none"> <li>• Development (at cell and mini-module level) of novel vacuum free cost efficient processes with very high throughput and process rate potential, based on printing of new ink formulations and ESAVD, that have been demonstrated at cell and mini-module level</li> <li>• Extension of these processes to technologies based on new kesterite absorbers that are proposed as the mid-long term replacement of CIGS for the future deployment of PV technologies to Terawatt levels, with the achievement of device prototypes with efficiencies already comparable to the current world record.</li> </ul>

**Table 4:** Main contributions of SCALENANO on the expected impacts that were listed in the work programme

From the exploitation point of view, the activities developed in SCALENANO have a potential impact in a wide range of industrial and relevant socio-economic fields, as described in the report of deliverable D8.5 (Final report on exploitation prospects, Month 42). These include among others the following ones:

- Photovoltaics: development of a low cost electrodeposition based PV CIGS technology for fabrication of large area PV modules (PV market sector). The flexibility of the processes allows also the development of semi-transparent glass based modules with potential applications for Building Integrated PV (BIPV), a market which has a strong potential growth in the next years. First demonstrators of semi-transparent PV modules for replacement of glass based façade elements have already been produced at NEXCIS adapting the processes developed in SCALENANO.
- Photovoltaics: development of vacuum-free cost-efficient kesterite technologies with potential reduction of costs. Cost analysis performed within the cost model developed in SCALENANO has corroborated the cost reduction potential of kesterites in relation to CIGS, provided that kesterites can reach 14% module efficiency. Technical barriers to overcome for the future transition to pre-industrial and industrial levels have been identified.



- Sensor devices and systems: Development of scalable procedures for the synthesis of chalcogenide nanocrystals with tuneable composition and crystalline structure, with relevant applications as active materials for very fast photodetectors and non-toxic fluorescent labelling probes.
- Deposition/coating processes: ESAVD scalable based processes for low cost low temperature growth of TCO layers with high optical transmission and electrical conductivity with potential industrial applications in PV and Electronic industries (thin film PV modules, OLEDs, touch screen and liquid crystal displays).
- Deposition/coating processes: Development of solution-based CBD ZnO coating processes. Low cost scalable processes requiring very low process temperatures have been demonstrated for the growth of ZnO layers, including TCO functional layers with potential industrial applications in different sectors including: PV, electronic displays and sensors industries (TCOs), Wood industries (UV-protective super-hydrophobic surfaces).
- Solid state devices: Development of electrodeposited based ZnO nanorod (NR) structures with tuneable morphology and controlled optical properties with potential industrial applications including PV (NR antireflective coatings, new cell architectures for bifacial CIGS solar cells) and Solar fuels (ZnO NR based electrodes for photo-assisted water oxidation).
- Photovoltaics: Development of hybrid PV module architectures with grid printing and laser patterning alternative to standard P1/P2/P2 monolithic integration. Higher flexibility of hybrid PV modularisation concepts facilitates design and production of customised modules for specific applications, as BIPV semi-transparent modules with different transparency and specific designs.
- Photovoltaics: Lamination/encapsulation processes. Analysis of different encapsulation materials and processes has allowed assessing optimal processes with highest potential for extended-life time of NEXCIS modules using edge sealants and polymers, allowing an extended resistance to moisture ingress that is critical for the installation of PV modules in many environments (tropical, marine...).
- Process assessment: Development and demonstration (at lab level) of a new real time light scattering methodology for the in-line assessment of the surface of the metallic precursors during their electrochemical growth. This methodology can be easily adapted to any kind of chemical based deposition or chemical/electrochemical based surface treatment, provided that the solution is transparent to the excitation light. This has applications in different fields related to Photovoltaics and, in a more general way, to Plating and Galvanostatic industries as those used in Automotive, Sensors, Electronics and Aeronautics fields.
- Metrology: Development and demonstration (at lab and pilot line levels) of a multi-excitation wavelength Raman scattering methodology for selective assessment of multi-layered complex structures and devices. This new metrology tool has been demonstrated for the monitoring of the different layers in the CIGS solar cells and modules. Adaptation to other system structures will allow extension of this methodology for advanced metrology applications in fields related to Semiconductors and Solid State devices industries, as well as optical industry.
- Metrology: Development and demonstration (at lab and pilot line levels) of Photoluminescence imaging systems for the assessment of the optoelectronic properties of large area CIGS absorbers, allowing the fast in-line prediction of the band gap uniformity and open circuit voltage of the PV modules. This has included a new methodology for hyperspectral PL-imaging that could greatly improve the prediction capacity of the open circuit voltage of CIGS cells right after the junction formation. The path for commercialisation of these systems involves aspects depending on the specific production line to be assessed, as the definition of the measurement area and time-interval and the design of in-line measurement apparatus to ensure laser safety, as well as a robust programming for the extraction of



the optoelectronic parameters, for which suitable algorithms have already been developed in SCALENANO.

