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CIGS multistage in-line pilot Machine demonstration

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Zusammenfassung

In der Abteilung für Dünnfilme und Photovoltaik an der Empa ist es gelungen durch innovative Methoden einen Prozess zur Herstellung hocheffizienter CIGS Solarzellen auf Kunststofffolien zu entwickeln. Leichte und biegbare Solarzellen mit Wirkungsgrad über 20% konnten so im Labormassstab hergestellt werden. Auf dem Weg zur Lösung der Energieproblematik ist es wichtig zu demonstrieren, dass dieser noch stationäre Prozess auch auf eine produktionsrelevante Beschichtungsanlage transferiert werden kann.

Das Ziel dieses P&D Projekts war es eine rolle-zu-rolle (R2R) CIGS Beschichtungsanlage zu demonstrieren, mithilfe derer man diesen Transfer des innovativen Prozesses vom Labor in eine vorindustrielle Produktion zeigen kann. Innerhalb des Projekts ist es gelungen diese Anlage fertigzustellen und in Betrieb zu nehmen. Die Demonstration einer solchen Pilotanlage spielt eine zentrale Rolle bei der industriellen Entwicklung von CIGS Beschichtungsanlagen für hoch effiziente und kostengünstige Solarmodule und bei der Bereitstellung von bezahlbarem Sonnenstrom.

Résumé

Le laboratoire Couches Minces & Photovoltaïque de l’Empa est parvenu à développer, au moyen de méthodes innovantes, un nouveau procédé de fabrication de cellules solaires CIGS à haute efficacité sur substrat plastique. Ce procédé a permis de fabriquer en laboratoire des cellules solaires légères et flexibles de plus de 20% d’efficacité. Pour que cette technologie contribue à résoudre le problème de l’approvisionnement en énergie, il est essentiel de démontrer que l’on peut transférer ce procédé statique dans des équipements de production industriels.

Le but de ce projet P&D est de réaliser un système de dépôt CIGS « roll-to-roll » (R2R) dans lequel le procédé innovant de co-évaporation multiniveau développé en laboratoire sur petites surfaces peut être implémenté à l’échelle industrielle, sur grande surface et en dépôt dynamique. La réalisation de ce réacteur pilote joue un rôle central dans l’industrialisation de la technologie CIGS à bas coûts et haute efficacité ainsi que dans la diffusion de l’énergie solaire.

Abstract

In the Laboratory for Thin Films and Photovoltaics at Empa a novel process for fabrication of highly efficient CIGS solar cells on plastic films was developed by using innovative concepts. Light-weight and flexible solar cells with efficiency above 20% were fabricated on laboratory scale. On the way contributing to a renewable energy supply it is essential to demonstrate the feasibility of transferring this still static process to industrial relevant deposition equipment.

The goal of this P&D project was to demonstrate a roll-to-roll (R2R) CIGS deposition system where the innovative multi-stage co-evaporation process developed in the Laboratory for Thin Films and Photovoltaics at Empa for small area substrates can be scaled up for coating on in-line moving large area substrates. Demonstration of such a pilot scale deposition system plays a pivotal role in industrialization of CIGS deposition technology for affordable, high efficiency solar cells and modules in future.

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1 Initial position

CIGS is recognized as an important thin film PV technology for cost effective manufacturing because of high efficiency potentials shown by the solar cells developed in the research laboratory at Empa and the possibility to benefit from roll-to-roll (R2R) processing. However, there is a lack of R2R CIGS production equipment supplies with proven process for high efficiency solar cell manufacturing on industrial scale.

There is no company providing CIGS evaporation systems with a process of multi-stage evaporation known to provide high efficiency devices. This is severely hindering the growth of CIGS PV industries since new companies with interest in CIGS production do not have possibilities of acquiring such systems. The proposed demonstration system would prove the industrial capability of the process and help in industrialisation of the technology and strengthen the position of Switzerland internationally in the field of photovoltaics.

