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# Microstructuring technology for large and cylindrical receivers for concentrated solar plants (CSP)

*M. Hochedel<sup>1</sup>, M. Bichotte<sup>1,2</sup>, F. Arnould<sup>1</sup>, F. Celle<sup>1</sup>, C. Veillas<sup>1</sup>, T. Pouit<sup>2</sup>, L. Dubost<sup>2</sup>, T. Kämpfe<sup>1</sup>, O. Dellea<sup>3</sup>,  
N. Crespo-Monteiro<sup>1</sup>, Y. Jourlin<sup>1\*</sup>*

<sup>1</sup>Laboratoire Hubert Curien UMR CNRS 5516, University of Lyon, 18 Rue Professeur Benoît Lauras, 42000 Saint-Étienne, France

<sup>2</sup>IREIS-HEF, 7 rue Salvador Dali, 42000 St-Etienne, France

<sup>3</sup>CEA/LITEN, Laboratoire d'Innovation pour les Technologies des Energies Nouvelles et des nanomatériaux, F-38054 Grenoble, France

\*Corresponding author

yves.jourlin@univ-st-etienne.fr

**Abstract:** This paper presents an innovative fabrication process of microstructured spectrally selective solar absorbers for concentrated solar power (CSP) plants. The authors describe suitable technologies and methods to microstructure large areas and non-conventional substrates, such as tubes, and substrates with large surface areas typically used in CSP plants. Specific colloidal lithography combined with layer deposition makes it possible to structure this type of substrate with a submicron periodicity within a hexagonal arrangement. Appropriate techniques, e.g. Langmuir-Blodgett deposition and customized UV insolation were developed to process 80 mm diameter and 300 mm long tubes and 300 x 300 mm<sup>2</sup> planar substrates. Absorbing and anti-reflection coatings were deposited using industrial vacuum deposition equipment with three different deposition sources: PECVD, magnetron sputtering and cathodic arc deposition. The design and assessment of the efficiency of the microstructure absorbing layers on smaller planar substrates are described elsewhere, assuming that optical performances are close to the large and tubular substrates. The technological processes and the resulting demonstrators are described in detail herein.

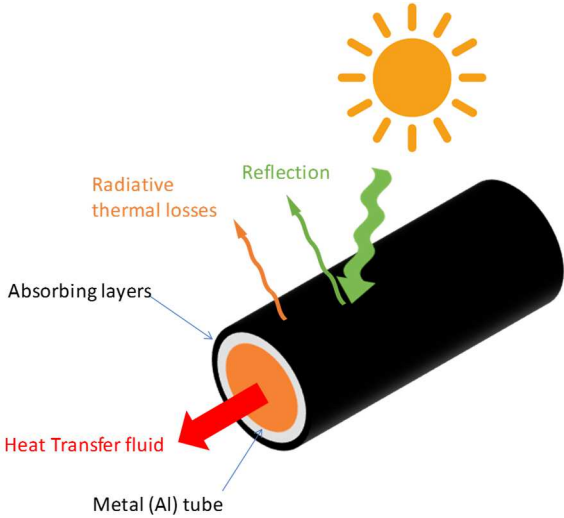
**Key words:** Colloidal Lithography, Microstructuring technology, spectrally selective absorbers, CSP

## 1. Introduction

Part of the solution to global warming and energy saving is the use of renewable energy. Among these renewable energies, concentrated solar power (CSP) technologies have excellent development potential. CSP plants could occupy a significant market position thanks to their improved efficiency, lower operating costs and good scale-up potential [1] [2]. However, industrial developments remain difficult due to both their efficiency and yield as well as cost developments and cost energy production.