- Metrology: Development and demonstration (at lab level) of a new Photoelectrochemical (PEC) methodology and tool for the prediction of the optoelectronic parameters of solar cell devices from non-destructive PEC measurements performed directly on the surface of the absorber layer or the buffer layer (buffer/absorber hetero-structures). The correlation between the PEC measurements and the I-V parameters of solar cells has been demonstrated in SCALENANO, and problems related to the implementation of a reproducible PEC gel probe need to be solved for the further development and exploitation of this methodology

### **Main Dissemination activities**

SCALENANO has given a special emphasis to the development of an efficient dissemination strategy that has been performed according to the Plan for the Use and Dissemination of Foreground described in Deliverable D8.1 (month 2) and its successive updates at month 18 (Deliverable D8.3) and at month 36 (Deliverable 8.4): The Plan has included the following actions:

#### **1) Press campaigns:**

Two press campaigns have been done at the initial (months 1-4) and final (month 42) stages of the project. These campaigns were based in the distribution of press notes that were translated and adapted by each of the partners to the local language of the immediate community each organisation is situated. The press releases were disseminated through the respective “news” section of the webs of the different institutions.

In the first campaign, the press note was also distributed through national news agencies (EFE, Spain). As result of this distribution, press releases were published in 45 media from different countries (mainly but not exclusively from Spain, Luxembourg, Germany, England), including newspapers and magazines with very high public impact as “La Vanguardia” (3<sup>rd</sup> more important newspaper in Spain) or Luxembourger Wort (main newspaper at Luxembourg).

At the end of the project, a news report (based in an extended version of the press note) has been published in the following media:

- La Vanguardia (issue Saturday 25<sup>th</sup> July 2015) : La Vanguardia constitutes one of the main and most influential newspapers in Spain, with an estimated audience that is close to 2,000.000.- readers
- Cinco Días (issue Monday 27<sup>th</sup> July 2015): this is a weekly Spanish journal mainly devoted to Economy news, with a relevant dissemination among company managers and industrial relevant sectors in Spain.

#### **2) Project flyer:**

A project flyer was designed and produced at month 2, describing the main objectives and motivation of SCALENANO and indicating the partners involved and contact data. The project flyer was included in the web of the project and diffusion of the flyer has been made at different International Conferences and events where members of SCALENANO consortium have taken part:

- 2012 & 2013 MRS Spring Meetings (San Francisco, CA, USA, April 2012, April 2013)
- 2012 & 2013 E-MRS Spring Meetings (Strasbourg, France May 2012, May 2013)
- 2013 E-MRS Fall Meeting (Warsaw, Poland, September 2013)
- 38<sup>th</sup> & 39<sup>th</sup> IEEE Photovoltaics Specialists Conferences (Austin, TX, USA, June 2012, Tampa, FL, USA, June 2013)
- 28<sup>th</sup> EU PVSEC (Paris, France, September 2013)
- Austrian Chemistry Days, Austrian Chemical Society (Graz, Austria, 23-26 September 2013)
- SUPSI's research days (SUPSI, Canobbio, Switzerland, 5-6 September 2012)
- Solar Energy UK 2014 Show, Birmingham, UK, October 2014
- Barcelona Global Energy Challenges Conferences (Barcelona, Spain, June 2012, June 2013)
- Destaca 2014: Feria Científica, Tecnológica y de Innovación (Villareal, Spain, November 2014)





At the final stage of the project (month 42) an updated version of the project flyer was produced, emphasising the main achievements of the project. The flyer has also been uploaded at the web site of the project, and has been distributed through the project partners for further dissemination. The flyer is being distributed at different events in the period from September 2015 to December 2015, including the following ones:

- EUPVSEC 2015: 31st European Photovoltaic Solar Energy Conference and Exhibition (Hamburg, Germany, 14-18 September 2015)
- 50º Congreso Mexicano de Química (Queretaro, Mexico, 7- 10 Octubre 2015)
- International Conference on Solution Processed Innovative Solar Cells SPINS 2015 (Santiago de Compostela, Spain, 9-11 September 2015)
- 2015 MRS Fall Meeting and Exhibition (Boston, MA, USA, November 29 - December 4 2015)