2 Goal of the project

The goal of the pilot project is to develop and demonstrate a R2R CIGS deposition system in which the innovative processes developed in the laboratory for very high solar cell efficiencies (above 20%) can be scaled up towards industrial applications. Demonstration of such a system plays a pivotal role in industrialisation of highly efficient PV technology for large volume production.

The overall objectives of the pilot project are therefore:

- to develop an in-line R2R vacuum deposition system for the demonstration that the multi-stage CIGS co-evaporation technology can be scaled up for large size moving substrates
- to demonstrate a pilot level deposition system proof of concept based on a proprietary design and process know-how
- to attract industries as potential end users for subsequent scale-up for production

3 Procedure

Innovative multi-stage evaporation processes for the CIGS absorber layer were developed at Empa for obtaining an optimum chemical composition grading which proved to be highly reliable and reproducible and therefore suitable for industrial scale up. The developed processes are, however, stationary, i.e. the substrate is not moving relative to the evaporation sources. In this P&D project Empa wants to demonstrate that the multistage co-evaporation process can be transferred to industrially relevant in-line equipment.

Key issue of the development of the demonstration machine is the design of appropriate vacuum deposition system with evaporation sources having appropriate sequences and vapor flux profiles which allow the same composition grading of the CIGS for a moving substrate. A schematic of such source sequence is shown in Figure 1.

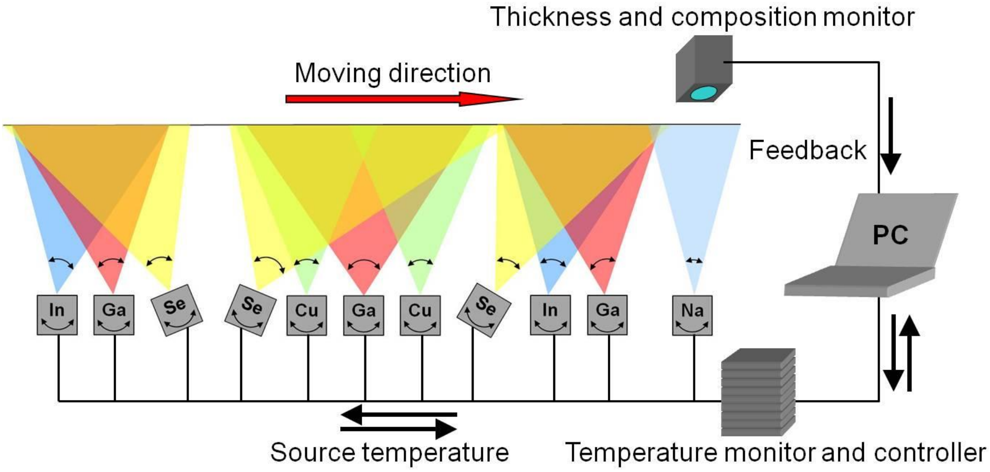

Figure 1: Simplified schematic of the pilot system for multistage evaporation of CIGS based on the Empa process which yields world record efficiencies flexible solar cells on polymer and metal foils. The processes are applicable also for high efficiency cells on glass.

The features of the R2R in-line multi-stage CIGS deposition machine are the following:

- Proprietary low-temperature CIGS process allowing various substrate materials including flexible polymer film and metal foil
- Continuous supply of moving substrates
- Multiple evaporation zones representing the different stages in the multi-stage process
- Evaporation sequences designed for reaching the same optimized composition grading in the CIGS layer as obtained from the laboratory scale process, for this optimization process the machine will be equipped with variable beam shapers and aligners for the evaporation sources
- Process control for chemical composition including feedback loop

Foreseen for the project was an in-line CIGS coating platform for flexible substrates of a width of maximum 50 cm. This is the standard substrate width where Partners can provide the necessary molybdenum coated substrate material with its existing sputtering equipment.

After extensive evaluation no equipment supplier could be identified to deliver a system with the required specifications within the budget constrains (see section Results and Findings). Finally, the Swiss based company Flisom AG joined the project for equipment development and end-user of the technology.

