NANOSCALE ENHANCED CHARACTERISATION
OF SOLAR SELECTIVE COATINGS

PROJECT FINAL REPORT

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Final publishable summary report

Executive summary

The Concentrated Solar Power (CSP) technology based on parabolic trough solar collector for large electricity generation purposes is currently the most mature of all CSP designs. The current parabolic trough design deals with a maximum operating temperature around 400ºC in the absorber collector tube but some recent designs are planned to increase the working temperature to 600ºC to increase the performance. These systems are expected to be working during 20-25 years.

However, there is a great lack of knowledge about the performance of the selective absorber coatings of the collectors during the whole life of the receiver. The main idea behind the NECSO project is to provide tools to the solar plants builders, to guarantee that the selective coating will work properly during 20 to 25 years. Thus, novel experimental methods for testing materials under extreme conditions will be developed in the project and nanoscale characterisation of the nanostructure parameters are correlated with coating performance. The resulting outcomes are expected to contribute significantly to the infrastructure of the solar energy research, development and industrial activities worldwide.

The two main objectives of the NECSO are:

1.- To correlate nanoscale structure related requirements (nanoroughness, nanohardness, crystallography, composition, vibrational spectra...) with performance requirements: optical and, more important, life expectancy.

2- To develop characterization and degradation protocols to serve as a powerful tool for coating developers, producers and end users for life prediction to push the collector parameters (temperatures and environments) to higher efficiency parameters.

Thus, during the project testing equipment, protocols and models for lifetime assessment have been developed in order to assess the expected lifetime of the solar selective coatings for high temperature parabollic through CSP applications. Different combinations of temperature and gas contents have been tested and mechanical and tribological properties of the coatings have assessed. A isoconversional kinetic model has been set up in order to assess the life ime of the coatings. To this purpose the degradation of the solar selectivity values under different loads was used as input dat for the model. Moreover, new degradation indicators based on changes of the vibrational bands obtained by spectroscopy techniques have been identified and used for the prediction of lhe lifetime of the coatings.

Another important issue is that equipments and methodology to perform ageing tests directly on cylindrical samples as well as to test cuantitavely the mechanical properties and solar selctivity values on this kind of geometry. these results are of great interest for the CSP industry as the testing and characterization can be done directly on real tubes avoiding the use of laboratory flat samples.

These protocols are being promoted to become standards for the CSP industry. On the other hand, the main mechanisms of degradation of the solar selective coatings have been identified and analyzed in order to help coating developers to improve their formulations for higher performance coatings

Summary description of project context and objectives

Optical coatings are commonly deposited over large areas on different substrates: glass, metals (steel, aluminium...) or polymeric foils (PET...). Production processes involve normally large machinery including many times roll-to-roll processes to deposit multilayers over several square meters of substrates. However, **performance of these coatings depends strongly on nanometric properties**: composition, crystallography, nanostructure, roughness, homogeneity... **Solar selective coatings** are considered a special case of optical coatings combining several layers with different properties, mainly: antireflection, solar absorptance and infrared mirror.

Nowadays the most demanding solar selective coatings are those used in tubes of high temperature parabolic trough solar collectors. Coatings have to operate in an **aggressive environment** (temperatures above 400ºC, thermal cycling) during 20-25 years. Homogeneity of the films along the whole distance of the tube and in its perimeter is critical to ensure a good performance of the coating. It is estimated that thickness should be controlled up to an accuracy of 5 nanometers (over the complete surface, 1 m^2 in 3D) to ensure a correct function of the multilayer. Besides, further developments require higher temperatures, improved scratch resistance and working under oxidant atmospheres (small quantities of water vapour and oxygen). In order to get significant advances in this field it is essential to have:

1. Nanoscale structure related requirements (nanoroughness, nanohardness, crystallography, composition, vibrational spectra...) and the correlation with performance requirements: optical and, more important, life expectancy.

2. Standard characterisation and degradation protocols to serve as a powerful tool to coating developers, producers and end users for life prediction and to push the collector parameters (temperatures and environment) to higher efficiency parameters.

Optical parameters of coatings (absorptance and emissivity) are relatively easy to measure on samples just after the deposition process. It is more complicate to characterise the evolution with time of these values in a relatively short period of time to ensure the proper performance of the coating during 25 years. And this is one of the aims of the NECSO project. But what is really critical is to predict the life expectancy (catastrophic failures, corrosion, delamination...) and nowadays these components are normally only guaranteed for 2 years. For this reason it is important **to have standard ageing protocols and link this information with the nanostructure and nanomechanical properties of these coatings.**

The timescale of the project (24 months) could be insufficient to assess and model the performance of any material for 25 years. For this reason, the broad previous experience of the consortium in this subject will be of utter importance. The collaboration with the standardization group AEN/CTN 206/SC /GT2 will be very valuable as well (see the section “standardization” underneath). It is worth mentioning the key role of **ARIES**, a Spanish **SME**, **solar plant builder** that also participates in their **exploitation** of those solar plants. Besides, correlation of nanoscale parameters with the performance of full scale collector (WP8) will be very valuable to extract useful information. Other 2 SMEs specialized in characterisation equipment and techniques are involved in the project.

Although the objectives of this NECSO project are focused in solar selective coatings much of the work and **the conclusions could be extrapolated or useful for a wide range of optical coatings**. Nanostructure based performance and ageing studies (protocols) could be adapted to a wide range of working conditions in different applications: solar mirrors, architectural glass coatings, antireflective coatings for photovoltaic modules, solar selective coatings for “low” temperature domestic collectors, coatings for electric ceramic glass cooktop and ovens, coatings for vehicle windows, for touch screens, light emitting diodes on glass or on metallic substrates, etc.

Scientific and Technological Needs

The world increasingly turns to renewable energy in order to maintain a sustainable standard of living, with solar energy being the largest global renewable resource available. CSP technologies offer access to this vast solar resource, with a proven track record of two decades on industrial scale, and potential breakthrough paths toward **higher operation temperatures**, higher conversion efficiency, thermal storage, and emerging offshoots such as solar chemistry and solar fuel generation. CSP can play a central role in the future mix of renewable energy supply. However, realizing this promise requires major advances in the science and technologies of high-temperature photothermal conversion.

The CSP technology based on **parabolic trough solar collector** for large electricity generation purposes is currently the most mature of all CSP designs in terms of previous operation experience and scientific and technical research and development. As a result, this technology has led the way to a world-wide extended implementation of CSP technologies: more than 4.7 GW are currently operating and more than 10 GW are estimated (in a conservative scenario according to CSP today) in the next 5 years. This means to have around **12000-16000 km of solar receivers**. The current parabolic trough design deals with a maximum operating temperature around 400ºC in the absorber collector tube but some recent designs are planned to increase the working temperature to 600ºC increasing the performance by 5-10% (when classical plants are around 12-14%) as this is considered to be the only way to attain the improved productivity that the market demands. These systems are expected to be working during 20-25 years.

However, there is a great lack of knowledge about the performance of the selective absorber coatings of the collectors during the whole life of the receiver as the new designs have not being working for too long. Even more, **it is necessary to establish characterization methods and standard protocols to guarantee the performance, durability and quality control and in order to get further advances in the development of high temperature** coatings. **It is also very important to design accelerated degradation and ageing protocols to test the evolution of these coatings under these new, more aggressive conditions in the newer designs**: higher temperatures combined with a low (but very damaging) level of molecular oxygen and water vapour. According to The Solar Power Generation Market Outlook the short operating life of CSP technologies compared to solar photovoltaics could be a key resistor to its growth.

The main idea behind this NECSO project is to provide tools to the end users, namely solar plants builders, to guarantee that the selective coating will work properly during 20 to 25 years. Novel experimental methods for testing materials under extreme conditions (temperature and radiation) are needed providing a deeper understanding of the interaction of electromagnetic radiation with nanomaterials, as basis for design of new spectrally selective absorber coatings. Nanoscale characterisation correlates the nanostructure parameters with coating performance. The resulting outcomes are expected to contribute significantly to the infrastructure of the solar energy research, development and industrial activities worldwide. Additionally, the designed testing protocols should help coating developers to compare available layers and newly designed ones, with standard procedures.