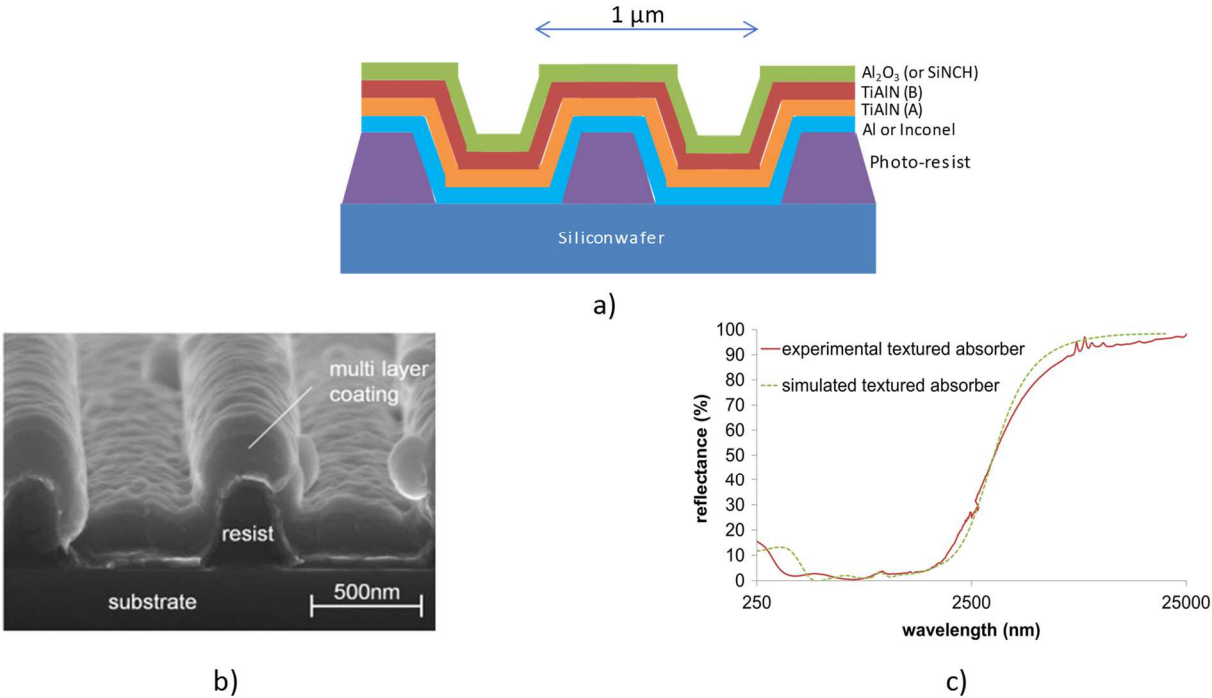
CSP technologies are based on the principle of converting solar radiation into heat. CSP can be used to generate electricity using a turbine in a thermodynamic cycle, or directly as an industrial process heat system. The absorber layer is the part of a CSP power plant that converts photons from sunlight into heat by absorption. An efficient absorber must have a high absorption coefficient in the wavelength range of the solar spectrum while limiting thermal losses due to reflection and radiative thermal losses as illustrated in Fig. 1. The main challenge is to design highly efficient solar absorbers that enable the CSP to produce a high yield. The geometry and technology used for the solar absorbers varies depending on the solar plant concerned and its operating temperature. Today, the different types of solar plant technologies installed include parabolic trough collectors (PTC), solar power towers (SPT), linear Fresnel reflectors (LFR) and parabolic dish systems (PDS) [1]. Among these technologies, tubular absorbers are very promising. They are composed of metal tubes in which a heat transfer fluid circulates. An absorbing layer covers the surface of the tube to increase photo-thermal conversion, and the absorber fits inside a vacuum glass to avoid heat loss by convection. The vacuum also extends the life of the absorbent coating by protecting it from oxidation. However, at high operating temperatures (above 500 °C), this type of absorber is subject to thermal losses due to radiation mainly in the infrared (IR) wavelength, which results in a reduction in photothermal efficiency.

In this context, spectrally selective absorbers are used to limit thermal losses at high operating temperatures. Indeed, there is a practical advantage in improving the spectral absorption characteristics of the absorber by optimizing its interface with high reflectivity in the IR domain in order to limit emissivity while keeping high absorbance in the solar spectrum.



**Fig.1.** Scheme of tubular absorber behavior

There are several ways to obtain spectrally selective absorbers for CSP or anti-reflection coating for PV [3] using microstructured surface. In CSP, surface texturing combined with thin multilayer coating is the most promising approach [4,5]. Bichotte et al. [4] demonstrated that it is possible to combine the two effects. Their aim was to create spectrally selective absorbers composed of four layers deposited on periodic microstructures using interferential lithography. The optimized design is described in Fig 2a. Fig 2b is the cross section of the fabricated and tested structure. The experimental results showed absorption over the whole solar spectrum increased from 95% to 96.6% with microstructured coatings compared to the ones with unstructured coatings (Fig. 2c). The CSP yield was thus increased by 1.3%, which is significant relative to the cost of energy production using solar plants.