The project was divided into the following phases:

- Design phase I (2 months): During the design phase the overall and detailed concept is elaborated, mechanical details are discussed, the plans of the chamber and electrical supplies are finalized and orders for core components are placed.
- Design phase II (3 months): While the Chamber is being constructed, the component designs are finalized and ordered and the electrical racks are prepared and programming of the system is started.
- Construction and Assembly phase (4 months): Chamber and components are assembled and connected to the control system and electrical supplies attached. Various testing procedures and debugging is started. First layers will be coated and evaluated.

- Debugging and Ramp-up (3 months): CIGS processes are being implemented and the control mechanisms fine-tuned. Empa scientists are instructed to the usage of the tool.
- Joint testing and observation phase (6 months): The operation of the tool is still closely monitored and robustness of functionality improved. Scientists of Empa test coatings and determine operation parameters
- Continuous phase: Transfer of the static multistage process to R2R equipment

The planned (green) and resulting (red) timelines of the project are visualized in the following Gantt chart (Figure ***2***):

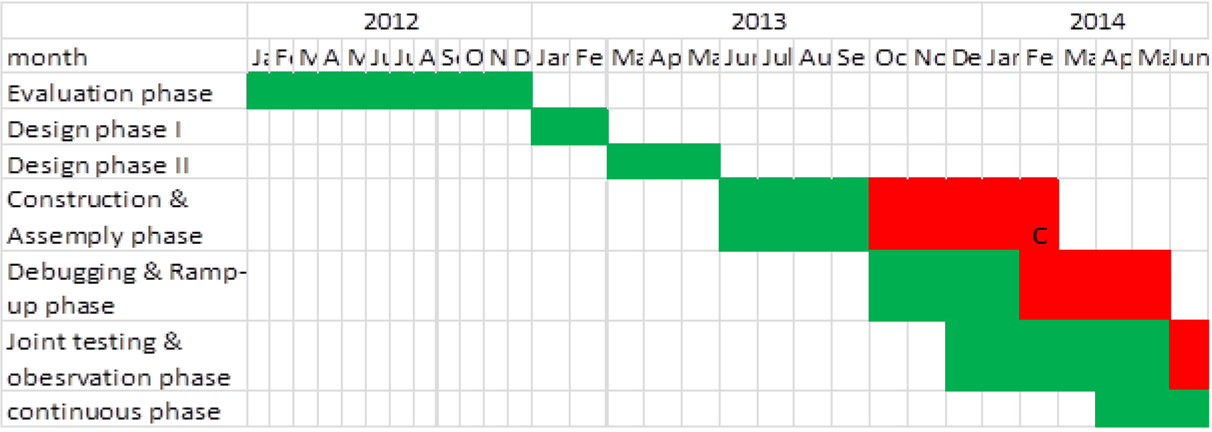

Figure 2: Gantt chart of project starting with effect of January 2013 to June 2014; C: first coatings deposited;

4 Results and Findings

4.1 Evaluation of equipment manufacturers

The quoted costs of manufacturing equipment by non-Swiss suppliers were significantly higher than what we estimated initially at the planning stage of the proposal. The difficulty was that equipment manufacturers have not made this type of equipment and some of the manufacturers have very close link with other CIGS companies.
For the purpose of the BFE supported project, “CIGS multi-stage in-line pilot machine demonstration”, Empa, Laboratory for Thin Films and Photovoltaics has requested Flisom AG Company to provide financial support and high level of technical guidance for delivering and installing a roll-to-roll (R2R) tool for continuous inline coating of CIGS layers on flexible substrates. The aim of the project was the transfer of high efficiency deposition processes from static to a dynamic coating process.
Flisom is one of the few companies worldwide that has developed and is continuously running a roll-to-roll tool for flexible polyimide substrates. It gained valuable experience in roll-to-roll substrate handling at high temperatures as necessary for CIGS deposition. Flisom has also developed propitiatory linear evaporation sources for evaporation of constituent elements (Cu, In, Ga, Se) and NaF compound in a cost-efficient way.
Flisom has developed various IP on hardware components, process control and tool design and is providing this know how to this project in order to allow the project to be carried out within the available restricted budget, short time frame and highest possibility of success.

