



Executive summary

IN-LIGHT project "Innovative bifunctional aircraft window for lighting control to enhance passenger comfort" has been a 3-years collaborative project funded by the European Commission under the call FP7-AAT-2012-RTD-1 and in particular under the activity AAT.2012.3.1-3.: "Systems and equipment". IN-LIGHT has put together a multidisciplinary and highly complementary consortium of 10 partners from 6 member states plus one partner from a third country. The project partnership included research organisations [IK4-CIDETEC, Fraunhofer Institut Silicatforschung (FRAUNHOFER ISC), VTT Technical Research Centre of Finland, IK4-TEKNIKER, Institut de Recherche d'Hydro Quebec (IREQ), Andalusian Foundation for Aerospace Development – Center for Advanced Aerospace Technologies (FADA-CATEC), Consorzio Venezia Ricerche (CVR)] and industries [AIRBUS Group, GKN Aerospace] and it was endorsed by EASN-TIS. The overall goal of IN-LIGHT was to develop a new concept of bifunctional smart aircraft window combining two technologies, Electrochromism (EC) and transparent organic light emitting diode (TOLED) lighting, enabling a tailored cabin environment regarding heat and light transmittance as well as ambient lighting according to passenger preferences.

Briefly, during the first period of the project (M1-M18), efforts were mainly focused on the definition of the technical requirements and the development, testing and selection of the key materials and coating processes for the fabrication of the electrochromic and TOLED devices. By the end of the first period, all-solid EC and TOLED devices of small size (up to 25 cm² and 18 mm² respectively) on flexible plastic were fabricated and fully-tested within a first testing campaign according to a work plan elaborated during the first months of the project.

During the second period (M19-M36), two core topics were addressed:

- 1- The combination of both technologies (EC-TOLED) in a single "tandem" bifunctional device.
- 2- The scaling-up of materials and coatings for the production of large-area devices.







By the end of the project, all-solid flexible EC devices with the dimensions of a real-life aircraft window (targeted size: $30x40 \text{ cm}^2$) and flexible large area interconnected TOLED modules (190 meters web; 180 cm^2 emissive area) were successfully produced.

The large-scale prototypes performance was assessed in accordance with the requirements established by the industrial partners within a third testing campaign.

Additionally to the technical aspects, special attention to dissemination activities was paid in order to amplify the impact of the project through a series of dissemination, communication and activities including scientific publications, participations at scientific and technical events, newsletters, news releases, etc. The IN-LIGHT consortium defined a "Plan for Using and Disseminating the Foreground", which will be considered for the subsequent exploitation actions.

Project context and main objectives

Comfort plays an increasingly important role in the interior design of airplanes. Comfort comprises many aspects including temperature and lighting. Aircraft operators and interior designers are demanding a new level of aesthetic quality and functionality in the cabin and think of the passengers during the design process, evaluating different elements such as lighting, shapes and colours in order to determine which will provide the maximum sense of comfort, especially to passengers on very long flights. Like at home, temperature and lighting in the passenger cabin plays a crucial role in overall comfort.

In this context, the overall objective of the IN-LIGHT project was to develop a new concept of bifunctional smart aircraft window combining two technologies: Electrochromism and TOLED lighting.

- Electrochromic technology will allow the passenger to control at will the amount of sun-light coming through the window, switching from a colourless transparent state to a deeply coloured transparent state.

- Additionally, a TOLED lighting system, also controllable by the passenger, will be integrated in the window, being invisible in the off state and providing a very innovative type of cabin interior lighting offering novel aesthetic effects.

This new concept of smart aircraft window will add significant value to conventional windows and will contribute to the passenger comfort enabling a tailored cabin environment regarding heat and light transmittance as well as ambient lighting according to passenger preferences.

The electrochromic (EC) technology developed in the project is based on redox-chromophores anchored on nanostructured semiconducting films. Traditionally, the nanostructured layer preparation comprises a high temperature sintering process (i.e. at 450°C) that is not compatible with plastic substrates, the preferred materials employed for aircraft windows for weight-reduction reasons. Furthermore, introduction of high temperature processes increases the manufacturing cost.

IN-LIGHT aimed to develop a high-performance plastic electrochromic aircraft window based on modified transparent nanostructured electrodes processed at low temperatures (< 120°C). Moreover, IN-LIGHT focused in the use of technologies that have led to all-solid state devices, avoiding the use of liquid components in order to eliminate any risk of leakage and simplify the manufacturing process.





Regarding the TOLED technology, is based on commercially available, soluble OLED materials (hole injection layer, emissive layer and electron injection layer) in combination with semitransparent or transparent electrodes.

The main technical goals of IN-LIGHT to be achieved were:

- Development of adequate basecoats on acrylic substrates accomplishing all of the requirements for the subsequent processes for the fabrication of EC devices.
- Creation of high performance TCO sheets with low resistivity onto plastic substrates to ensure fast and homogeneous coloration/bleaching, mainly in large-area devices.
- Development of highly transparent (90% VIS transmittance) electrochromic nanostructured coatings through low temperature processes (< 120°C) onto the conducting plastic substrates.
- Development of suitable high transparent solid electrolytes with high ionic conductivity and optimal adhesion.
- Selection of a UV stabilizer concept tailored to the needs in terms of electrochromic molecules stability and aircraft cabin window components.
- Production of flexible all-solid electrochromic devices with proper switching between a colorless and a deep colored state.
- Preparation of TOLED stack with altogether five functional layers (ANODE-HIL-ELM-ETL-CATHODE) manufactured with high-throughput wet and/or vacuum deposition technologies and reaching an overall transparency > 65%.
- Combination of both technologies (EC and TOLED) in a single device.
- Scaling-up the production of the materials and coatings to the quantities required for the preparation of targeted size prototypes.
- Fabrication of all-solid EC devices with the dimensions of a real-life aircraft window (targeted size: 30x40 cm²).
- Fabrication of large flexible TOLED devices with rectangular/stripe shapes in a first stage and functional arbitrary size/shape light surfaces in a second stage.
- Fabrication of tandem (EC-TOLED) aircraft window prototypes (size 30x40 cm²).