### 3) Further dissemination at outreach level

Interviews to project coordinator: three interviews have been published in the following media

- Journal Solar News (issue 40, 2012) ([www.solarnews.es](http://www.solarnews.es))
- PV Magazine ("A Giant Leap Towards the Future", A. Williams, 09/2012 issue, pages 172-175). This included also an interview to the coordinator of the Technology Task Force ([www.pv-magazinew.com](http://www.pv-magazinew.com))
- Pan European Networks Science & Technology Journal, March 2013 issue, [www.paneuropeannetworks.com/science](http://www.paneuropeannetworks.com/science)), answering to an Editorial Invitation to contribute to this publication. In this interview, first results achieved in SCALENANO were already presented, with contributions from A. Pérez-Rodríguez, J. López-García and V- Bermúdez

Publication of outreach papers describing SCALENANO:

- "Material Matters for Photovoltaics", M. Freebody, Europhotonics Magazine, December 2012, <http://www.photonics.com>)
- "Better, more versatile silicon-free solar cell technologies", Horizon, The EU research & Innovation Magazine, 16<sup>th</sup> October 2013, [http://horizon-magazine.eu/article/better-more-versatile-silicon-free-solar-cell-technologies\\_en.html](http://horizon-magazine.eu/article/better-more-versatile-silicon-free-solar-cell-technologies_en.html)
- "Development a cheaper alternative solar cell for Europe", Cordis News Section, 1<sup>st</sup> Nov. 2013, [http://cordis.europa.eu/fetch?CALLER=EN\\_NEWS&ACTION=D&SESSION=&RCN=36210](http://cordis.europa.eu/fetch?CALLER=EN_NEWS&ACTION=D&SESSION=&RCN=36210),

### 4) Project website

The website of the project ([www.scalenano.eu](http://www.scalenano.eu)) is operative since month 3 of the project. The web has received more than 21,645 visits until September 2015. The homepage of the web includes different sections that are regularly updated:

- Project overview and summary of relevant information (Project full title, Project code, consortium members, timeframe and acknowledgment to the support of the Commission);
- Project description: overall objectives and those specific of each WP are clearly indicated;
- Project partners: a short description of the partners is provided, including an explanation of their task in the Project as well as their key "Experience & Competences" relevant to the activity. The website address of each Partner is shown, in which more information about them is available;
- Job Offers: section in which calls for research positions involved in the project were progressively announced;
- List of Publications and conferences (including link to the published papers and the abstracts of the contributions to International conferences);
- Project flyers;
- Newsletters;
- Press releases and clipping, including all the releases that have been published in the different media;
- Contact details.

The website also includes a direct link to the CORDIS and Euraxess websites. Finally, the website allows log in the project intranet that is only accessible for the Partners, PO and PTA. There, confidential information in the frame of SCALENANO activity is available for authorized users. The structure of the intranet includes the following sections:



- Project Information, with copies of the Grant Agreement, DOW and CA;
- Summary: with the summary of deliverables and milestones and their time schedule;
- Partner Details;
- Meeting Minutes;
- Reports and Deliverables;
- Reprints of published papers for internal consortium use.

Access to these sections is subjected to the role of the different partners in the project. In principle, only the coordinator has access to all sections, with the possibility to upload/remove documents.

#### 5) Organisation of Workshops:

- **EU PV Clusters 2nd Workshop and General Assembly “Progress in Photovoltaics and Nanotechnology: from FP7 to Horizon 2020” (Barcelona, Spain, 26<sup>th</sup>-28<sup>th</sup> November 2013)**

SCALENANO hosted the EU PV Clusters 2<sup>nd</sup> workshop and General Assembly “Progress in Photovoltaics and Nanotechnology: from FP7 to Horizon 2020”. The workshop was held in the premises of the University of Barcelona (Spain) from 26<sup>th</sup> to 28<sup>th</sup> November 2013. The workshop gathered 80 participants from 16 different countries who are involved in more than 40 projects from different programmes funded by the European Commission. Most of the on-going projects involved in the EU PV Clusters ([www.eupvclusters.eu](http://www.eupvclusters.eu)) participated in the workshop. This event gave the opportunity to both representative of these projects and members of several European Initiatives to come together during three full days to build up constructive interactions for the present and the future. The workshop allowed reviewing the European key performance achievements in PV technologies, as well as to identify strategic common research priorities and to disseminate relevant information on several European stakeholders and associations. The workshop finished with the 1<sup>st</sup> SCALENANO workshop that was opened to all participants, giving the opportunity to disseminate the main results and achievements of the project to the European PV research community.