**Fig. 2.** Structure design (a); Cross section of the microstructured absorber(b); Experimental and simulated reflectance of the microstructured absorber (c) [3].

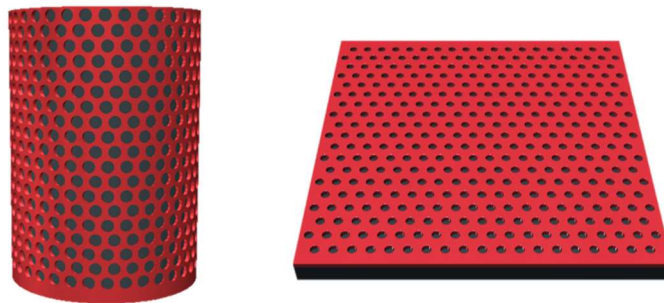
Here, our aim is to present different technological processes that can be used to produce microstructured spectrally selective absorbers compatible with real size tubular receivers (for example 2.57 m in length and 7.6 cm in diameter) for CSP technologies such as linear Fresnel plants and PTC plants. Metal based substrates are used for these type of absorbers [6], since metals (mainly aluminum based alloys) are excellent IR reflectors and can consequently reduce emissivity. To microstructure these substrates, for each step, appropriate methods have to be used that can be adapted to non-conventional substrates and large surface areas. Since laser interferential lithography (LIL) cannot be used to structure substrates with varying geometries, colloidal lithography [7] was chosen and turned out to be one of the most promising techniques for homogenous microstructuring. The absorbing layers are deposited by vacuum deposition, which is already widely used in the production of selective solar absorbers. The optimized structure incorporating the stacking of absorbing layers and the 2D microstructuring parameters were identified in a previous study on 50 mm diameter planar substrates [4]. The aim of the present study was to transpose the design to tubular substrates and to large surface areas that can be used in CSP.

The paper is organized according to the process flow with two main sections, that are the layer microstructuring process with the colloidal lithography and the absorbing layers process adapted to large and tubular substrates.

## 2. Colloidal lithography based microstructuring

The first step in the production of these large area absorbers is 2D microstructuring on the substrate. Metal substrates have already been shown to be the most suitable for this purpose [6]. Consequently, a 300 mm long metal tube with a diameter of 76 mm was used. For the planar demonstrator, we chose to use standard 300 mm wide and 400 mm long glass substrates. Based on the optimized 1D microstructure (Fig. 1a), a periodic structure with a 2D hexagonal arrangement of  $1\mu\text{m}$  period was chosen in order to be polarization independent. Colloidal lithography which imposes this 2D arrangement with  $1\mu\text{m}$  diameter microspheres is thus well suited. It is known that when microstructures are within the range of the selected spectrum, periodic microstructures act like an anti-reflection coating (ARC) [8]. The expected microstructure for each type of substrate is illustrated in Fig. 3.

Structuring the absorbers using colloidal lithography happens in several steps, including photoresist and microsphere deposition, and UV-exposure of substrates. Here, these processes were developed for large areas and non-conventional substrates.



**Fig. 3.** 3D illustration of a metal tube and an Inconel plate, with the expected hexagonal microstructuring of the photoresist (red layer).

### 2.1. Photoresist deposition

MICROPOSIT S1805 positive photoresist was used for microstructuring. Photoresist deposition on the plate was performed by spin coating using a large area dip-coater (model RC22 of Suss Microtec), able of depositing on substrates up to 22 inches in diameter. The non-diluted S1805 photoresist was deposited at 3000 tr/min for 60 seconds producing a homogeneous 600 nm thick layer over on the entire surface of the substrate.

For the long tube, the dip coating method was used to obtain a uniform photoresist layer. A customized dip coating set up was developed using a pulley fixed to a motor to enable us to keep the linear draw constant. A customized system was machined to hold the tube without damaging it when it was removed from the photoresist tank, as shown in Fig. 4. A slow drawing speed, below 10 mm/s was necessary to obtain a homogeneous layer on the tube. To this end, the S1805 photoresist was diluted 50% with ethyl lactate to obtain a thin film at this pulling speed. The authors assumed that with these dip coating parameters, photoresist thickness was estimated in the range of hundreds of nm, assuming that it is close to the conditions of smaller commercial dip-coaters with the diluted photoresist.