4.2 Design of the roll-to-roll CIGS deposition equipment

Within a pre-study of various possible chamber geometries, the rectangular shape showed most advantages as the long, preferably horizontal, deposition zone could best be accommodated. Several of the side plates can be considered as modular to allow testing of various concepts within the same frame. Figure 3 shows the schematic design with a large modular side and bottom plate. The side plate is used to fix the winding and substrate heating hardware, whereas the bottom plate mainly contains the evaporation sources and controls.

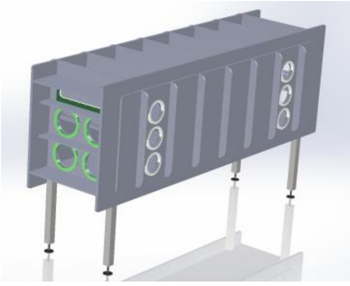

Figure 3: Preliminary concept of chamber design.

The suitability of the vacuum chamber for the specified base and working pressures was confirmed by an external engineering company in the first instance. For further optimization Flisom used a construction and simulation software to simulate the stiffness and torsion of the vacuum chamber by finite element analysis.

The detailed drawings with optimized stiffness were released to the manufacture of the vacuum chamber after evaluation of three different vacuum chamber manufacturers in Europe. After pre-acceptance test at manufacturer's site the vacuum chamber was delivered.

The chamber layout separates winding/outgassing and coating zones with functionalities as described in Table 1.

Outgasing of incorporated water and gases in the large amount of polyimide material in the vacuum chamber is a crucial step to allow good deposition conditions. The system allows outgassing and layer deposition at the same time by providing sufficient pumping capacity and gas separation between the process chamber and the winding units.

Table 1: Processing units within the vacuum chamber

Unwinding/Preheating Unit	CIGS coating Unit	Rewinding Unit
• Unwinder • Independent heating zones • Web tension controller • Web spreading • Cryo pumping	• Webguiding • Independent heating zones • Multiple evaporation sources • Selenium overpressure • Insitu composition monitoring • Condensation protected pumping	• Rewinder • Independent heating zones • Selenium degasing • Web position and tension controller

A special focus is set on integrating maximum process and tool diagnostics. Due to the flexible and modular concept it is easily possible to do upgrades or amendments:

- By placing additional flange for later buffer layer coatings that can be directly integrated without breaking the vacuum process.
- Adjustable number of heater circuits and position
- Fully adjustable crucible type, number, position
- Additional flanges for feedthroughs for supplies, measurements etc.

The uniqueness of this pre-industrial pilot machine is the high level of degrees of freedom in the evaporation geometry which will allow the transfer of the sophisticated multistage co-evaporation process developed at Empa.
In order to allow high degree of freedom for the pre-industrial coating equipment a fully modular concept was adapted. The vacuum chamber is equipped with large number of spare flanges with only three flange sizes to ensure reuse of component on different locations of the machine. The evaporation sources are installed to facilitate simple and quick readjustment of evaporation sequences and profiles.

4.3 Lab space preparation

Due to the large dimensions of the R2R coater new lab space was required. Therefore, a storage room had to be converted into a laboratory space. A clean room has been installed in order to guarantee the required working environment. Figure 4 shows the 3D simulation of the clean room facility. Separate cooling water supply for cooling of the machine is installed. A mechanical crane is included into the room concept. Different crane concepts were evaluated and the most appropriate one was selected. Foundation for the deposition machine enforced and track lines are installed in the floor for moving the door of the deposition machine.