The scientific and technological cooperation in IN-LIGHT consortium and the roles of the different partners in the project were well balanced covering the complete chain from raw materials, scientific comprehension, technological research and end users.

Main Scientific and Technical results/foregrounds

The project was structured into 8 workpackages (WP). WP1 to WP4 consisted of the fundamental research effort of the project. WP1 included the definition of the technical requirements and a continuous technological watch in the project related fields. WP2 was concerned with the synthesis and development of materials covering the different required components of the electrochromic and TOLEDs devices structure. WP3 dealt with the fabrication of devices at lab-scale in order to test the performance associated to each specific technical material developed in WP2, enabling the optimization before proceeding with the scaling up of the technologies. WP4 focused on scaling up the production of the adequate materials and coatings evaluated and selected in WP3 as well as on the fabrication of prototypes.

Product validation activities were included in WP5 by testing the final prototypes according to the end-users and customer requirements. WP6 provided the life cycle assessment of the new developed product system. The potentials health and safety impacts associated with the use, synthesis, and final application of materials for the novel windows were also evaluated within



this WP. Management activities were addressed in WP7 while dissemination and exploitation activities were performed under WP8.

The main scientific and technical achievements are briefly described below following the WPs structure. A brief summary of the management (WP7) and dissemination/exploitation activities (WP8) is also included.

WP1- Customer requirements specifications

The main functional requirements were defined at the beginning of the project. Because the research on an electrochromic shadable window in combination with a TOLED lighting was far beyond the state of the art and each device for its own had to be further developed, the description of the requirements was not based on typical industrial AIRBUS standards. For most of the functional requirements there were no specific standards applicable, since AIRBUS has no electrochromic devices or TOLEDs in their product portfolio. Since IN-LIGHT was dealing with basic research and high-level development, it was of most interest to know where the limits of the produced samples, electrochomic cells, TOLEDs and their combination were.

A good guideline for the testing procedures was the standard RTCA DO-160G "Environmental Conditions and Test Procedures for Airborne Equipment".

The DO-160G covers standard procedures and environmental test criteria for testing airborne electrical and electronic equipment. The tests specified in the DO-160G are typically performed to meet international regulations covering electrical or electronic equipment that is installed on commercial aircrafts. The tests and test levels or limits found there are applicable to nearly every type of today used aircraft, including small general aviation aircraft, business jets, helicopters, and Jumbo Jets and should cover even very new airplanes like the Airbus A350.

A continuous technological watch in the project related fields as well as an identification of new competitive technologies and further applications was also performed within WP1. Three annual technological watch reports were elaborated during the life of the project. The reports included a compilation of news, and recent patents and scientific publications on technical advances and competitive technologies related to the IN-LIGHT project research areas.

WP2- Materials design and development

The main objectives of this WP were: 1) The evaluation of available conductive substrates and development of transparent conducting oxides (TCO) layers with low resistivity and good mechanical properties on plastic substrates. 2) Development of highly transparent solid electrolytes 3) Preparation of transparent nanostructured electrodes with good adhesion on TCO and with ability to immobilize electrochromic molecules. 4) Design and synthesis of organic electrodes. 5) Evaluation and selection of UV stabilizers. 6) Formulation of commercially available TOLED materials to make them suitable for high through-put deposition technologies.

Suitable TCO compositions. GKN and TEKNIKER collaborated for the development of suitable transparent conducting substrates to be used as the electrodes in the electrochromic and TOLED devices. Thus, to support the development of the electrochromic devices, GKN originally supplied a dual layer coating system on acrylic to TEKNIKER to which multiple sputter layers were to be deposited onto the surface. The dual-layer coating system comprised of a basecoat layer and a UV curable topcoat layer which provided the right surface properties to enable the subsequent application of the sputter layers. A number of factors drove the change of the dual-layer system towards a single layer solution: a) Neither coating formulations originally supplied on acrylic could be distributed to external companies due to IP constraints; b) A





reduction in the number of complex application and cure processes presently involved with the dual layer system; c) Reduction in the cost of manufacturing substrates for development work or product in the final application.

The final coating solution developed in the project has extremely high optical clarity and does not present defects or performance variability between batches or when exposed to durability tests.



The single layer system could be integrated with the subsequent processes given the achievement of the following properties: 1) It withstands the TCO deposition temperature, 2) Has a high surface energy and increased wettability to promote adhesion of solid particles, 3) Has the sufficient hardness to support stress generated by particles and flexibility to resist the impact of deposited particles, 4) Has excellent adhesion to acrylic substrate. Most importantly, the development work carried out

enabled the creation of a novel IP under this project and demonstrates the suitability of the final coating solution for the application of electrochromic devices.

Regarding the TCOs, the activities were focused on the development of transparent conductive aluminium doped zinc oxide (AZO) layers on glass and polymeric substrates acting as front and back contact for OLED and the electrochromic devices with: Sheet resistance values ≤ 50 ohm/sq, Transmittance $\geq 80\%$, chemically stable and compatible with the electrochromic and TOLED manufacturing process. Magnetron sputtering has been used as the PVD (Physical Vapor Deposition) technology, using DC-pulsed and RF power sources.

AZO layers on lacquered PC and acrylic substrates with sheet resistances of 110 -275 ohm/sq and 75% of transmittance were produced. However, during the course of the project, it was envisaged the interest of having AZO on flexible PET substrate, instead of on rigid PC or acrylic, for a better compatibility with the fabrication techniques of EC and TOLED devices. For this purpose, research on AZO coatings on commercial PET substrates with RF source was carried out.