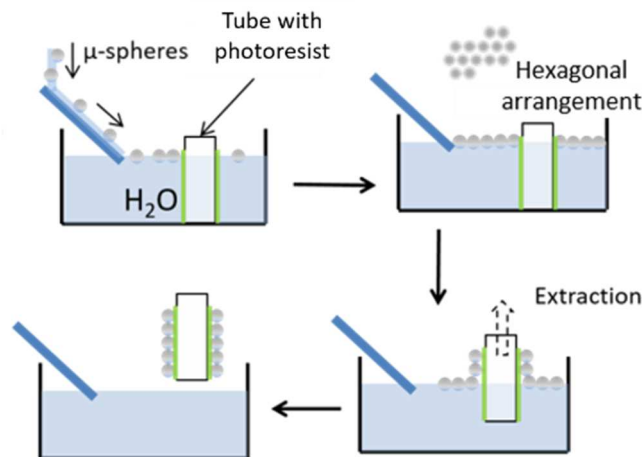


**Fig.4.** Dip coating pulley system used to deposit the photoresist on the tubular substrate.

The substrates were then baked in the oven at 60 °C for three minutes. This annealing allowed partial evaporation of photoresist solvents.

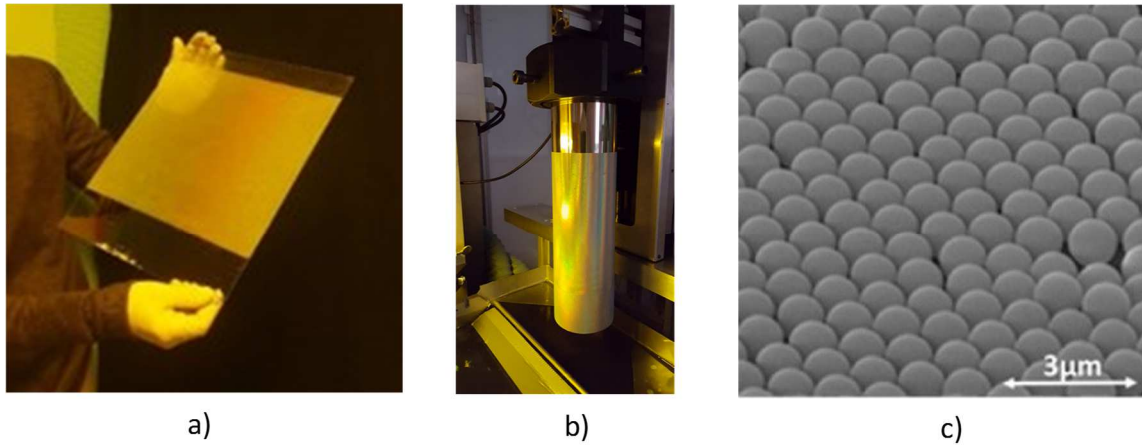
## 2.2. Microspheres deposition

The Langmuir Blodgett (LB) technique [8] was used to deposit 1  $\mu\text{m}$  silica microsphere arrays using a Boostream® machine [10] as illustrated in Fig. 5. The silica spheres were suspended in a solution of butanol and dispersed in an ultrasonic bath. They were then injected onto the surface of the water where, thanks to hydrodynamic forces, they formed a compact hexagonal film on the surface. The quality of the self-assembly of the beads in the transfer area was controlled by a camera connected to custom software to visualize the arrangement of the microspheres in real time. When the software detected the compactness of the bead assembly had reached optimum, a preliminarily immersed photoresist-coated substrate was carefully withdrawn from the tank and the microsphere monolayer transferred onto the substrate, according to quasi-perfect hexagonal arrangement.