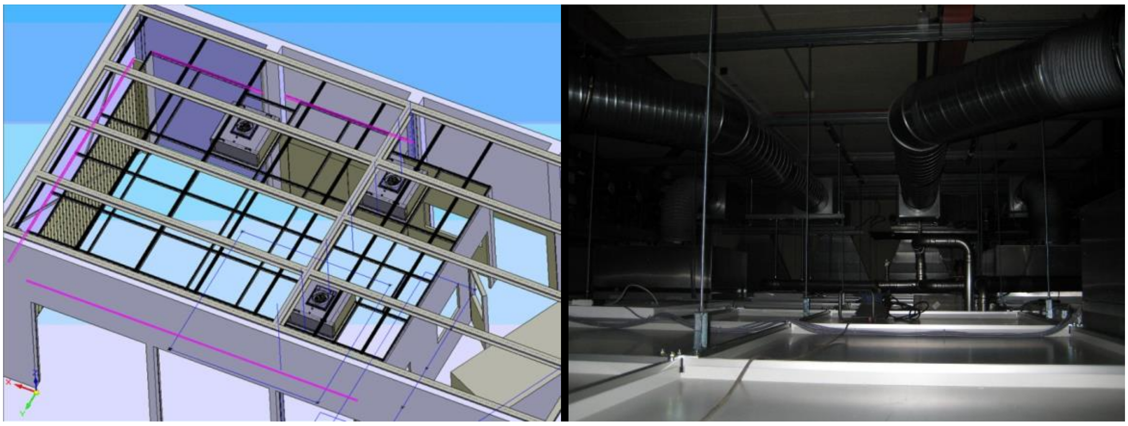

Figure 4: Plan of the clean room facility for the CIGS pilot machine (left) and installed ventilation and cooling water system on top of the roof of the clean room facility (right).

4.4 Chamber assembly and components installation

After reception and placement of the chamber, the assembly work of the components and pre-assembled tools was started.
The two stage pumping system is defined with pre-vacuum pump, external exhaust filter system and turbo pumps which is equipped to the vacuum chamber. In order to reach the required pressure in the deposition chamber dedicated processing zones were separated from each other so that outgassing processes and coating processes can happen simultaneously.

Process monitoring is required in order to guarantee a reliable and reproducible process. Therefore, X-ray fluorescence (XRF) is used to measure chemical composition and thickness of the coatings.

4.5 Control system

The control system architecture has been specified based on Beckhoff hardware. The Programmable Logic Controller (PLC), Motors and drives have been assembled and mounted to the chamber outside the vacuum. The electrical cabinets were specified and ordered from a local supplier. Figure 5 shows arrangement of the electrical components in the cabinet. The order was placed after four different manufacturers for the electrical cabinet were evaluated.

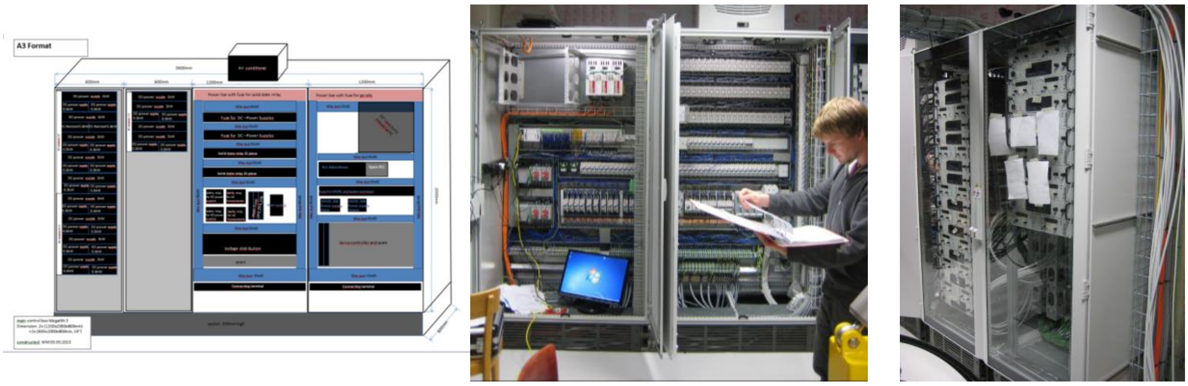

Figure 5: left: Schematic of the arrangement of the components in the electrical cabinets; middle: photograph of the installed cabinet containing the control units for winding and substrate heating; right: cabinet containing the power supplies and control units for the evaporation sources

The software to control the process has been provided. Nevertheless the automatic evaluation of the process diagnostics still has to be implemented and tested in conjunction with the establishment of the baseline process.