After establishing the optimum RF process conditions, AZO layers on flexible PET substrates with the following characteristics were produced:

•Resistivity values as low as 1.6×10^{-3} ohm cm and sheet resistances as low as 31 ohm/sq (lower than target: sheet resistance < 50 ohm/sq)

•Transmittance values as high as 83% in the visible range (400-800 nm) and 82% in the visiblenear IR (400-1500 nm) (higher than target: Transmittance > 80%) •Non absorption in the blue range=> No vellow coloration

These results are highly promising since they are beyond nowadays commercially available transparent conductors. AZO/PET samples developed in IN-LIGHT project have shown optoelectronic properties as good as those of commercial ITO/ PET system. But as being an Indium free material, it avoids the use of one of the 14 raw materials that the European Commission has identified as critical both economically and strategically. In this way the commercialization of this AZO/PET system would allow to overcome important drawbacks related to the use of ITO such as toxicity and import dependence of base material to warranty competitiveness of the European optoelectronic industry.



IN-LIGHT- G.A. 314233 Final report



The AZO-coated substrates were suitable for the fabrication of the TOLED devices. However, they were found to be not compatible with the manufacturing processes of the electrochormic since such processes chemically attack the AZO layers due to their instability against acidic and basic solutions normally used for over-coating. In order to overcome this limitation, a bilayer

electrode formed by an AZO layer and a TiO_2 layer, both deposited by magnetron sputtering, was developed. The aim of the TiO_2 layer was to chemically protect the AZO layer against subsequent acidic solutions used to deposit EC films while not affecting negatively the optoelectronic properties of the AZO layers. This resulted to be a very effective approach to protect the AZO enabling the fabrication of EC devices with good performance on the new AZO-coated substrates.



Transparent solid electrolytes. In most of the technologies and devices reported in the state-ofart, the electrolyte, which is the intermediate ion-conducting layer in contact with the electrochromic layer and the counter-electrode, is a liquid or gel-type material. Liquid electrolytes are convenient for fundamental electrochromism studies (i.e. with lab-scale devices), but they are not suitable for practical applications due to the obvious risks of leakage and since gravity-induced forces may be excessive in large area devices. Optimal sealant of the device is crucial when liquid electrolytes are used and even though, total absence leakage is not guaranteed.

In this context, the advantages of the solid polymeric ionic conductors are recognized. However, the lower ionic conductivity presented usually in "dry" polymers it is a disadvantage to overcome. Furthermore, for its application in electrochormic windows, high transparency is also a vital requirement. This task addressed the identification of polymer electrolytes having appropriate electro-optical properties such as high ionic conductivity (> 10^{-5} S/cm) and optical transparency as well as optimal adhesion.

In order to achieve these objectives, two types of polymer structures were investigated: a linear (DAP) and multibranch (4Brs). Lithium bis(trifluoromethane sulphone)imide (LiTFSI for



short) was used to provide the ionic conductivity in the polymer matrix. These polymers were selected based on their solvating power of the imide salt, which is easy dissociated on the polymer chains. In order to ensure mechanical strength of the polymer and separator film, a small amount of initiator was added to the polymer composition. To meet the project requirements, different polymer formulations and curing conditions (thermal, UV and hibrid crosslink) were explored.

Deep investigation of the above-mentioned aspects performed by the Institut de Recherche d'Hydro Quebec led to the successful production of a suitable solid electrolyte accomplishing the targeted requirements and enabling the fabrication of all-solid electrochromic devices in the project.

Transparent nanostructured coatings. The main goal of this task was the development of highly transparent nanostructured semiconducting layers for the subsequent attachment of electrochromic molecules. Conventional methods for the preparation of semiconducting TiO₂ nanostructured films involve high temperature treatments (> 400°C), which are not compatible with the use of plastic substrates. The critical point of this task was to achieve layers



IN-LIGHT- G.A. 314233 Final report



with adequate conductivity and high active surface suitable for the anchoring of electrochromic molecules, avoiding high temperature processes. Proprietary sol-gel and hydrothermal processes have been utilized by FRAUNHOFER ISC within the project to prepare well dispersed nanocrystalline TiO_2 particles with sizes well below the scattering limit (< 15 nm) to ensure transparency.

dispersions, From these nanostructured films have been successfully prepared TCO substrates, on accomplishing the required characteristics in terms of transparency (above 90%), conductivity and surface area after а thermal treatment at only 120°C.



Tailoring the pore characteristics and the thicknesses was found to be crucial for the EC devices performance. The pore characteristics of the TiO_2 were tailored by varying the size of the nanoparticles while the thickness of the films was adjusted by changing the drawing rate during dip-coating. It is noteworthy that some limitations to achieve transparent mesoporous TiO_2 films on the AZO-coated substrates provided by TEKNIKER were found, due to a chemical incompatibility between the TiO_2 dispersions (being acidic) and the AZO (being basic).

New TiO₂ dispersions, specifically formulated to be suitable for AZO-coated substrates, were successfully developed by FRAUNHOFER ISC, overcoming the mentioned limitations.

Electrochromic systems. Viologens (1,1'-disubstituted-4,4'-bipyridilium salts) are a class of cathodic electrochromic compounds displaying different colours in dependence of their oxidation state and the nature of the substituents at the nitrogen atoms. A series of viologens with different substituents have been synthesized by CIDETEC in order to get blue or neutral colorations as required by the end-users. Moreover, phosphonic acid ending groups have been introduced in order to enable the anchoring of the viologens on the surface of the nanostructured TiO_2 films.

Electrochromic films have been prepared on TCO-substrates by modifying the TiO_2 films with the viologens. The modification was performed by immersing the films in viologen aqueous solution. The optimal viologen concentration and grafting time were adjusted. A new viologen providing colorless-to-blue switching and enabling a very fast grafting onto the TiO_2 surface (reducing the production times and avoiding damage of the TCO layer caused by other typical viologens) was designed and developed specifically.

Modification of TiO_2 films by immersion in viologen solutions (named as "Route A" within the IN-LIGHT project) is well-known in the state-of-the-art. A new strategy (named as "Route C" within the project), involving the direct synthesis of viologen-modified TiO_2 nanoparticles, was developed in close collaboration by FRAUNHOFER ISC and IK4-CIDETEC. In this manner, the step of immersion of the TiO_2 films in viologen solution is avoided and electrochromic films can be directly coated (one-step production).