**Fig. 5.** Successive steps of microsphere deposition: injection of microspheres.

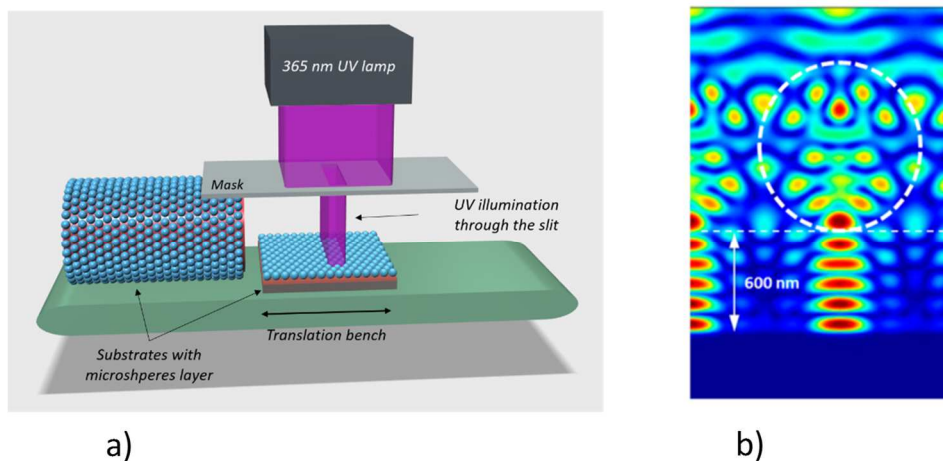
Thanks to the electrostatic forces between each microsphere during the substrate extraction step, the microspheres retained their hexagonal arrangement and their cohesion whatever the geometry of the substrate, that is flat (Fig. 6a) or cylindrical (Fig. 6b). The crystallographic orientation of this hexagonal pattern remained constant (Fig. 6c) over large areas i.e. areas of several hundred micrometers [11,12]. Even though this uniformity is not preserved on the overall of the 300 x 400 mm substrate (and along the cylinder), scattering losses are reduced and the effect is negligible.



**Fig.6.** Deposition of 1- $\mu\text{m}$  diameter silica microspheres by Langmuir Blodgett method on the planar substrate(a) and the tube(b); SEM image of the bead monolayer(c).

### 2.3 UV illumination

A special piece of equipment was created for UV illumination during the process, using a UV LED lamp (UWAVE equipment) at a wavelength of 365 nm. During UV irradiation, the power lamp was adjusted to its minimum, i.e.  $270 \text{ W}\cdot\text{cm}^{-2}$ . The incident beam size was reduced using a homemade mask thus making it possible to illuminate the substrate and the tube with a homogenous beam line of 140 mm long and 5 mm wide. The UV lamp was fixed to a suspended holder that enabled the substrate to be moved under the light source and exposed (Fig. 7a). UV exposure is not static but dynamic as the sample moves during illumination. Indeed, by varying the speed of movement during the illumination, the exposure power on the sample can be well controlled and adjusted. For the flat substrate, the illumination was homogenous all along the width of the substrate (perpendicular to the direction of the displacement). Only one dynamic illumination was necessary. For the cylinder, several UV illuminations were necessary to cover the whole area of the cylinder using a rotary stage allowing an adjusted rotation angle after each displacement. This dynamic exposure is possible because the microspheres are fixed to the substrate, which is one of the advantages of colloidal lithography.



**Fig.7.** Scheme of the dynamic UV insolation system adapted to large area and non-conventional substrates (a); Electromagnetic field amplitude distribution (b), calculated using the RCWA method, over  $xOz$  plane passing through the center of a  $1\mu\text{m}$  diameter microsphere, under a 365 nm wavelength UV lamp [10].