4.6 Functional tests

After assembly of the chamber and leak elimination, several tests had to be conducted before doing first coating experiments:

- I/O tests for all electrical components
- Vacuum pump down tests (leak testing with helium, leak rate evaluation)
- Winding tests (webguiding, telescoping)

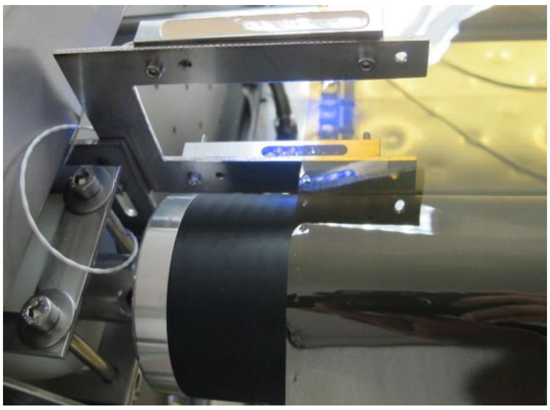

Figure 6: Band edge detection with optical sensors are required to control the web guiding system in the unwinding and rewinding area to prevent that telescoped rolls will cause coating issues in the deposition zone.

Keeping the web flat and in minimal stress conditions is the key task of the winding system in this application (Figure 6). A challenge is to allow natural expansion and contraction of the polymer substrate during heating and cooling without creation of wrinkles or creases during the coating process. Spreading functions of the web and active repositioning of the coils have been implemented to control the web winding throughout the process path in the chamber.

- Substrate heating tests with running web

The CIGS deposition process involves substrate temperatures up to 500°C. As massive heat radiation is also emitted from evaporation sources, good control of the temperature with fine granularity over the web surface is required.

- Single source evaporation experiments

Figure 7: rewinding zone with copper coated web after a copper deposition test.

Before coating various elements simultaneously, basic checks with single evaporators were conducted. The picture above is showing copper coated substrate foil in the rewinding section of the chamber. Temperature measurements at various locations in the chamber were made to test the correct working of the cooling circuits. Figure 7 shows a copper coated polyimide web after single element experiment of the Cu sources.

- Data logging and safety procedures

A sophisticated server infrastructure with data logging function has been established and implemented. Automatic logging of data and powerful data display functions are very helpful in long term process development.

4.7 Coatings

After reaching a satisfactory state of all the tested elements, coating experiments with selenium vapor were started.
From the beginning onwards it is important to evaluate the maintenance possibilities of the system. Long evaporation cycles will require cleaning of the exposed shielding areas after each run. Therefore ergonomic mounting and dismounting of the components will have a major impact on the output of a production system. Especially the deposition zone has been designed for easy maintenance by hand mountable and replaceable shielding. They consist of thin replaceable metal foils that can be easily mounted and replaced.
Further, the profiles of the evaporators have to be checked and calibrated for layer homogeneity across the web width. Using ex situ XRF, the coated layers were analyzed.
As process development has only started recently, optimization of the coating profiles is still ongoing. Nevertheless reasonable starting homogeneities could be achieved after a few initial source reconfigurations.

As a multistage inline coating tool requires a large substrate area in the process zone, a major concern at the beginning of the project was to assure a good web quality of the coated areas. Significant amount of time has been spent to design the right winding and webtransport system that can handle the large temperature differences in the heating and cooling zones as well as to make sure, all the used components can support the required process temperatures. After assembly and initial testing of the winding system, the real tests had to be done with all the evaporators running and coating at deposition speed. The optimization phase resulted in very important learnings and finally a system that has yielded very good web quality at the desired coating speed. The next steps will be to finalize the baseline process development and generate solar modules from the coated layers to verify any influence of the winding system on the electronic parameters and further fine-tuning of the mechanical setup.