- **EU PV Clusters & SCALENANO Workshop “Advanced Materials & Nanotechnology for Energy: KETs challenges in Photovoltaics (II)” (CEA, Caradache, France, 17<sup>th</sup>-18<sup>th</sup> September 2014),**

The EU PV Clusters and 2<sup>nd</sup> Scalenario Workshop was held on September 17<sup>th</sup>-18<sup>th</sup> 2014 in Cadarache (France), with the participation of the coordinators of different EU PV Clusters and members from several European Initiatives as NANO futures, Solarrok, and EMIRI, together with representatives from the SCALENANO consortium (IREC, CEA, UCL and NEXCIS). The main objective of the workshop was to align the roadmap and priorities of EU PV Clusters, NANO futures, Solarrok, and EMIRI in the PV field, following the main conclusions of the previous workshop on KETs challenges in Photovoltaics that was held during the Industrial Technologies 2014 Conference in Athens (Greece) in April 2014. These workshops were celebrated to answer the needs identified during the EU PV Clusters 2<sup>nd</sup> Workshop to thus align both the key issues and bottlenecks listed by the different EU PV Clusters and the existing European roadmaps. The workshop finished with a SCALENANO session and a technical visit to the NEXCIS pilot line at Rousset (France), which included a detailed overview provided to the workshop participants of the objectives and approach of the project, as well as the most relevant achievements at month 32.

- **3<sup>rd</sup> SCALENANO Workshop at the NEXTGEN NANO PV 2015 International School and Workshop (Mao, Menorca, Spain, 24<sup>th</sup> April 2015).**

The 3<sup>rd</sup> SCALENANO workshop was hosted by the “NEXTGEN NANO PV 2015: Nanotechnology for Next Generation High Efficiency Photovoltaics Spring International School and Workshop” that was held in Mao (Menorca, Balearic Islands, Spain) on 20<sup>th</sup>-24<sup>th</sup> April 2015 ([www.nextgennanopv2015.com](http://www.nextgennanopv2015.com)).

The NEXTGEN NANO PV 2015 Workshop gathered 90 participants from 14 countries with a significant participation of young researchers, and was organised following the previous successful edition that was held in Cargèse (Corsica, France) in April 1- 6 2013. The main objective of these workshops was to provide a suitable forum for reviewing and discussion of the state of the art and existing challenges related to the implementation of new nanotechnology based concepts for solving the main problems in the development of cost efficient and





sustainable PV. The workshop included also an International School that constituted a suitable platform for an efficient sharing of experience and visions between well-known experts from different academic, research and industry groups and young researchers and PhD students. Inclusion of the 3<sup>rd</sup> SCALENANO Workshop in this broader event allowed a more relevant diffusion of the main achievements of the project to relevant actors from the PV world R+D community.

#### 6) Dissemination at International Conferences and Workshops

As part of the standard scientific dissemination channels, a very relevant activity has been developed in dissemination actions at the main International Scientific Conferences and Workshops. During the whole duration of the project, 88 contributions were presented, including 16 invited contributions. All of them duly acknowledged European Commission for the support through the SCALENANO project.

The attended events includes the main scientific international conferences on materials and processes for advanced PV devices and PV technologies, as the Electrochemical Society Meetings, the PV related symposia from the MRS and the European MRS Spring Meetings, the IEEE Photovoltaics Specialists Conference (which is organised yearly in the USA and constitutes one of the main and bigger International Conferences in the field of PV technologies), the European PV Solar Energy Conference and Exhibition (which constitutes the largest European Conference in the field of Photovoltaics), and the European Kesterite Workshops (which constitute the main Workshops organised in Europe specifically devoted to emerging kesterite PV technologies), among others.

#### 7) Publications in Scientific Journals and Books

Scientific publications of SCALENANO include 32 papers that have been published in Scientific Journals and 5 papers that have been published in Books and Proceedings Series. All the papers acknowledge the European Commission for the support through SCALENANO.

These papers have been published in leading scientific international Journals with high Impact factor (Advanced Functional Materials, Progress in Photovoltaics, Solar Energy Materials & Solar Cells, ACS Applied Materials & Interfaces, Journal of the American Chemical Society, Applied Physics Letters, Chemistry of Materials, Solar Energy, Journal of Alloys and Compounds...) and in well recognised Journals with a strong tradition in the fields related to thin film PV technologies and chalcogenide based materials (Thin Solid Films, Physica Status Solidi a).