In some new designs tribological issues are also very important. ARIES receiver tube presents several characteristics that require a better understanding of the performance of the selective coating, which is exposed to challenging boundary conditions. The receiver is designed to operate under medium temperature applications (<400º C) or at higher temperature applications (<600º C) depending on the Heat Transfer Fluid to be used, so it is necessary to have a selective coating that could withstand the whole range of operating temperatures presenting adequate absorption/emission characteristics. The axial thermal expansion of the inner steel tube is free from the glass tube, but slips with marginal friction throughout the entire semi Solar Collector Assembly (SCA, 75 m) and along of several pieces specifically designed to suspend the steel tube. The contact between those pieces and the coating could damage the coating itself, so some of key points to be analyzed in deep regards the tribological contact between both. So, the tribological performance of the selective coating has also to be assessed. It is critical to know if the wear scar will promote a further degradation of the coating. In this design the receiver directly integrates the vacuum maintenance system, enabling pressure control, with vacuum pumps and pressure sensors. The receiver is designed to operate at high pressure levels (from 10^{-3} mbar to 10 mbar) including the potential use of insulating gases. **The selective coating is therefore exposed to different gas atmospheres**, since some of the marginal products from outgassing (typical process of vacuum chambers) and permeation can be found in the system, as well as other gases to be applied. Oxygen, water vapour and potentially hydrogen are present in the vacuum chamber in different amounts. It is necessary to exhaustively analyze the impact of these gases on the selective coating.

On the other hand, **it is of utter importance to assess the evolution of the nanostructure of the thin films and the nanoroughness of some of the interfaces that affect absorptance due to aging of the coating during service in these harder conditions**. In particular, the size, form and distribution of the metallic nanometric islets inside the dielectric matrix of the cermet is a critical parameter in the optical performance of this absorbent layer of the coating, and one that could be quite easily modified by the prolonged action of temperature. Nanoroughness, mainly in the surface of the metallic thin film acting as an infrared mirror, is also a key factor affecting absorptance and emissivity and it could be modified during service.

In this context the main goals in NECSO project are the following:

1. To develop accelerated ageing testing protocols and models for lifetime prediction of the solar selective coatings to be promoted as standards for the solar industry.
2. To obtain information on the degradation mechanisms of the solar selective coatings in order to help coating designers in further developments targeting better optical performance and durability, even under more aggressive working conditions.

To achieve these goals the following tasks have been addressed:

-Development of the ageing protocol: to analyze the thermal cycling with different gases.

-Analysis of nanoscale structure and requirements: the composition, nanoroughness and crystallography.

-Mechanical properties characterization: Study of the thermal stability, adhesion, nanohardness, and wear.

-Degradation analysis by dry and wet corrosion and the possible synergistic impact of vibration.

-Study of the optical properties and its evolution.

Consortium

In order to achieve the objectives in NECSO the consortium is composed by entities from 5 different countries (Slovenia, Switzerland, Belgium, France and Spain): 3 industrial partners (SMEs) and 4 researching groups (research centres or universities).

The consortium has high industrial participation with 3 small and medium companies (ARIES INGENIERÍA Y SISTEMAS, TECNOVAC, CSM instruments) highly concerned with the research activity as strategic lines for their futures. These companies are supported by 4 research centres and universities (IK4-TEKNIKER, Kemijski Institut, Katholieke Universiteit Leuven and CENTRALE RECHERCHE SA) experts in the areas developed in the project.

Description of the main S&T results/foregrounds

1. Working conditions

Initially the working conditions of the coatings were established by ARIES. Two different regimes were chosen. The first one at medium temperature corresponds to the current state of the art technologies already working in commercial plants. The second one at high temperature is for new concepts as the developed by ARIES in the HITECO project.

Medium Temperature: Typical working temperature with a range between 20 ^{o}C and 425 ºC

Work conditions in the internal atmosphere

- Typical composition in the vacuum chamber: ≈ 10^{-4} mbar of 0_2
- Worse case of composition in vacuum chamber: 0,2 bar of 0_2
- Little presence of hydrogen inside the chamber

High Temperature: High working temperature case with a range between 20 ^{o}C and 625 ºC

Work conditions in the internal atmosphere

- Typical composition in the vacuum chamber: < 10^{-2} mbar of 0_2
- Worse case of composition in vacuum chamber: 0,2 bar of 0_2

The expected working life of the coatings is 25 years.

Optimal optical values for the selective coating are:

- a: (integrated solar absortance AM1,5) >96
- e: (emissivity in the IR) < 0,09 at 400 ºC
- e: (emissivity in the IR) < 0,13 at 580 ºC

2. The solar selective coatings

2.1. Sputtering system

An in-house developed PVD sputtering system was used to deposit the solar absorber coatings. The system was designed and manufactured to coat 4 m long tubes which is the current standard size in the industry. The chamber is equipped with 4 magnetrons to deposit 4 different metals and reactive sputtering is used to deposit nitrides and oxides. Figure 1 shows a general view of the equipment. The magnetrons are located at in the central module were the coatings are deposited. The lateral modules are used to provide space for the 4 meter long tubes. Thus, the tube is located at one end of the equipment and moves with linear motion and rotation towards the other end of the chamber going back and forth from one end to the other and its coated while passing through the central module.

The sputtering evaporators have an unbalanced magnetic field to improve coating properties. The evaporators are supplied by a Pulsed-DC power system with arc-handling to avoid the microarcs. It also includes 4 shutters to keep the target as clean as possible avoiding being coated with the evaporated materials. Inside the chamber, the distance between the tube and the evaporator is fixed for a good homogeneity of the coating in reactive processes. For the reactive sputtering of dielectrics a pulsing DC power supply is used, as well as the feedback control of the reactive gas introduced to the chamber by monitoring the plasma impedance or the optical emission of the plasma.

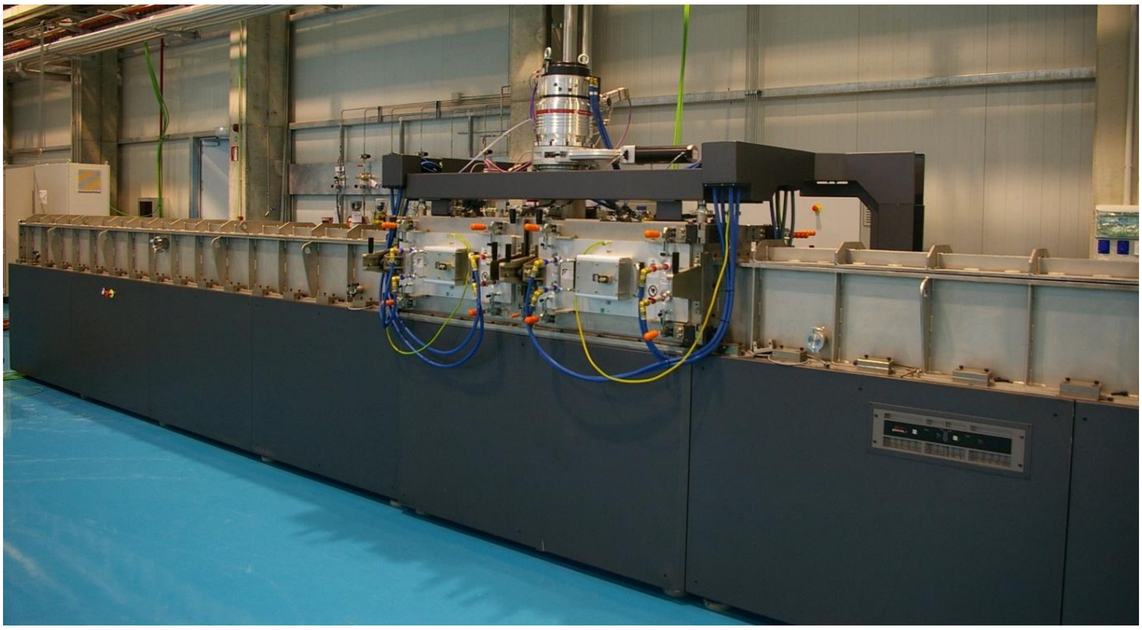


Figure 1. Sputtering chamber used to deposit the solar selective coatings.

2.2. Solar selective coatings

The basic structure of a solar selective coatings deposited is composed by the following layers ordered from top to bottom. Anti reflection layer (AR), absorber cermet layer (AC), infrared reflector layer (IRR) and anti diffusion layer (AD). The AR layer is used to avoid losing absorptance due to the reflection of a part of the solar spectra. The AC has to absorb the maximum possible radiation in the solar spectrum range, so it is expected to have reflectivity close to zero in the solar spectrum range. The IRR mirror is used to avoid radiation thermal losses in the infrared range, so it is expected to have high reflectivity in that range of the spectrum. Finally AD layer is to avoid the migration of species from the stainless steel substrate tube to the solar selective coating.

In order to obtain the desired optical properties for the solar absorber the software Thin Film Wizard was used to optimize the thickness of each layer. The software uses as inputs the optical properties of the layers which in turn depend on the deposition parameters. Thus, preliminary depositions of each coating varying the deposition conditions are used to obtain the optical properties. This is especially important for the design of the cermet AC layer as its optical properties depend strongly on the proportion of metal and dielectric.