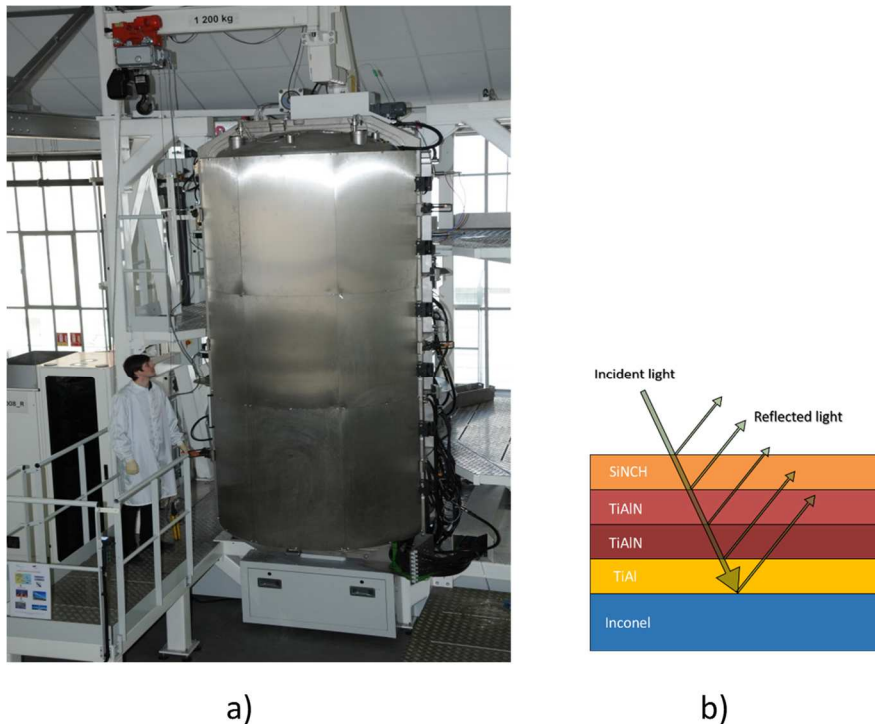
Each microsphere in the monolayer behaved like a micro-lens by focusing incident UV light and created a 600 nm long photonic nanojet emerging from the sphere in the photoresist layer with subwavelength transverse dimensions and low divergence (Fig. 7b). The RCWA method has been used to study electromagnetic field propagation [12]. Standing waves appear in the photoresist layer due to back reflection of the metal substrate. After development of photoresist, this effect was reduced thanks to an adapted photoresist layer thickness to reduce the effect of standing waves. The photoresist thickness was chosen to be far from an entire number of fringes of the fringes pattern created in the photoresist layer. In addition, one knows that the effect can be considerably reduced using a Bottom Anti-Reflection Coating (BARC).

After UV illumination, the samples were developed to reveal patterns in MF319 developer containing 2.2% Tetramethylammonium hydroxide (TMAH). The insulated zones were dissolved during this development step. However, the development time was extremely short (between 3 and 5 seconds), so the process was not suitable for large substrates. Consequently, it had to be adapted to leave sufficient time to handle the samples properly in suitable containers both for the tubular and the large planar substrates. The development time was between 20 and 25s with the diluted MF 319 developer. In-situ control of resin development was carried out on 2-inch substrates to calibrate the optimal development time to be applied for large substrates.

### 3. Absorbing multilayer coatings

#### 3.1. Layers deposition

Steel plates (30 cm x 40 cm) and tubes (30 cm long), on which a microstructure was created were then coated with selective absorber, consisting in a multilayer. The selective absorber coating demonstrators have been fixed in a large industrial TSD 2800R (Fig. 8a) at HEF Group. This vacuum deposition machine is used as a pilot for future industrial projects. It is equipped with a magnetron sputtering cathode, microwave plasma sources and cathodic arc sources. The deposition sources are installed on the vertical wall of the vessel to cover a coating length of 2.8 meters. The substrates are fixed in a rotating stage in the center of the machine in order to perform homogenous layers on planar or cylindrical substrates. The coating is made of four main layers (Fig. 8b) comprising the absorbing layers (TiAlN) and the anti-reflective layer according to the design (Fig 2.a). In the first step, the function of the layer made of titanium aluminum nitride is to absorb solar radiation. A reactive magnetron sputtering process was developed for this application based a TiAl target sputtered in a reactive atmosphere of argon and nitrogen.

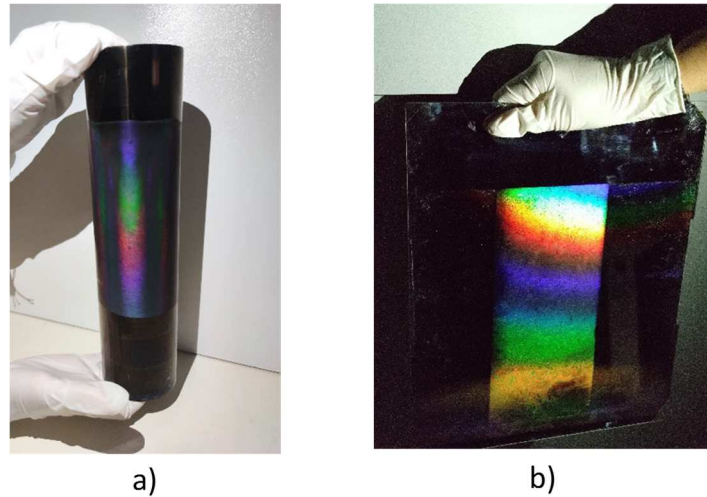


**Fig. 8.** Photo of the huge deposition machine (a) (courtesy of HEF IREIS); Scheme of the structure of the selective absorber (b).