The development of this new procedure for the production of electrochromic nanoparticles and films represents one of the most significant achievements of the project. This innovative method introduces major advantages *vs* the state-of-the-art method, such as being more cost-efficient and easier to scale-up, being compatible with AZO-coated plastic substrates and providing higher optical contrast for the same film thickness.

Negative aspects of <u>SoA</u> method (Route A)	New method by IN-LIGHT (Route C)
 Problems on AZO: instability of AZO against acidic TiO₂ coating solution instability of TiO₂-coated AZO against acidic viologen solution > Solution: sputtered TiO₂/AZO substrates 	 acidity of Route C coating solutions (-) subsequent modification with viologen not needed (+) Solution: sputtered TiO₂/AZO substrates
 Up-Scaling / Tandem device-topic difficult: Two-steps preparation of EC films: 1-Deposition of TiO₂ films; 2-Modification with viologen 	 One-step preparation of the EC films: 1-Deposition of viologen-TiO₂ films (lower-cost)
 Modification step: long immersion time in standard viologen solution damages TCO/plastic substrates Solution: New viologen specifically designed and synthesized in IN-LIGHT required 	 Standard viologens are suitable (lower-cost)

UV stabilizers. For electronic and electrochemical devices comprising organic materials, an effective light stabilizing concept is crucial to ensure a long lifetime. Task 2.5 of the IN-LIGHT project addressed the selection of type and intensity of radiation that the organic EC device components will be exposed to, as well as the screening of suitable UV stabilizers, taking into account chemical and optical compatibility considerations.

Several approaches were considered such as the addition of UV absorbers or other light stabilizing products (inner solution) and the use of UV screening plies and films (external solution). Moreover, the effect of the metal oxide films employed in the device themselves had to be assessed.

After basic considerations and preliminary tests, a concept had to be developed of how a viologen-based device could be protected from harmful UV radiation. Based upon the aforementioned concept, weathering experiments were performed.

The findings obtained clearly suggested an external solution to be the most viable approach. This is mainly due to three facts: First, the TiO_2 carrier films effectively screen UV radiation up to 360 nm. The same would occur for the doped zinc oxide films under consideration for use as transparent electrodes. Second, any light protecting additive introduced directly in the electrolyte layer has strong adverse effects on the cross-linking process (which is affected by absorbers and radical quenchers) or on the optical properties (discoloration by colored species). Third, using an external solution, in particular such as a UV protecting film, would probably help sealing the device from oxygen and moisture.

Transparent OLEDs. An organic light-emitting diode (OLED) device consists of very thin sandwiched solid layers of materials. When an electric current is supplied, the negatively charged electrons in the cathode layer move through the organic substances towards the





positively charged anode layer. At the anode's side, electrons drawn towards the anode leave holes in the conductive material. These positively charged holes jump to the organic material to recombine with electrons, which causes electroluminescent light.

Transparent OLEDs (TOLEDs) use only transparent or semi-transparent components (substrate, cathode and anode) on both sides of the device. Consequently, when a TOLED display is turned on, it allows light to pass in both directions. Thus, this technology enables to create displays that can be made to be both top and bottom emitting (transparent).

Within this task, commercially available materials for the TOLED active layers (hole injection layer, emissive layer and electron injection layer) were formulated by VTT to a printable/coatable form by adjusting the ink properties. Variable roll-to-roll manufacturing methods e.g. gravure, flexography, screen printing and slot dye coating for each TOLED layer were evaluated in laboratory scale. This included the evaluation and optimization of processing parameters such as speed, cylinder design and thickness control which needs to be optimized for each layer separately. The rheological and wetting characteristics of the inks and substrates in terms of viscosity, surface tension, and surface energy were systematically characterized during the formulation process. The aim was to provide a TOLED stack with an overall transparency higher than 65%.

Among the numerous functional materials evaluated, a white light emitting polymer from Merck (SPW-111) was finally selected since provided the best efficiency while ZnO from Nanograde was used as electron transport layer for its good performance and printing properties and a silver ink (ECI5003) for the top cathode.



WP3-Fabrication and characterization of devices.

This WP dealt with the fabrication, characterization and optimization of EC and TOLED devices at lab-scale utilizing the materials and processes developed in WP2. Both workpackages were closely interconnected, since depending on the results obtained from the characterization measurements carried out in WP3, the materials and processes were properly modified and adapted in the frame of WP2 in order to improve the devices performance.

EC devices on glass substrates were initially assembled and tested using standard liquid electrolyte in order to find out the optimum characteristics of the electrochromic layer (thickness, porosity, etc) to achieve a high optical contrast (up to 71% was reached) and a fast response (colouring in less than 7 seconds and bleaching in less of 4 seconds was achieved).







Once the key parameters were optimized, all-solid EC devices on flexible plastic substrates with final dimensions up to $5x5 \text{ cm}^2$ were successfully manufactured and fully tested in the frame of WP5.

Regarding the TOLED system, after optimizing and selecting the most suitable materials and parameters in WP2, a five-layer (ANODE/HIL/ELM/ETL/CATHODE) structured TOLED

stack with a transmittance around 75% at 550 nm (well above the targeted value) and white-light emissive area of 18 mm² was successfully manufactured by highthroughput printed methods.



Tandem devices combining the 2 technologies (electrochromism and TOLED lighting) were successfully fabricated following 2 different approaches:

-Approach 1: Two separate devices (EC and TOLED) are joint together through a transparent adhesive layer;

-Approach 2: An intermediate substrate, TCO-coated on both sides, is used for the construction. The electrochromic layers are applied on one side of the substrate while the TOLED is built on the other side.

The first one is the simplest approach while the second strategy presented some technical issues at the beginning due to incompatibilities between the fabrication procedures associated to each technology (EC and TOLED), that were finally overcome by defining an appropriate sequence of fabrication steps.



Both the TOLED and the electrochromic system operated properly in the final tandem devices, demonstrating the feasibility of the proposed fabrication method.