The optical indexes obtained experimentally are used in the optical design software to optimize the right proportion of metal/dielectric and the thickness of the layer.

Three different stacks having different compositions were deposited in the project. Preliminary testing was conducted in order to assess that the coatings deposited had the potential to be used in further testing. From this preliminary testing two of the coatings were discarded and the final stack to be used in the project has the following composition and optical values shown in table 1 and SEM crosssection revealing the structure of the stack is shown in Figure 2.

Table 1 Stacks used in the project

Layer	Material	Thickness	Absortance	Emissivity (400ºC)	Emissivity (600ºC)
AR	SiO_2	75	92	7	9
AC	Mo/Al_2O_3	60			
IRR	Mo	200			
AD	Al_2O_3	150			

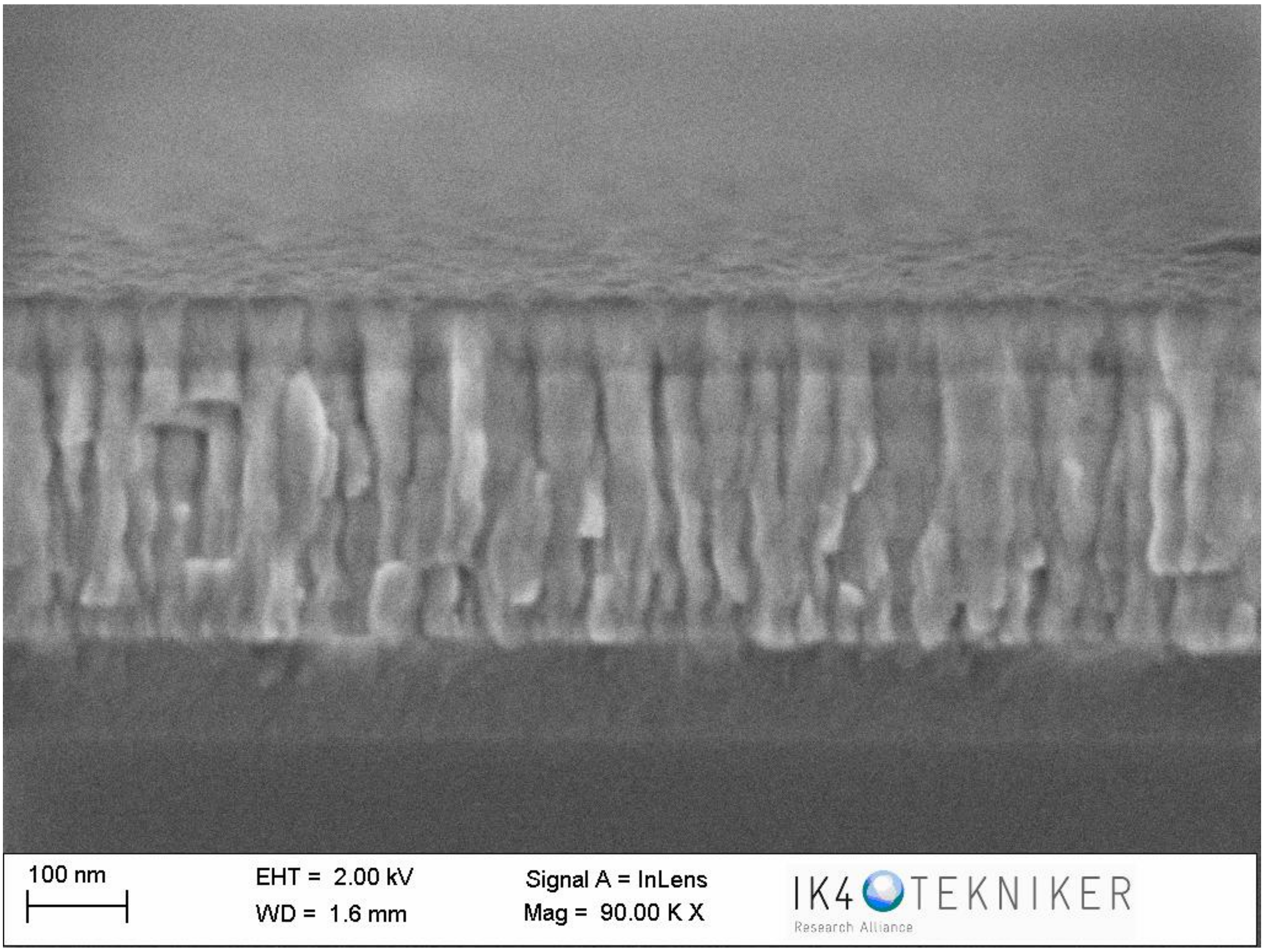


Figure 2. Cross section of the solar absorber coatings

The coatings were deposited on small flat stainless steel substrates as well as in small cylindrical samples and 4 meter long tubes for different characterization purposes.

For the best performance of the CSP system the tube must maintain the optical properties all along its length. This has been taken into account within the design of the PVD pre-industrial line. The design of the deposition system guarantees the homogeneity of the coating along the tube by moving the tube through the sputtering evaporators and rotating it at the same time. Figure 3 shows a coated tube on the left side, the center and the right side of the tube having the same color. As this is a color obtained by optical interference, the homogeneity of the color is a clear indicator of homogeneity of the layers as small differences in coating thickness would produce different colors.

Profilometry measurements on absorber coatings deposited on microscope slides for the quantitative assessment of the homogeneity of the coating. Measured differences of thickness between points of located at both ends of the tube and at the center are <5% (less than 10 nm).

Figure 3. Images of the left, center and right positions of the tube coated with the absorber showing a homogeneous distribution of the color.

3. Development of equipment and tooling for ageing and testing

The working conditions defined for the coatings required the manufacturing of special equipment to obtain the desired conditions during the ageing tests. Thus two different ageing systems were developed during the project to be used for ageing tests.

3.1. Ageing system for cylindrical samples

A laboratory ageing system was designed and manufactured by TECNOVAC in order to perform the ageing of cylindrical samples coated with selective coatings. The system can handle different vacuum levels and temperatures <800º C as well as the possibility of working with different mixtures of gases, monitored in real time during the testing.

The main features of the ageing system are the following:

- Pressure in vacuum conditions can reach $P<5x10^{-6}$ mbar. Different gases can be introduced in the chamber with a high control of partial pressures which are monitored using a quadrupole mass spectrometer. The control system allows to work on static and dynamic vacuum conditions.

- The system allows to reach T<800ºC on the samples surface, with maximum accuracy by using infrared heating lamps and 8 thermocouples to monitor the temperature.

-Flexible software ready to work in automatic and manual way and recording of many output signal, such as: temperature, pressure, power, residual gas composition.

The ageing system has shown to be able to stand the required harsh working conditions: long time cycle time, high temperature, oxygen concentration giving reproducible results and was used to age cylindrical samples coated with the solar selective coating. Figure 4 shows a picture of the ageing system.

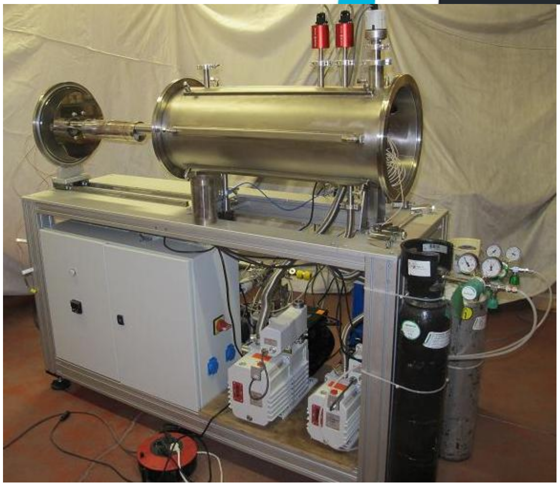


Figure 4. Ageing system developed by TECNOVAC

3.2. 9-chamber system for flat samples

9-chamber oven system enables simultaneous treatment of equal samples under 9 different conditions was constructed by NIC. The ovens are precisely controlled and heating rate could be precisely applied. The computer regulation enables both the isothermal treatment or even temperature cycling at any temperatures till 750 °C, while vacuum or different partial pressures of Ar/O_2 can be applied as second variable at each chamber/oven The variety of aging conditions is needed for reliable source of large-enough number of data for calculations for the estimation of the life-time. On the other hand each chamber can be loaded with 8-10 samples (depending on the size) enough to obtain statistics under the same conditions to control the deviations for life time calculations or over different substrates for specific needs of spectroscopic techniques.