4.8 Development of CIGS multistage co-evaporation process and process transfer

In parallel to the design and ramp-up of the roll-to-roll CIGS deposition equipment, Empa is further developing its multistage co-evaporation process in a stationary deposition chamber. The main goal is the identification of critical process parameter and a better understanding of the process window of CIGS growth for high efficiency solar cells, in order to facilitate the transfer from the stationary lab-scale CIGS deposition process onto the inline coating tool. Additionally, innovative process steps are developed in order to push the record efficiency of the CIGS technology even further.
This resulted in additional input and required modifications to the equipment specifications.
Due to the high flexibility of the initial design of the R2R deposition equipment most of the additional required specifications discovered during this parallel process development were already realized during construction and assembly phase of the machine.

Process reproducibility – average composition
The stationary multi-stage co-evaporation process was developed in order to carefully control the nanoscale compositional grading that forms during growth. It was found that this process is highly reproducible and can yield solar cells with high open circuit voltage (V_{oc}) of about 700 mV for at least 20 consecutive depositions. Moreover, it was found that the average Ga content of the layer (which determines the average band gap of the material) and the final Cu content can be varied to some extent without severe detrimental effects on the final cell efficiency.

Ga grading
The shape of the Ga grading is key for high efficiency solar cells. Typically, a double-Ga grading is naturally formed during growth, with a higher Ga content at the front and at the back of the layer, and with a lower Ga content region (notch) in the middle. It was shown that a too pronounced notch leads to enhanced recombination in the solar cell and the multi-stage co-evaporation was developed in order to allow control over the notch shape and position. There is still no consensus in the research community about an optimum shape of the Ga grading, but we could show that there exist several different shapes and widths of the notch that are not detrimental to the cell efficiency.

Se pressure
The growth of the CIGS layers is based on the co-evaporation of the metals (Cu, In, Ga) along with a high Se overpressure to ensure good material quality. Control of the optimal Se flux is important in order to ensure highest efficiency. However, it remains challenging to precisely measure and control the Se flux. Several experiments were made with a new Se source setup that allows monitoring the relative amount of evaporated Se, and a better control of the Se

overpressure.

Deposition rate and absorber thickness

From an industrial production perspective, high production throughput is required. Higher deposition rate and reduced absorber thickness are two aspects that can be pursued in order to reduce the overall deposition time and material cost in a roll-to-roll deposition chamber. However, they usually come along with a reduction in the cell efficiency due either to a lower material quality of the layers grown at higher rate or to insufficient light absorption in thinner absorbers.

Empa was able to reduce the absorber thickness from the standard thickness of 2-3 µm for highest efficiency devices to about 1 µm. Efficiency above 16 % was obtained with a 1.2 µm absorber thickness, by adapting the low-temperature multistage process for absorbers with such a thickness.

Alkali post-deposition treatment (PDT)

Alkali doping of the CIGS layers is required in all high efficiency devices. Na has long been known to be beneficial, either diffusing from the soda-lime glass substrate during growth at high temperature, or added extrinsically after growth for CIGS layers grown at low temperature. Empa has developed a NaF post-deposition treatment, where NaF is evaporated onto the heated CIGS layer at the end of the multistage deposition process. Recently, Empa has also developed and introduced an additional post-deposition treatment with KF, which led to a new world record efficiency of 20.4% on flexible substrate. The addition of KF greatly modifies the surface of the CIGS layer and allows the formation of better junction with the buffer layer and to reduce optical losses by reducing its thickness. The implementation of the KF PDT by other research groups and companies in the world has led to a wave of new record efficiency announcements for the CIGS technology, bringing it now to the current 21.7% efficiency level. This new process step does not need major changes in the planning of the inline deposition chamber, as it only necessitates an additional source in a similar manner as already planned for NaF.

Module

Based on the knowledge described above, mini-modules were processed to show homogeneity and scale-up potential of the technology developed at Empa. After process optimization, a 16.9% efficiency mini-module was obtained, which represents the world record efficiency for a flexible mini-module.