It is worth mentioning that AZO-patterned/PET/ITO was used as the intermediate substrate, depositing the EC layers on the ITO side and the TOLED system on the patterned AZO coating. It is also noteworthy that the novel procedure for the production of nanostructured EC coatings (the aforementioned "Route C") was found to be most suitable than the standard method (Route A) for the production of the tandem devices.

WP4-Up-scaling.

After fabricating and optimizing EC and TOLED devices of small dimensions in the previous WP, scaling-up activities were performed in the frame of WP4 with the aim of producing large area devices. Thus, the objectives were: 1) Scaling-up the production of the materials and coatings developed in WP2 to the quantities required for the preparation of targeted size prototypes. 2) The fabrication of all-solid EC devices on plastic substrates with the dimensions of a real-life aircraft window (targeted size: $30x40 \text{ cm}^2$). 3) The fabrication of large flexible TOLED devices with rectangular/stripe shapes in a first stage and functional arbitrary size/shape light surfaces in a second stage. 4) Fabrication of tandem (EC-TOLED) aircraft window prototypes (size $30x40 \text{ cm}^2$).

In the beginning of IN-LIGHT, mesoporous TiO_2 films on TCO substrates for the subsequent production of EC devices were prepared via dip-coating. However, the dip-coating procedure was considered unsuitable for the preparation of large area coatings in terms of time and materials consumption. In March 2014 (M17), a roll-to-roll (R2R) pilot coater was established at FRAUNHOFER ISC. In September 2014 (M23), the R2R pilot coater was used to deposit TiO₂ films on commercially available ITO/PET roll material.



Using the TiO₂ dispersion was straightforward for slot-die coating and the produced TiO₂ films on ITO/PET showed proper function in order to be used for the preparation of ECDs of 15x20 cm^2 (half size of prototype). Thereafter, a "Route C" coating solution was successfully applied by means of the R2R pilot coater. As it was explained, "Route C" films do not require a grafting step because the coating solution already contains viologen-modified TiO₂ nanoparticles. This innovative route very much facilitated the preparation of IN-LIGHT ECDs. In February 2015 (M28), TiO₂ films were produced (on ITO/PET roll material) to yield samples of the envisaged prototype size (30x40 cm²).

The progress towards the final electrochromic prototype size was successfully reached after optimizing several experimental parameters such as: large R2R electrode fabrication, TiO_2 film thickness, grafting time for the modification of the TiO_2 films with viologen, polymer





electrolyte over-coating, assembly and crosslink conditions (including UV lamp power, distance between the device and the lamp and the cross-link exposure time).



Flexible all-solid large area (30x40 cm²) EC devices were successfully produced based on electrochromic films processed by R2R on ITO/PET and the solid electrolyte developed in WP2 and Prussian blue films electrodeposited on ITO/PET as the counterelectrode.



A series of EC devices with dimensions $15x10 \text{ cm}^2$ and $30x20 \text{ cm}^2$ were also produced aiming at the mechanical/impact tests and the optical/environmental tests respectively to be performed within the corresponding testing campaign (WP5).

Regarding the TOLED devices, efforts were focused on enlarge the emissive area keeping the device structure previously developed. Three different R2R TOLED pilot runs were carried out. In October 2014 (M24) a TOLED module with four directly interconnected devices having the size of $1.2 \times 30 \text{ cm}^2$ (rectangular shape) was produced on ITO/PET substrates with a length of 190 metres and a width of 0.15 metres. Unlike in the case of the TOLEDs produced at labscale, a green light emitting polymer (PFBT) was selected for the production of the large area TOLEDs due to its lower price. The prototype transparency was 72% at 550 nm (targeted transparency was 65%) and provided a luminance of 273 cd/m² at 12.5V.

After the successful production of the large area interconnected modules, the main target was to produce functional arbitrary size and shape light emitting surfaces instead of rectangular/stripe shapes. It is worth mentioning that the introduction of a MAXI printing line at VTT facilitated





the manufacture of the above-mentioned TOLEDs that were successfully produced and, in overall, provided a very high yield when the layers were aligned well enough.

Pilot run	Date	Description	Outcome
1 st	04/2014 (M18)	70 m web; 1.5 cm ² , 6.0 cm ² and 10 cm ² emissive areas. Proof-of-principle	1** run10cm ²
2 nd	09/2014 (M23)	140 m web; new gravure cylinders for the printing of various single stripes, of up to 10 cm ² emissive areas.	2 nd run 5x10cm ²
3 rd	10/2014 (M24)	190 m web; interconnected TOLED modules, emissive area max. 180 cm ²	3 rd run 180 cm ²
4 th	09/2015 (M35)	300 m web; arbitrary size and shape light surfaces in the MAXI pilot line	4 th rún 88 cm ²

Thus, a very remarkable achievement within this activity was the development of a method based on R2R assembly of gravure-printed layers to create patterned and flexible light-emitting surfaces, enabling significantly larger light areas and expanding the usage possibilities of the technology. This type of light-emitting plastic film and processing in ambient atmosphere has not been created before on this scale.

For the production of large-area tandem devices, electrochromic (EC) devices with the targeted size $(30x40 \text{ cm}^2)$ were produced and interconnected TOLED modules were prepared in parallel.

The main target was to utilize two laminated and aligned TOLED elements, which had emissive stripe patterns (3 stripes in TOLED #1, 2 stripes in TOLED #2), in order to produce uniform light emissive area with the dimensions of 6 cm x 30 cm. Therefore, two one-side barrier film encapsulated TOLED elements were laminated together using a pressure sensitive adhesive (PSA). Further, the laminated TOLED element was subsequently laminated with the large area EC element using PSA. The total thickness of the laminated TOLED element was approximately 0.6 mm.



WP5-Application testing and validation.

The main objective for WP5, leaded by FADA-CATEC, was the characterisation of the 1:1 scale and other intermediate prototypes developed during the IN-LIGHT project for function diagnostic. In order to achieve these objectives, environmental tests in accordance with the operating conditions of the devices and performance characterisation for their application were carried out according to the requirements established by the consortium partners and/or end users. The WP was divided into 3 tasks:





Task 5.1 "General evaluation": In this task, the characterization of samples/prototypes developed in WP3 was performed. The characterization of the prototypes was focused on functional aspects, especially in determining the operational and destructive limits of the system in order to get confidence after performing the scaling up of the system. Furthermore, Non Destructive Tests campaign (NDT) was included to assure the correct manufacturing of the technological prototype.