Figure 5. Ageing system developed by NIC

3.3. Sample holder for mechanical measurements on cylindrical geometries

A sample holder for cylindrical samples was designed and manufactured by Anton Paar TriTec (CSM Instruments) that enable the measurement of the mechanical properties of the solar selective coatings using nanoindentation and nanoscratch techniques. The sample holder was used to measure the mechanical properties of the coatings before and after the ageing tests.

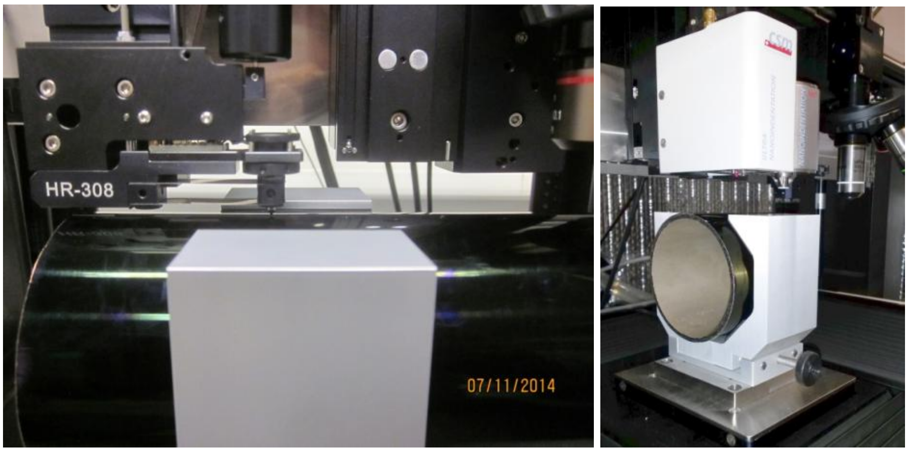


Figure 6. Nanoscratch and nanoindentation on cylindrical samples

4. Spectral selectivity values and new degradation indicators based on spectroscopic techniques

The spectral selectivity values (absortance and emissivity) have been measured in order to perform calculations of lifetime expectancy of the coatings based on accelerated ageing tests both on flat and curved samples. In order to measure these selectivity values on curved samples a methodology for measurement in curved samples have been set up. On the other hand different spectroscopy techniques where studied in order to analyze their feasibility to be used as new degradation indicators.

4.1. Measurement of spectral selectivity values on curved samples

The absortance and emissivity are responsible of the performance of the coating. Thus, the change of these values is the main way to identify the degradation of the coatings. The spectral selectivity is obtained by measuring the UV-VIS-NIR and the FTIR spectra from which the values of absortance and emissivity are calculated using the equation of blackbody radiation. Nevertheless, the standard laboratory equipments used to measure the spectra are designed to be used on flat samples. As ageing experiments were conducted on cylindrical samples, it was necessary to set up the measurements for this kind of geometry. IK4-TEKNIKER analyzed the differences on the spectral selectivity values showing that the main deviations come from the FTIR measurement which underestimates the values of emissivity when measured on curved samples. Further research showed that in order to measure curved samples it is necessary to use an initial reference sample (used to establish the background of the measurement on the FTIR) having the same curvature than the sample to be measured. In this way the values obtained for the emissivity values are the same as in the obtained with flat samples. This approach allows to obtain the emissivity value in curved samples using a standard FTIR laboratory equipment. This result has proven to be very interesting both for plant builders that want to assess the values of the coatings of their suppliers and for coating developers that want improve the performance of their coatings. In this sense the result is already under exploitation and IK4-

TEKNIKER has already provided services to its customers on the CSP market to assess the properties of coatings on curved samples.

4.2. New degradation indicators

One of the objectives of the project was to study if there are other degradation indicators beyond the spectral selectivity values. Thus different vibrational techniques were analyzed in order to understand if they are suitable to be used as the degradation indicators upon which life-time assessment can be made. The techniques that were considered were:

- IR absorptance spectroscopy (samples deposited on partly IR transmissive silicon wafers, thickness ~ 200 nm),
- ATR IR spectroscopy (diamond (n = 2) and Ge crystal (n = 4)), (samples deposited on stainless steel, thickness ~ 1000 nm and 100 nm were tested),
- Near Grazing Incidence Angle IR reflection-absorption (NGIA IR RA) spectroscopy (samples deposited on stainless steel 2x5 cm^2, thickness ~ 100 nm),
- Raman spectroscopy (samples deposited on stainless steel, thickness ~ 1000 nm)
    - confocal Raman with green laser at 532 nm,
    - Raman with red laser at 632 nm,
    - FT-IR Raman with excitation line at 1065 nm.

To this purpose ageing of coatings at different conditions was performed and the aged coatings were examined using different vibrational techniques to check if the variation of the bands due to the aging could be quantified. These vibrational techniques were evaluated with regard to the intensity of the signal, its reproducibility (different measured spots on the surface of the coatings) and accuracy. The appropriateness of these techniques as degradation indicators was then evaluated. Most of techniques was not found suitable and were disregarded from further study.

4.2.1. IR absorptance spectroscopy

It was found that IR absorptance spectroscopy can be interesting as degradation indicator since the IR spectrum starts to appear when IR reflective Mo layer started to oxidise. Namely, the Mo as reflective IR layer prevents the transmission of IR radiation and therefore the IR spectrum does not appear. It is interesting to notice from Fig. 7 that for SA stacks exposed at 420 °C in Ar even after 215 h the spectrum of oxidised Mo did not appear. At increased temperatures of 460 and 500 °C, the oxidation of Mo started after 67 and 50 h, respectively. Any presence of oxygen accelerated the oxidation as evident from spectra obtained for 2% O_2/Ar. However, in order to use IR absorption technique as degradation indicator, the observed effect should be quantified. **This technique is very suitable for detection of any oxidation, but there remains a problem of how to quantify the process of oxidation to use it as a degradation indicator.**

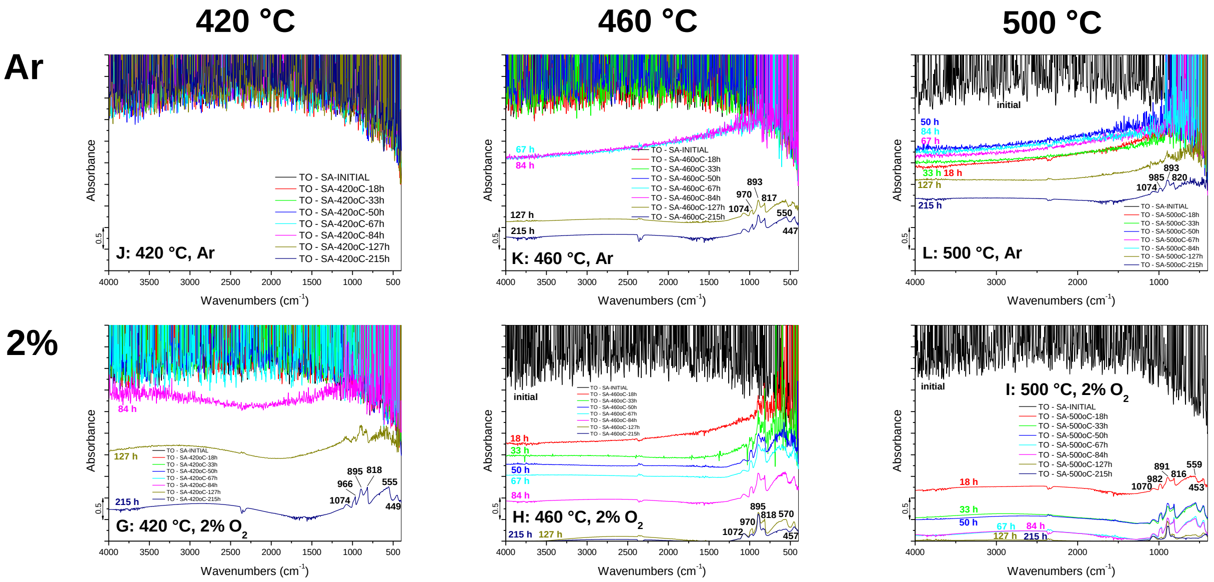


Figure 7. IR absorptance spectra of multilayered SA stacks and the same SA stacks exposed to 2% O_2/Ar and Ar itself at 420, 460 and 500 °C.