The last layer has to act as an antireflective. This SiNCH layer is obtained from a PECVD process using microwave sources. A TetraMethylSilane evaporator feeds the equipment with a mixture of argon and nitrogen to deposit the SiNCH layer. The selective coating process was validated using the thermo-optical characterization and aging tests reported in [13].

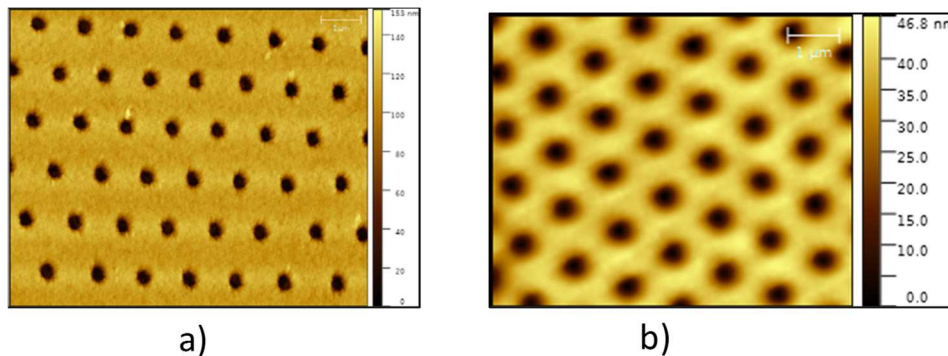
#### 3.2. Demonstrators

The absorber demonstrators obtained at the end of the process appeared to be homogeneous over the entire surface, both for the tube (Fig. 9a) and for the large planar substrate (Fig. 9b). Even though they appear as a black coating, indeed, the diffraction of light in the visible range exists since the period of the structure is in the range of the visible wavelength range.



**Fig. 9.** Microstructured and coated demonstrators. Inconel tube (a); stainless plate (b).

AFM measurements revealed homogenous hexagonal gratings on the glass square plate before and after absorbing multilayer coatings (Fig. 10). The AFM profile identified a periodicity of about 1  $\mu\text{m}$  which is imposed by the 2D hexagonal pattern of the 1  $\mu\text{m}$  diameter spheres. It is thus constant on the whole area of the tubular and planar substrates. The depth of the microstructure was measured close to a depth of 300 nm.



**Fig. 10.** AFM images on the square glass substrate of (a) S1805 photoresist layer structured by colloidal lithography and (b) gratings after deposition of absorbing multilayers.

For these demonstrators, no etching process has been carried out since the photoresist layer haven't been removed or been used as mask for further etching process. SiNCH layer is a very stable thermal, mechanical and chemical layer and the authors demonstrated that we could kept the residual photoresist layer on the large substrates provided that this layer was deposited on the substrate in the groove of the photoresist grating for its adhesion to the substrate.

#### 4. Conclusion

The authors have demonstrated that it is possible to combine microstructuring and layer deposition on large planar and tubular substrates, particularly those used for CSP receivers. This was achieved thanks to dedicated colloidal lithography, both at the level of microsphere deposition (with a unique Langmuir Blodgett based machine) and UV illumination, as well as deposition of specific layers in a huge vacuum deposition chamber. The simulations and optical characterizations of the resulting structures were conducted on standard substrates and have already been published elsewhere. Colloidal lithography is completely up-scalable as soon as microspheres layer deposition is achievable on non-standard substrates. Only AFM measurements on large substrates have been carried out, but the authors assumed that comparable microstructured layers have been achieved and reproduced on the tubular and large substrates. Given that these results are applicable to both large and non-planar substrates, we showed that this technology can be implemented to fabricate high performances microstructured solar absorbers with similar dimensions to those currently installed in CSP plants. This study demonstrates the adaptability of fabrication processes used both for microstructuring using colloidal lithography and for vacuum deposition of absorbing layers.



## Acknowledgments

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