Task 5.2 "Environmental testing": Within this task it was included the exposure of target devices at sample level and scaled-up components to radiation testing, temperature changes, altitude and humidity testing according to the environmental conditions established by the consortium partners and/or end users. Those studies allowed the evaluation of components performance at different thermal stress and fatigue conditions. The testing activities were performed in accordance with the specifications defined in the work plan (elaborated at the beginning of the project) to validate the range of use and limits of destruction.



Task 5.3 "Application testing": Application-oriented evaluation of fully operational prototypes according to internal and customer requirement specifications imposed on by the end-user partners. FADA-CATEC carried out low energy impact and bonding strength test. A NDT evaluation was performed as well for characterizing the severity of the applied damage. AIRBUS, CIDETEC and GKN performed optical tests on the samples according to aerospace requirements in the lab and under environmental conditions. Continuous cycling was performed by AIRBUS and determination of storage stability, self-regeneration ability and memory effect tests were executed by different partners.





Most of the requirements established by the consortium/end user have been met, except from the light transmittance requirement during the dark state of the device (even if deep colouration was reached), and the number of switching cycles supported by the electrochromic device. This aspects need to be further improved. One of the milestones achieved is the testing of full scale prototypes, apart from intermediate size devices.

It is worth mentioning that the execution of all the testing campaigns can be considered a complete success thanks to partners coordination and collaboration.

WP6- Technology and impact assessment.

The main objectives of this work package were to investigate the Environmental, Health and Safety (EHS) aspects of chemicals (e.g. polymer additives) and materials (e.g. nanoparticles) to be used according to their application in coatings / substrate and to perform the Life Cycle Assessment (LCA) and to evaluate the cost of the proposed solutions.

The EHS analysis was focused on the definition of the hazard profile and the REACH compliance of the chemicals used for the fabrication of the aircraft window integrating both the EC and TOLED technologies. A toxicological and ecotoxicological assessment of the raw materials selected in WP2 was carried out to highlight which raw materials are hazardous and for which endpoint there is concern. The REACH compliance analysis of the raw materials was performed taking into account the production processes and the final product to identify to which obligations the supply chain actors and window manufacturer will be subjected. Briefly, the results show that two of the raw materials used are identified under REACH as substances of very high concern (SVHC). However, for these two substances the identification as an SVHC does not in itself encompass any use restrictions. Among the numerous raw materials used, nine of them have toxicity. However, because during the aircraft window's life cycle, the emission of dangerous substances is not expected, human exposure to these substances is excluded.

Regarding the life cycle assessment, the boundaries were selected as a cradle to gate which means that the study was carried out from resource extraction to the factory gate. The environmental impacts associated to the materials and processes employed in the production of nanostructured electrochromic coatings by route A and route C and for the fabrication of the tandem (EC-TOLED) device were calculated by two methodologies: Eco-Indicator-99 and CML2001. It is remarkable that the impact of the new method developed in the project (the so-called route C) is lower than the impact of the traditional method (route A) in all the categories analysed. Moreover, the cost analysis revealed that the new method is also less costly than the traditional one. These aspects reinforce the innovative process for the production of nanostructured electrochromic coatings developed in IN-LIGHT.

WP7- Project management and coordination.

The main objectives of this work package were: a) to ensure that the activities in each of the different work packages (tasks, deliverables and milestones) were carried out in line with the agreed budget and timing; b) to coordinate the communication among the project's partners; c) to manage the communication with the European Commission and the respect to the reporting and financial management as detailed in the contract between the EU and IN-LIGHT consortium.

In general, the project management was efficient and well appreciated by the partners at the end of the project. Briefly, CIDETEC provided administrative support for financial management and reporting, submitting the deliverables and reports to the European Commission in time. Furthermore, CIDETEC paid special attention to promote and facilitate the interaction and collaboration between the different partners. It is worth to note that the high collaborative spirit and fluent exchange of information was crucially exploited to reach the ambitious project goals, going from the synthesis of the materials and the design of new procedures to the production of prototypes.





WP8- Dissemination and exploitation.

WP8 was dedicated to the development and realization of an effective dissemination and exploitation plan, which would provide a guideline for the major activities of the consortium as a whole, as well as of the contributions of the individual beneficiaries, in order to ensure the efficient dissemination of gained knowledge, publication of project results, and exploitation for market-relevant applications.

During the first half of the project lifetime (M1-M18), WP8 objectives were mainly focused on creating the project's visual identity, developing the primary dissemination tools (e.g. set up the project website, create posters and leaflets, presentation and poster templates), and identifying initial dissemination and exploitation opportunities. Furthermore, several dissemination activities realized during that period raised awareness upon the project and stimulated the engagement of a full range of potential end users (from the industry, research establishments and academia) to the project's progress and results.

During the second half of the project's lifetime (M19-M36), WP8 focus was accordingly placed on performing dissemination and exploitation activities following the initial plan and opportunities defined during the first half, towards keeping all potential users and IN-LIGHT "followers" updated and informed about the project, while not failing to increase the potential of market uptake and of maximum use of its results.

-The IN-LIGHT PUDF: From the first months of the project, the IN-LIGHT Plan for Use and Dissemination of Foreground (PUDF) was developed with the active contributions of all consortium partners. Being an extrovert communication methodology which considers the objectives and needs of the project, consortium strengths and external opportunities, the IN-LIGHT PUDF was continuously monitored and evolving during the entire project lifetime. The latest version of the IN-LIGHT PUDF summarizes the activities realized during the entire project duration, while it also includes some of the partners' future considerations for dissemination and exploitation opportunities, after the project end.