4.2.2. NGIA IR RA technique (LO modes)

The vibrational technique that was chosen as a possible degradation indicator is NGIA IR RA technique, which gives longitudinal optical (LO) modes in the spectra. For this technique, the IR radiation fall to the sample at a near grazing incidence angle of 80° and should be p-polarised light (perpendicular to the substrate). The coating should be deposited on a reflective substrate and should be thin, for instance of about 100 nm. We used SA stacks that were deposited on stainless steel and the NGIA IR RA spectrum of initial SA stack depicts the characteristics of the uppermost SiO_2 layer.

In Fig. 8 there are depicted the LO spectra of initial SA stacks used for measurements and the same SA stacks exposed to defined thermal and oxidative conditions. The black (uppermost) spectra represent the initial SA stacks. The most intense SiO_2 mode at 1232 cm^{-1} was used for estimation of life-time. The background was subtracted from these SiO_2 bands and then the integral intensity was calculated using the program written. The numerical integral intensity values were then used for calculation of life-time.

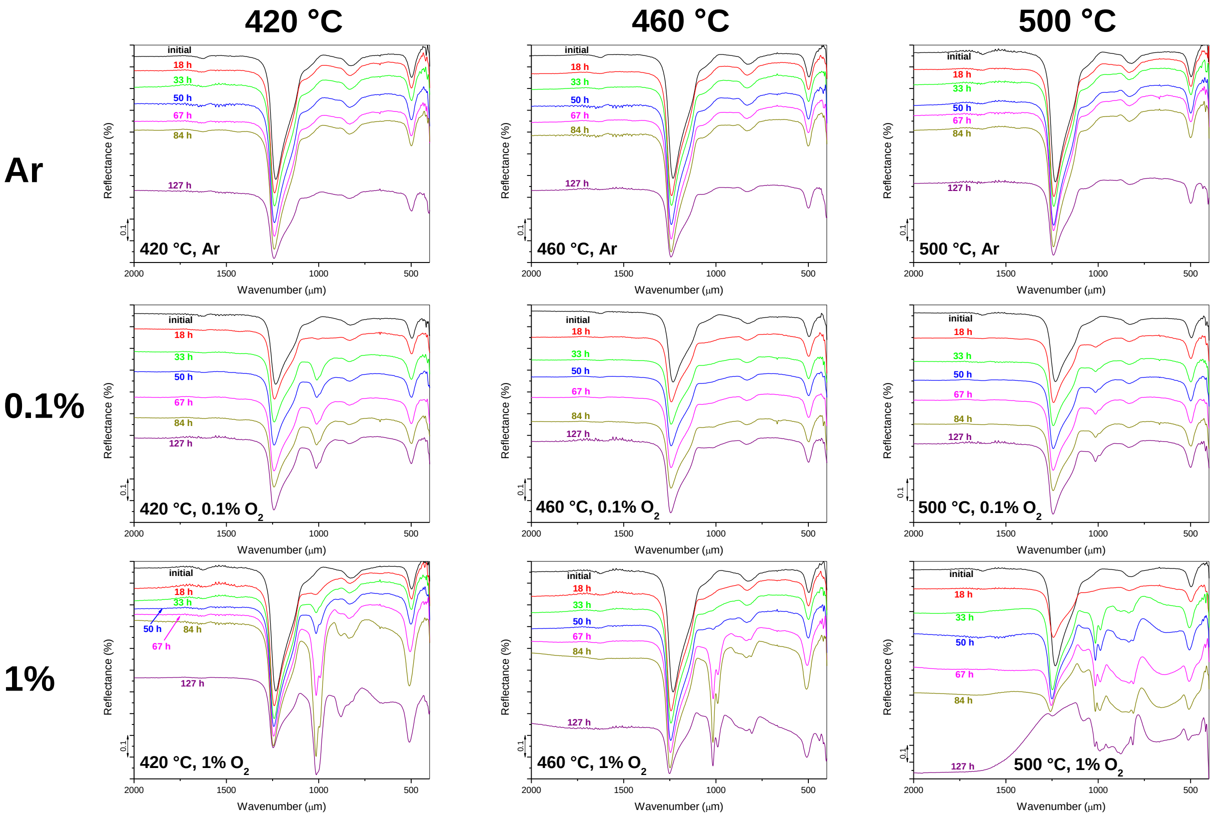


Figure 8. NGIA IR RA spectra (LO modes) of multilayered SA stacks in initial state (black spectra at each graph) and the same SA stacks exposed to 1% and 0.1% O_2/Ar and Ar itself at 420, 460 and 500 °C. The most intensive SiO_2 mode at 1232 cm^{-1} was used for life-time assessment.

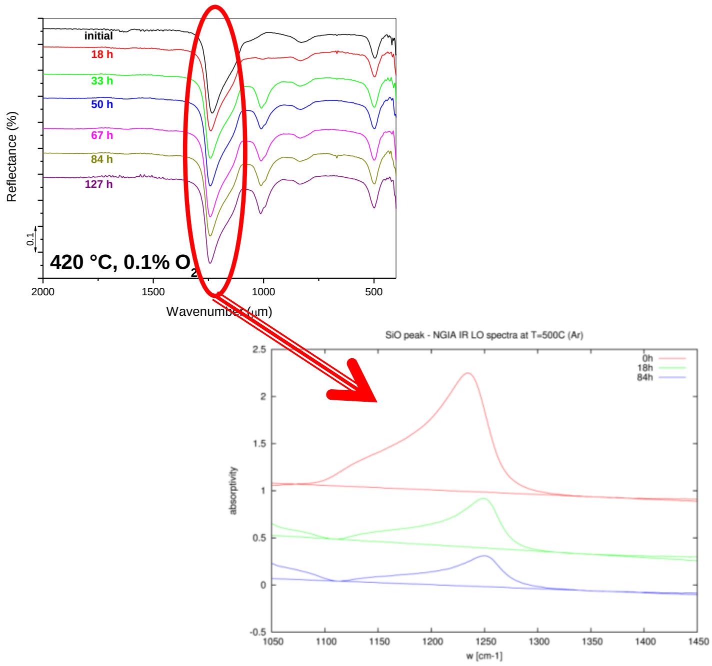


Figure 9. LO mode of SiO_2 that was used for calculation of integral intensity and then also life-time assessment.

5. Development of testing protocols for accelerated ageing

A testing protocol for accelerated ageing was developed by ECP-CRSA. The protocol takes all solicitations likely to occur under the condition of use. This approach includes three strategies: **Thermal Solicitations**, **Mechanical Solicitations** and **Chemical Solicitations**.

Thermal Solicitations

Both thermal shock and thermal dilatation tests were performed on the samples. The aim of *Thermal Shock* test is to verify the multilayer coatings endurance for quick temperature change. In the case of *Thermal Dilatation* test, we want to know the stability of multilayer coatings at constant high temperature. All thermal solicitation tests were performed under air or inert gas (He).

Mechanical Solicitations

A pin-on-disk tribological test was carried out, since it is no doubt that multilayer stack will be worn in the tube-fixation contact. The defaults, created by the tribological test, can lead to the galvanic coupling between different layers that may influence significantly the properties of multilayer coatings versus corrosion. In this case, to better study the properties of each layer and mimic the real applied conditions, we removed the multilayer coatings layer by layer. All tribological tests are performed under the protection of Ar.

Chemical Solicitations

Corrosion cycling tests will be performed on the pristine and worn multilayer stack before and after mechanical solicitations either under inert gas (He) or under oxygen (controlled partial pressure).

Figure 10 shows the scheme of the accelerated testing protocol chosen for multilayer coatings.

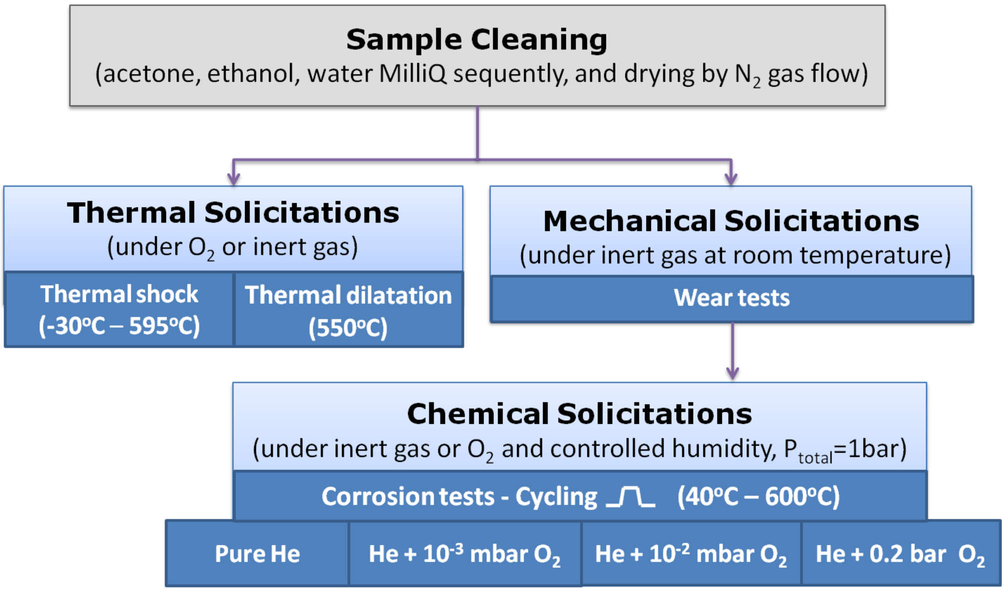


Figure 10. Accelerated testing protocol.