Process transfer

The novel processes (KF-PDT) and process parameters (compositional grading, Se flux) discovered in this parallel study were directly implemented in the ongoing design of the R2R deposition system. Furthermore, all necessary baseline data were acquired for highly efficient CIGS solar cells including compositional depth profiles, thicknesses and compositional & thickness tolerances as reference for the in-situ XRF measurements and ex-situ materials and device characterization in order to allow a fast process transfer on the R2R tool.

5 Discussion and appraisal of the results

We encountered severe challenges related to the non-availability of suitable vendors to construct a customized R2R pilot system to our specifications within the approved budget and the desired timeframe. The response of companies indicated difficulties for reliable and economical procurement of some crucial components needed for the customized development of the pilot system. Long delivery time of the equipment by some companies was another problem. Therefore, we reconsidered the execution plan of the project and more involvement of the project partner company Flisom in terms of their contribution. It was decided to acquire the system from Flisom since the company has already demonstrated a system for CIGS coating on rolls albeit with lower efficiency than we want to achieve.
This jointly developed equipment by Empa and Flisom under a close collaboration, furthermore allowed proper protection of the proprietary knowledge of Empa and Flisom and provide a competitive edge over other partnerships of competitors in other countries.
The targeted timing of the project proved to be very ambitious and not realistic within in the limited resources available but at the end of the project the assembly of the R2R CIGS deposition equipment was finalized. The following table summarizes the status of the tasks in the different project phases (refer to Gantt chart in Figure ***2***):

<u>Design phase I (2 months):</u>

Task	Status compared to original planning /Remark
overall and detailed concept is elaborated	achieved
mechanical details are defined	achieved
plans of the vacuum chamber ready	achieved with delay
electrical supplies are finalized	achieved
orders for core components are placed	achieved

<u>Design phase II (3 months):</u>

Task	Status/Remark
Construction of vacuum chamber	achieved with delay
component designs are finalized and ordered	achieved with delay
electrical racks are prepared	achieved
programming of the system is started	achieved

Construction and Assembly phase (4 months):

Task	Status/Remark
Chamber and components are assembled and connected to the control system	achieved with delay
Various testing procedures and debugging is started	achieved with delay
First layers will be coated and evaluated	achieved with delay

Debugging and ramp up phase (4 months):

Task	Status/Remark
Debugging winding system	achieved with delay, longtime learning in progress
Debugging substrate heaters	achieved with delay
Debugging evaporation sources	achieved with delay, continuous improvements ongoing
Metal and CIGS coating runs	Achieved with delay

Joint testing and observation phase (6 months):

Task	Status/Remark
Process development runs	started
Reproducibility of process parameters	Observation ongoing
Implementation of automated feedback loops	To be started after XRF calibration and

Currently Flisom has finished the debugging and ramp up phase and is in the joint testing and observation phase (although long term learnings and debugging is still ongoing) where the CIGS specialists are implementing baseline processes and conduct material analysis of the coated substrate and layers. In the beginning of June the pre-acceptance tests was achieved. Fine tuning and feedback loop implementation is ongoing.

6 International collaboration

Part of the presented work is co-financed by a FP7 project "R2R-CIGS". The European consortium consists of 4 research institutes (TNO, Netherlands; Empa, Switzerland; ZSW, Germany; CPI, UK) and 6 companies (Manz CIGS GmbH, Germany; Flisom, Switzerland; Isovoltaic, Austria; Beneq, Finland; Solaytec, Netherlands; Mondragon Assembly; Spain).

7 Conclusion and next steps after closure of the project

The CIGS multistage in-line pilot machine was successfully developed within the project despite the big difficulties encountered in the first year (2012) to identify suitable equipment suppliers and the huge technical challenges in equipment design and construction. Also important reference data of highly efficient CIGS solar cells for in-situ XRF monitoring and ex-situ depth profile measurements were collected within the project period.

At the end of the project the pilot machine is ready to demonstrate the transferability of the multistage low-temperature CIGS deposition process from small scale, static laboratory equipment to medium scale, in-line equipment with industrial relevance.

After the closure of the project the partners Empa and Flisom will start with continuous operation and process transfer. In order to demonstrate final success of the process transfer the partners will continue to achieve the cell and module efficiency targets.