- Public website: Being one of the main and measurable objectives of WP8, an official website was developed during the early months of the project, in order to act as a beacon for the project's news and activities. Over the course of the project, the IN-LIGHT website (http://inlight-project.eu/), has followed the latest progress and activities, providing in this way up-to-date information to visitors, keeping up with the latest IN-LIGHT-related news (events, presentations, etc.), project progress and obtained results, performed dissemination activities and outreach actions. The IN-LIGHT website will be fully-functional and online for additional two years after the project end.

- Communication pack: IN-LIGHT leaflets/posters were distributed/displayed in different

events. Furthermore and towards the end of the project, updated versions leaflets and posters were of developed. These emphasize on the technical results and achievements of the project, showcasing its (exploitable) impact and the expertise introduced by each partner in the project. This updated communication material provides a compact end-user summary and will be used for the dissemination activities which will take place after the project's end.



- Newsletters, web-based information and press releases: Since the beginning of the project, the European Aeronautics Science Network (EASN) has been exploited as a dissemination amplifier towards communicating the project's news to the European aeronautics academia,





industry, as well as policy makers. During the second half of the project, a total of four (4) progress updates were published at the EASN Association's periodic newsletter, which has an outreach of 10,000 professionals in the aeronautics sector.

Another communication channel used for major outreach was the Transport Research and Innovation Portal – TRIP, where a permanent reference on the project was published (http://www.transport-research.info/web/projects/project_details.cfm?ID=47551, September 2014). Additionally, different press releases have been issued by different partners during the life of the project.

-Dissemination activities: The dissemination of the IN-LIGHT results was actively pursued by the entire consortium. In particular, the following actions were realized during the project

- 3 Publications in Scientific Journals
- 5 Publications in Conference Proceedings
- 2 Poster Presentation
- 12 Conference Presentations
- 1 Workshop Presentation
- 14 Seminar Presentations
- 9 Newsletter entries
- 16 leaflet distributions
- 7 IN-LIGHT poster displays
- 2 press releases
- 2 articles in press

Moreover, 5-7 scientific publications are foreseen for the next 1-2 years.

While dissemination activities aim to raise awareness upon the project's scope, methods and results, exploitation is equally important in order to fabricate ways for introducing the project's developed foreground into the commercial market, as a final end-user product/service.

Having successfully completed the activities of the IN-LIGHT project and having introduced some important novel developments, beyond the current state of the art, the partners have explored ways towards maximizing the impact of the project to their organizations through internal optimized techniques or even commercialization of products and processes.

The methodology developed within the EU funded TIPS action "Transforming research results into; innovative products and services" (http://www.transport-tips.eu/), aiming to facilitate and support consortia to identify the exploitable projects' results and guide them through the process of planning and implementing their individual exploitation strategy was applied during the last months of the project.

Potential impact and main dissemination activities and exploitation results

The key objective of IN-LIGHT is to contribute to the improvement of the passengers comfort ensuring customer satisfaction by introducing a new concept of aircraft cabin window that combines two advanced technologies: Electrochromim and TOLED lighting. The combination of these two advance technologies in the aircraft window will enable versatile home-like and/or office-like cabin environments fully controllable by the passenger with regard to his preferences.

The new bifunctional aircraft windows will serve both as transparent shading panels during day and as lighting system at night. The new EC windows will provide a better visual and thermal comfort than traditional windows by decreasing the amount of energy entering into the cabin from outside in the form of light and heat. Thus, electrochromic windows have a clear added value advantage over standard passenger cabin windows with associated shutters and over other technologies performing similar functions which are faster switching but are either less durable or do not have shade options (variable light transmittance, rather than light and dark only).

Additionally, the new aircraft window developed in IN-LIGHT will provide a ground-breaking type of ambient interior lighting inside the cabin, based on transparent OLEDs integrated in the





window. Including the windows for ambient lighting in the innovative way realized in the IN-LIGHT project may increase passenger satisfaction.

Results from the research on these smart systems will result in a real competitive advantage for European industry: strong research activity in the field of smart shading systems has been observed in USA and Japan. However, the very most of what is known about these activities deals with high cost glass-based systems produced through vacuum sputtering. The adoption by participating companies of a low-cost plastic-based product will therefore strongly increase their competitiveness and penetration in this market.

Furthermore, the industrialists' strategies clearly identify the need to move towards high added value products. Electrochromic shading devices are multicomponent products and, including the related processes and techniques, are of high added value. The added value of the products is easily demonstrable to the customer by prototype windows with a readily recognisable fast and strong colour change.

The IN-LIGHT project results will have a major impact on production technologies, especially regarding cheaper production methods of electrochromic windows for aerospace environment applications. From the technical point of view, success in the development of electrochromic nanoparticle-based formulations will represent a significant breakthrough, allowing the use of this high-performance electrochromic technology on plastic (rigid or flexible) devices for different applications in a broad range of industrial sectors.

In addition to civil aircraft (large civil aircraft, business jets and regional jets), there is also a market for cockpit windows and cabin class dividers. Electrochromic sunroofs or cockpit windows are of interest for helicopters. The aeronautics industry is showing a growing interest in the development of visors and windows that can control glare for pilots and passengers, as well as in the introduction of innovative lighting systems. Therefore, opportunities for smart electrochromic as well as OLED-based lighting systems in the aircraft market are clear and will increase in the next years.

Apart from aeronautics and aerospace, there are also a number of potential commercial markets for which the technology developed in IN-LIGHT might find application such as automotive, locomotive and building (office, hotel and home windows). These potential markets offer big exploitation opportunities and IN-LIGHT results have important economic development perspectives. The growth of smart windows markets over the next years implies that firms that produce smart coatings and their components have increasing opportunities to supply those materials to smart windows manufacturers.