The accelerated testing protocol followed by measurements using SEM, EDS, AFM and GIXRD techniques were used to identify the main degradation mechanisms of the coatings.

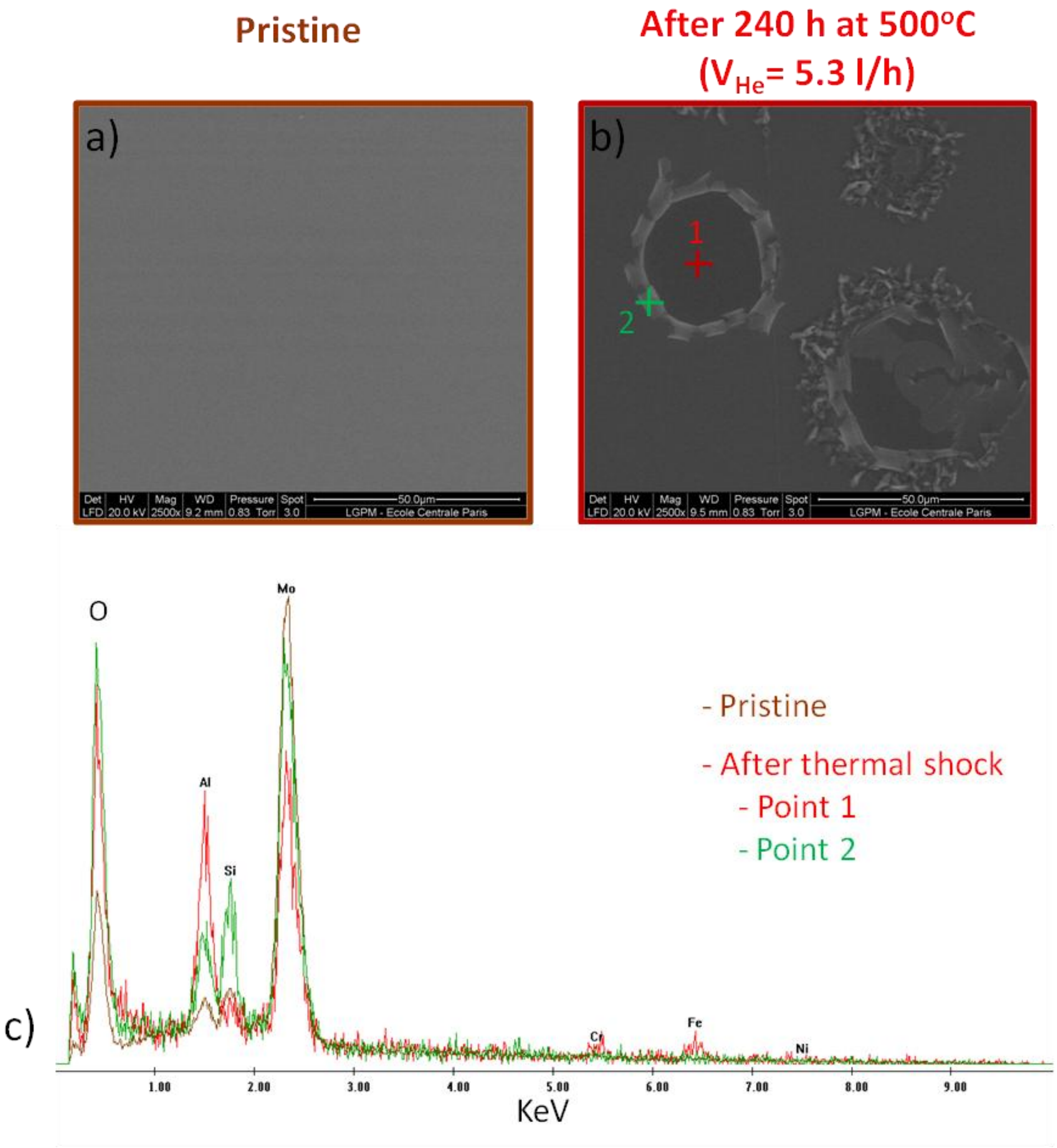


Figure 11. SEM micrographs of solar selective stack a) before and b) after shock test between 40°C to 595°C; and their related c) EDS spectrograms.

Thus, oxygen content, temperature and wear were identified as the main loads leading to the degradation of the coatings, being the oxygen content the most important load affecting the coatings leading to the oxidation of the Mo. Moreover from the application of the degradation protocols the following conclusions about the coating stability have been obtained:

- Multilayer samples showed good thermal stability. The adhesion of layers was observed up to 500 °C. For actual application, it hold the good quality if the temperature is lower or equal to 400 °C.

- The oxidation behaviour of Mo, including the formation of MoO_2 and MoO_3, and volatilization of MoO_3, is the key issue of the degradation of multilayer stack. It seems can limit the volatilization rate of MoO_3 by:
    - Making compact upper layer (SiO_2) without the microholes (µ-porosity) which can play a good role as molybdenum isolation from oxidizing media such as oxygen, water, CO_2.
    - Controlling atmosphere environment, ex. inert gas flowing rate which was proved by heating 3rd batch of sample at 500 °C under controlled He flowing.

6. Mechanical properties of the coatings

The characterization of mechanical properties of the absorbing coating multilayer stands out as one of the few methods that allows for complete quantitative characterization of the integrity, mechanical quality and adhesion of the coatings. For that reason nanoscratch and nanointendation was performed by Anton Paar TriTec (CSM Instruments) in order to obtain the mechanical properties and adhesion of the coatings before and after the ageing tests.

At an initial stage the testing of the mechanical properties was done on flat samples coated with the individual layers of the stack as well as on the whole stack in order to establish the methodology of testing.

Once this preliminary work was done the sample holder developed for cylindrical samples was used to perform the test on cylindrical samples before and after the ageing tests.

Since the measurements have been performed on a large number of samples with various configurations of layers during the first period, the probability of usability of the tests also on the aged samples was considerably increased. The crucial part concerned mainly scratch testing that would rely on comparability of the critical loads before and after the ageing tests. Thanks to the experience with scratch testing gained during years of work on applications and also due to experience obtained during the NECSO project we could successfully apply the scratching protocol also on aged samples. It was therefore possible to compare the wear and scratch behavior of the tube samples before and after the ageing tests.

In comparison to scratch testing, indentation experiments were relatively less prone to variability of samples before and after the tests as these tests do not rely on optical observation. Also, as confirmed by AFM images, the surface roughness after ageing did not significantly increase and the indentation loads did not have to be adapted for the aged samples.

6.1. Nanoscratch

Two diffent load regimes were used for the testing:

- 0.03 mN to 11 mN – low loads (two upper most layers, R = 1 µm tip)
- 1.0 mN to 500 mN – high loads (all layers, R = 10 µm tip)