It is interesting to remark the relevant SCALENANO contribution that was made at the 2<sup>nd</sup> EU PV Clusters Workshop special issue of Physica Status Solidi (a): Advanced Materials and Nanotechnology for Photovoltaics, with the publication of 11 papers, being two of them selected for both the front cover page and the back cover page.

In addition, 4 papers were published in relevant Proceedings Series from the PV materials and technologies fields (as the Mater. Res. Soc. Symp. Proceedings Series, the IEEE Photovoltaics Specialists Conference Proceedings and the Electrochemical Society Proceedings), and a Chapter contribution has been made at the book "Advanced Concepts in Photovoltaics" published by the Royal Society of Chemistry (London, UK, 2014).

#### **Main Exploitation activities**

Exploitation activities in SCALENANO have been performed with the aim to identify and promote exploitation of the results of the project, either by the industrial partners in the consortium or by third part companies through suitable IPR licensing agreements. The task has been coordinated by the Technology Task Force (TTF) who was chaired by NEXCIS and included the WP leaders and representatives of all the industrial partners in the consortium.

During the whole period, the TTF leader has been in strong link with all the partners gathering the advances in new and innovative concepts that are coming out in the frame of the project to assure the optimal way to transfer them to commercial available concepts. The main activities performed included:



- The identification of main processes developed in SCALENANO alternative to ED that have been included in the analysis of the industrial transferability to the NEXCIS pilot line (in cooperation with WP7). Main processes identified have included the vacuum-free growth of TCO layers and the printing based processes of kesterite absorbers, and the results of this analysis are described in the reports of deliverables D7.3 and D7.4 from WP7 (submitted in Month 42);
- The identification of opportunities for exploitation of processes, concepts and methodologies which are outputs of the project in PV and/or other fields. On the whole, 22 possible exploitable results (described in deliverable D8.5: Final report on exploitation prospects) with different TRLs have been identified, that are distributed over the different WPs and activities that have been developed in the project, mainly from the fields of (i) Technological Processes, (ii) Metrology and (iii) Novel Applications, as described in the report of deliverable.

Table 5 shows the main examples of exploitable results that have been identified in the project. This includes new approaches and methodologies that have been validated in the project, identifying in all cases the additional actions that are required to reach the market.

Related WP	Exploitable result
WP1	Manufacturing of 60x120cm <sup>2</sup> ED-based CIGS modules
	Kesterite by ED
WP2	CIZS crystals with very high luminescence
WP3	ESAVD equipment and process for thin films deposition
	Solution-based CZTS for PV
	Solution process for ZnO coating
WP4	Electrodeposited ZnO Nanorods
	New architectures for PV cells
WP5	Modules architecture with Grid printing and laser patterning
	Lamination / Encapsulation processes
	Qualification of PV product and outdoor monitoring
WP6	In-line process monitoring of ED layers growth
	PL-Raman multi excitation CIGS/CdS/TCO monitoring tool
	Coupled PL & EL-imaging / Hyperspectral PL imaging
	PEC tool & methodology
General	Chalcogenide-based photocathodes for solar water splitting

**Table 5.** Main exploitable results identified in the different WP's of SCALENANO

From these results, the most mature ones have achieved a TRL of 6-7, and include the processes for the fabrication of CIGS modules (both full opaque or semi-transparent) and the development and validation of improved metrology tools for CIGS. Additional support (beyond the scope of SCALENANO) is required for reach a TRL of 8-9, for the completion of the transition of these processes and tools to industrial exploitation.



## **SECTION 2- USE AND DISSEMINATION OF FOREGROUND**

The data of Section 2 have been reported through the EC Participant Portal. The corresponding information is presented in the report which is automatically generated by the system.

### **2.1 Section A**

Additional details about the final list of all scientific (peer reviewed) publications relating to the foreground of the project (Table A1) are given in Deliverable 8.6. The same applies to the list of Dissemination Activities (Table A2).

### **2.2 Section B**

Additional details about the exploitable foreground and the plans for the future (Tables A1 & A2) are given in Deliverable 8.5. It should be pointed out that any trademark, registered design and utility model have been obtained. As far as patents are concerned, there is one application of EMPA which still does not appear in the databases. Details are given in the corresponding deliverable.

## **SECTION 3- REPORT ON SOCIAL IMPLICATIONS**

The questions and statistics of the project have been provided by the Coordinator on behalf of the entire Consortium via the EC Participant Portal. The corresponding information is presented in the report which is automatically generated by the system.