Main dissemination activities:

- Evaluation of the optoelectronic properties and corrosion behaviour of Al₂O₃-Doped ZnO films prepared by dc pulsed magnetron sputtering. Journal of Physics D: Applied Physics 2014, Vol. 47. Number 48.
- Corrosion resistance analysis of AZO layers deposited by DC pulsed magnetron sputtering. Journal of Thin Solid Films 2015, Volume 594, Part B.
- Study of the optical, electrical and corrosion resistance properties of AZO layers deposited by DC pulsed magnetron sputtering. Journal of Surface & Coatings Technology 2015, 271.
- Corrosion behaviour of transparent oxide films deposited by pulsed DC magnetron sputtering. Proceedings of the International Conference on Coatings on Glass and Plastics (ICCG 10). 22nd 26th June 2014.
- Innovative bifunctional aircraft window the IN-LIGHT project. Proceedings of the 6th NanotechITALY 2013 Conference. 27th 29th November 2013.
- Innovative bifunctional aircraft window for lighting control to enhance passenger comfort. Proceedings of the 4th International EASN Association Workshop on Flight Physics & Aircraft Design. 27th 29th October, 2014.

IN-LIGHT- G.A. 314233 Final report





- Characteristics of all-solution printed transparent OLED signage and lighting panels. Proceedings of the LOPEC 2014. 24th-25th April 2014.
- Plastic electrochromic windows based on viologen-modified TiO₂ films prepared at low temperature. Proceedings of the 65th Annual Meeting of the International Society of Electrochemistry Book of abstracts. 31st August-5th September 2014.
- Corrosion behaviour of transparent oxide films deposited by pulsed DC magnetron sputtering. Poster presentation at the International Conference on Coatings on Glass and Plastics (ICCG 10), Dresden, Germany; 22nd 26th June 2014.
- Study of the optical, electrical and corrosion resistance properties of AZO layers deposited by DC pulsed magnetron sputtering. Conference presentation at the Nanosmat Conference, Dublin, Ireland; 8th 12th September 2014.
- Corrosion resistance analysis of AZO layers deposited by DC pulsed magnetron sputtering. Conference presentation at the 5th International symposium on transparent conductive materials, Crete, Greece; 12th – 17th October 2014.
- TiO₂/AZO bilayer thin films by magnetron sputtering as transparent electrodes for electrochromic devices. Conference presentation at the Nanosmat Conference, Manchester, UK; 13th 16th Sept 2015.
- The influence of target erosion grade in the optoelectronic properties of AZO coatings growth by magnetron sputtering. Conference presentation at the Nanosmat Conference, Manchester, UK; 13th 16th Sept 2015.
- Innovative bifunctional aircraft window for lighting control to enhance passenger comfort. Conference presentation at the 4th International EASN Association Workshop on Flight Physics & Aircraft Design, Aachen, Germany; 27th 29th October 2014.
- Plastic electrochromic windows based on viologen-modified TiO2 films prepared at low temperature. Conference presentation at the 65th Annual Meeting of the International Society of Electrochemistry, Lausanne, Switzerland; 31st August – 5th September 2014.
- IN-LIGHT: Innovative bifunctional aircraft window for lighting control to enhance passenger comfort. Conference presentation at AERODAYS 2015, London, UK; 20th-23rd October 2015.
- Roll-to-roll Manufacturing of Printed OLEDs. Conference presentation at the SID Display Week 2013, Vancouver, Canada; 19th 24th May 2013.
- Fully roll-to-roll printed PLEDs for signage, display & lighting applications. Conference presentation at the International Conference on Flexible and Printed Electronics 2013, ICFPE 2013 Jeju Island, Korea; 11th – 13th September 2013.
- Characteristics of all-solution printed transparent OLED signage and lighting panels. LOPE-C 2014, Munich, Germany; 26th -28th May 2014.
- Printed transparent cathode structure. Conference presentation at the Korean Society of Mechanical Engineers, Jeju Island, Korea; 24th 25th April 2014.
- Printed transparent cathode structure. Conference presentation at Cambrios company, presented at LOPE-C 2014, Munich, Germany; May 2014.

Main exploitable results:

- 1) AZO (aluminium dopes zinc oxide) coated plastics; Commercialization of the AZO coatings on plastic substrates developed during the project. Owner: TEKNIKER.
- Roll-to-roll processing of thick mesoporous titanium dioxide films; Deposition of mesoporous titanium dioxide film on flexible film substrates or metal foil by means of roll-to-roll techniques, followed by drying/densification steps at low temperatures. Owner: FRAUNHOFER ISC.
- 3) Synthesis of a new viologen suitable for the modification of coatings on plastic substrates; Synthesis of a blue viologen suitable for the modification of coatings (based on Result 2) on ITO/PET substrates enabling the subsequent fabrication of flexible plastic electrochromic devices offering colorless-to-blue switching. Owner: CIDETEC.





- 4) New method for the production of viologen-based electrochromic nanoparticles; Hydrothermal processes used to create nanoparticle dispersions with functional moieties, such as electrochromic dye, enabling a one-step preparation of viologen-based electrochromic coatings and corresponding devices, for instance by dip-coating on rigid (glass) substrates. Owner: FRAUNHOFER ISC/CIDETEC.
- 5) Flexible electrochromic cell; Deposition of viologen-modified titanium dioxide film (based on Result 4) on flexible film substrates by means of roll-to-roll techniques, followed by drying/densification steps at low temperatures, enabling a one-step preparation of viologen-based electrochromic nanostructured coatings on flexible film substrates and corresponding devices. Owner: FRAUNHOFER ISC/CIDETEC.
- 6) Processes for producing a colourless electrochromic film; High bright state transmittance and full colourlessness in the bright state of an electrochromic device is crucial for a number of application. The result concerns a combination of a carrier film and functional dyes producing a colourless electrochromic film. Owner: FRAUNHOFER ISC/CIDETEC.
- 7) Electrochromic element based on the viologen-modified nanostructured coatings, a complementary responding counter electrode, and a polymer electrolyte. Owner: FRAUNHOFER ISC/CIDETEC.
- 8) Hybrid Electrochromic-OLED devices for comfort and light modulation. Owner: Fraunhofer ISC / CIDETEC / Hydro-Quebec/VTT/TEKNIKER.

Project website and contact details:

Project website: http://inlight-project.eu/

Relevant contact details: Ana Viñuales (<u>avinuales@cidetec.es</u>) Jon Lacunza (<u>jlacunza@cidetec.es</u>)