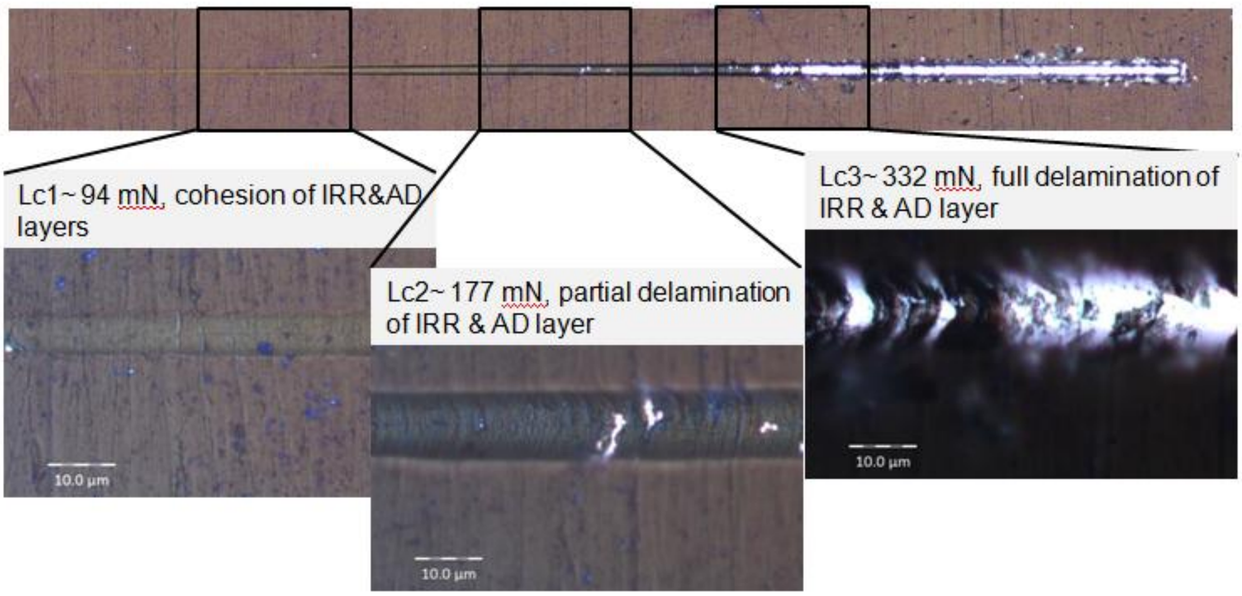


Figure 12. Panorama image showing the critical loads for high loadson unaged samples.

The Panorama images were used to identify the critical loads showing good adhesion of the layers. The testing on aged samples showed that in the case of temperatures below 500ºC, oxygen conten below 0.1 mbar and less than 3 days of ageing, the adhesion of the layers is not affected by ageing. Nevertheless when ageing time up to one week and temperature was increased while the oxygen content was kept very low, the adhesion of the *top layers* decreased. On the other hand, higher temperature lead to decrease of adhesion of the *entire* coating. In this sense it is worth to mention that the influence of oxygen on adhesion of the entire coating is minor compared to temperature.

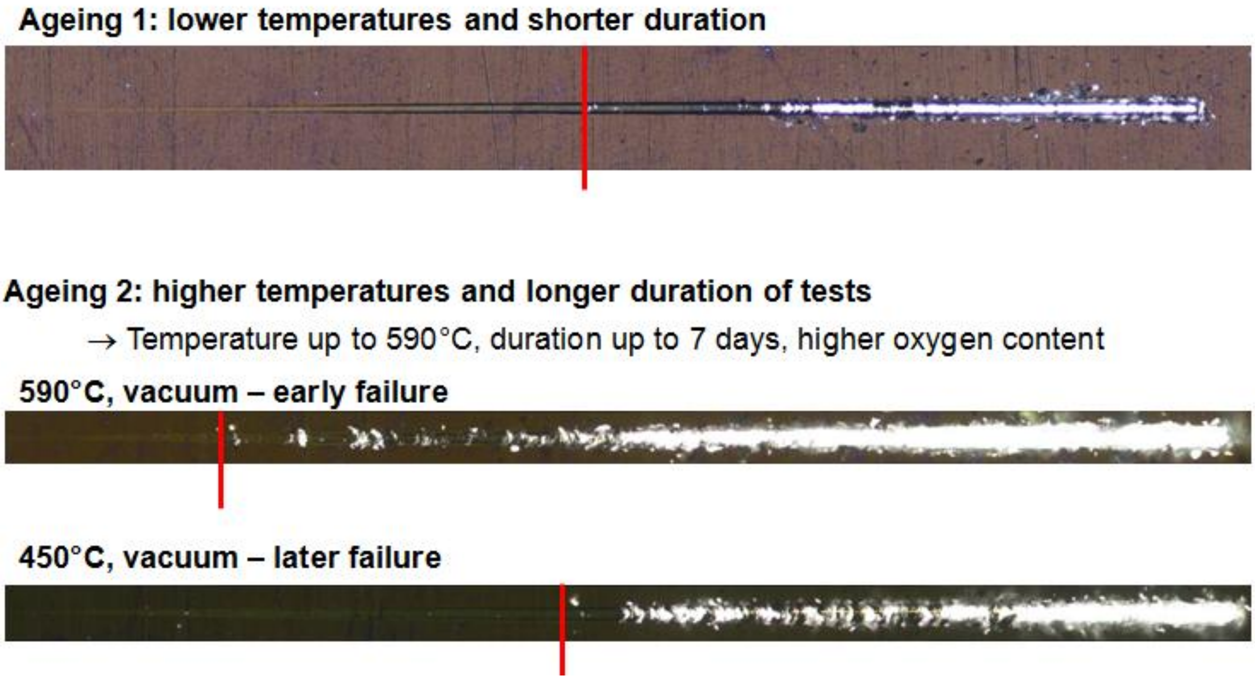


Figure 13. Panorama images comparing low temperatures and short ageing times with higher temperatures and longer ageing times.

6.2. Nanoindentation

Again, as in the case of nanoscratch two different load regimes were used to perform the nanoindentation in order to obtain the hardness and Young's modulus.

- **Low load (20 µN)**. Testing of the upper most layers (AR+AB), maximal indentation depth ~14 nm.
- **High load (3 mN)**. Testing of overall properties of the entire coating, maximal indentation depth ~140 nm.

The results showed that a small decrease of the modulus of the top layers, negligible effect on modulus of the entire coating after ageing.

7. Tribological evaluation of solar selective coatings under contact conditions

The selective coatings currently used in solar collectors do not present tribological issues. Nevertheless, tribological problems appear on new concepts such as the HITECO receiver for higher temperatures by ARIES. For this concept wear and tribological issues are important as there are contacts that may damage the coatings. Moreover, the testing performed by ECP-CRSA to study the corrosion behaviour showed that scratches on the surface of the coatings exposes the Mo layer leading to oxidation.

Based on the conditions established by ARIES, there are different types of wear appearing in the solar selective coatings, due to the different movements in the tube fixation contacts. In particular, during the contact analysis performed, four different movements are likely to cause wear issues. These are:

a. the rotation of tubes (every 20 s) to adjust to incoming solar light,
b. a lateral tube expansion due to heating up (every 24 hours there is 1 cycle of expansion and compression),
c. minor tube vibrations due to liquid pumping inside the solar tube, and
d. major tube vibrations due to variations in wind speed and direction.

In order to simulate the in-field contact conditions to lab scale tests, two different types of experimental approaches were used. The rotation of the tubes and the lateral tube expansion can be simulated by reciprocating sliding, whereas the minor and major tube vibrations by fretting mode II tests.

a calculation methodology was developed to determine the type and level of applied stress. On this basis KUL performed tribological tests at different scales relevant for the system.

Performing lab tests over a broad load range as relevant for NECSO set ups, is a significant challenge. The selection of an appropriate lab test equipment is here also essential. A variety of techniques operating at different load scales but with a similar contact pressure can be used as shown in Fig. 14.

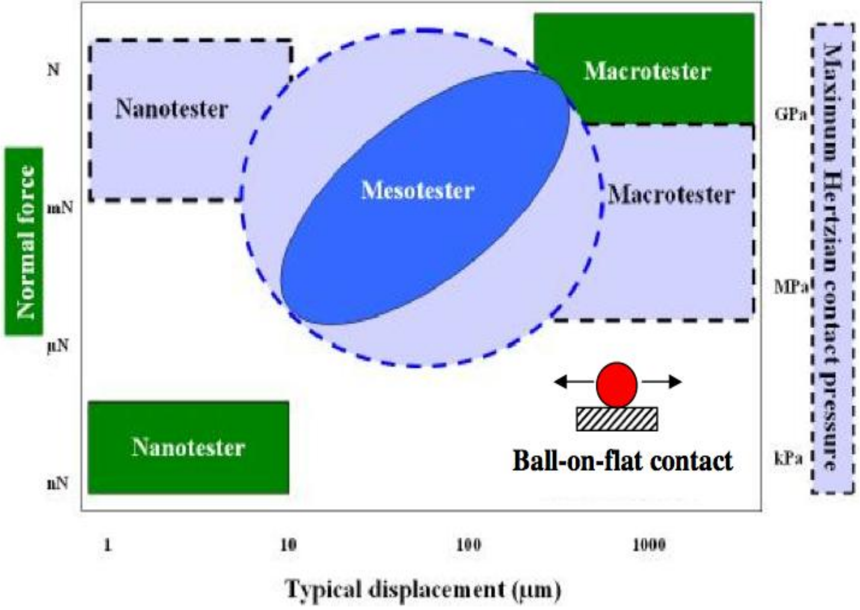


Figure 14: Measurement ranges of three different tribological testers operating under ball-on-flat contact conditions

It was found that no measurable wear was obtained when applying loads at the micro range. Modular Universal Surface Tester testing is suitable for evaluating the frictional and wear characteristics of NECSO coatings under sliding conditions at meso-loads. However, the applied contact pressures should be higher than the actual ones, in order to induce sufficient wear.

Oxidative wear should occur on the surface of the material. SEM analyses of the wear tracks confirmed the existence of an oxide based tribolayer after sliding.

In order to define the thickness of that tribolayer and to calculate the critical oxide thickness ξ, FIB cross sections through the wear tracks were performed (Fig. 15). A thickness of the tribolayer in the range of 70 nm was obtained.

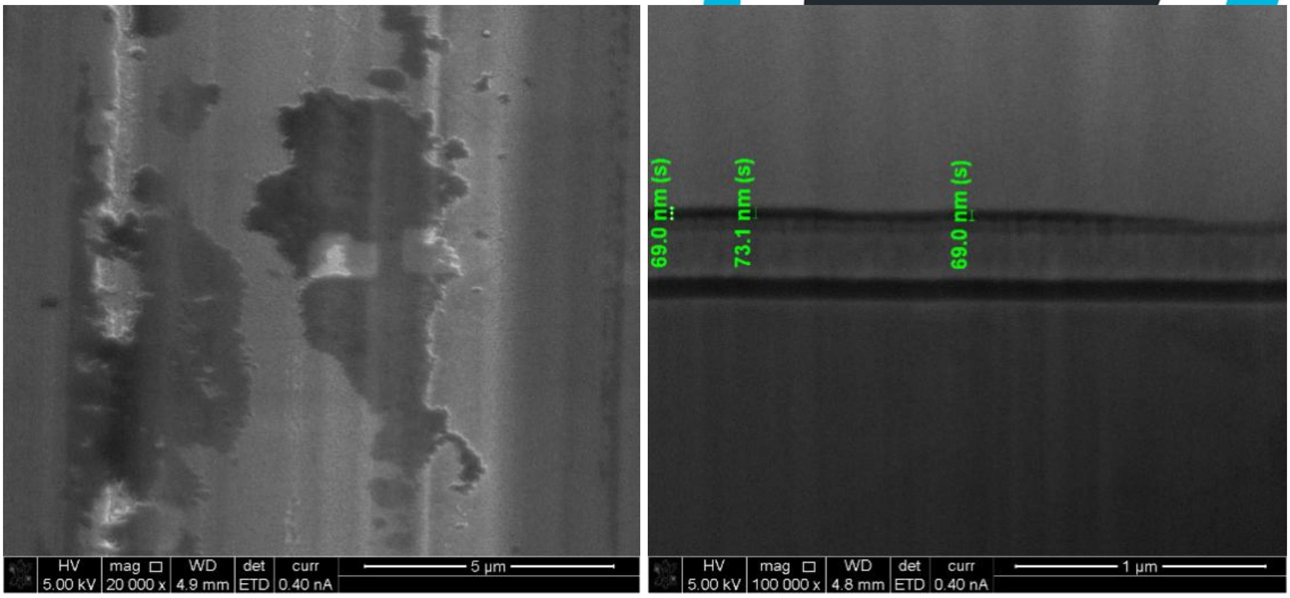


Figure 15: (a) surface morphology of wear track on full stack NECSO coatings after sliding test done at a meso-load of 20 mN for 10000 sliding cycles, and (b) cross section through the wear track perpendicular to the sliding direction

Testing of the individual layers and the full stack (SA) NECSO coating indicated a totally different frictional behavior between them. Thus it is believed that by utilizing a “sensitive” frictional technique it is possible to discern the failure (e.g. cracking, delamination) of a sub-layer of the full stack coating and or changes of the contacting interface (debris formation, oxidation etc.). Having in mind that the frictional behavior (coefficient of friction) can provide an indirect, first indication of the tribological stability of the tribosystem.

The analysis of the different frictional behavior is observed at low (20 mN) and higher (200 mN) applied loads. This difference is attributed to the rupture of the SiO_2 top-layer. Indeed, frictional analysis of the full stack coatings without the SiO_2 top-layer showed a higher coefficient of friction in the range of 0.7-0.8. The observed fluctuation is due to debris formation. In addition, this hypothesis is confirmed by the confocal analysis. Therefore the NECSO coatings can be used safely at low load contact conditions.

8. Accelerated ageing tests and lifetime predictions

Ageing tests were performed on the 9-chamber oven built by NIC and on the ageing chamber built by TECNOVAC under different O_2 pressures and temperatures which were identified as the main loads responsible of the degradation of the coatings.

The solar selectivity values were measured in order to feed an isoconversional kinetic model developed by NIC to predict the lifetime of the coatings. Additionally as mentioned in previous sections the changes in the vibrational modes of the SiO_2 layer measured using the NGIA IR RA technique after ageing was used.

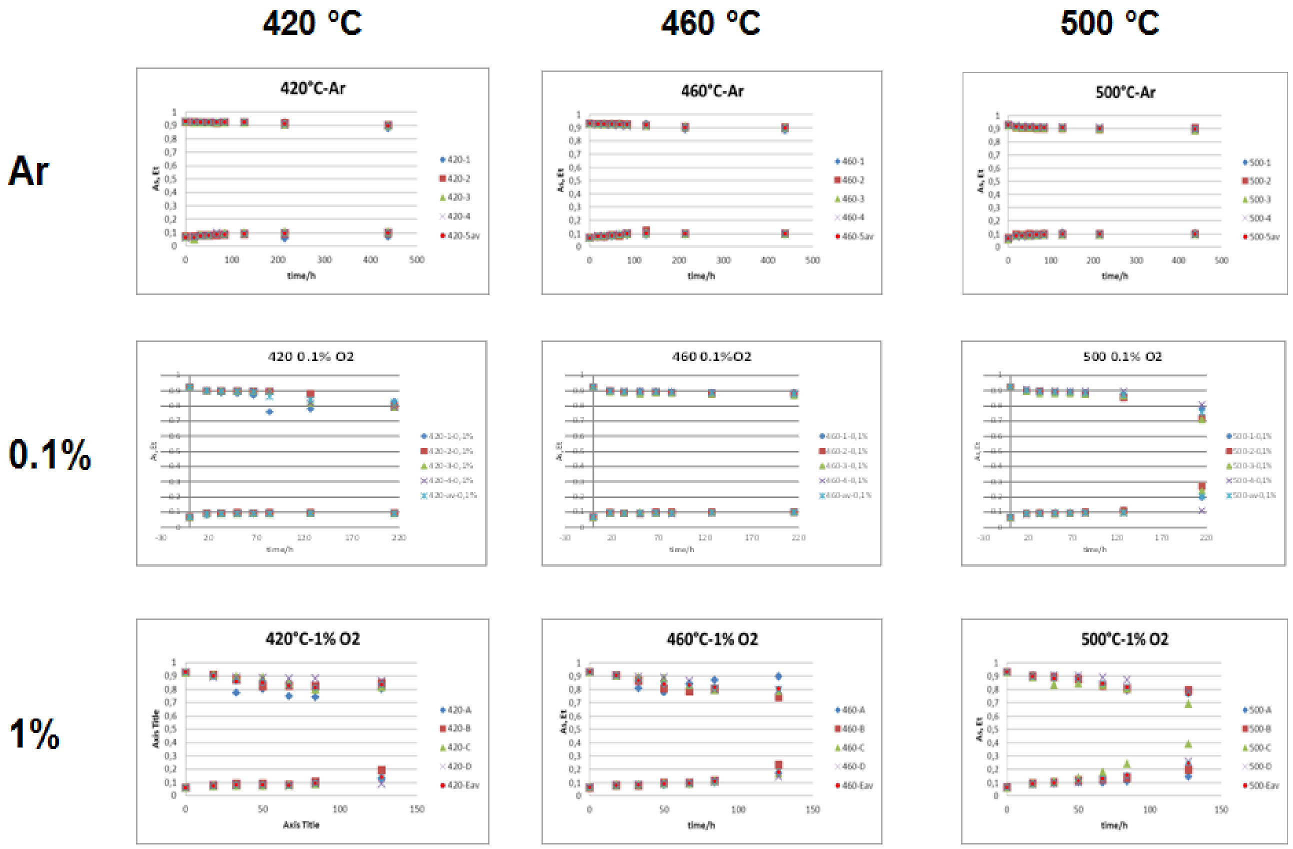


Figure 16. Absortance and emissivity obtained on samples aged in the 9-chamber system

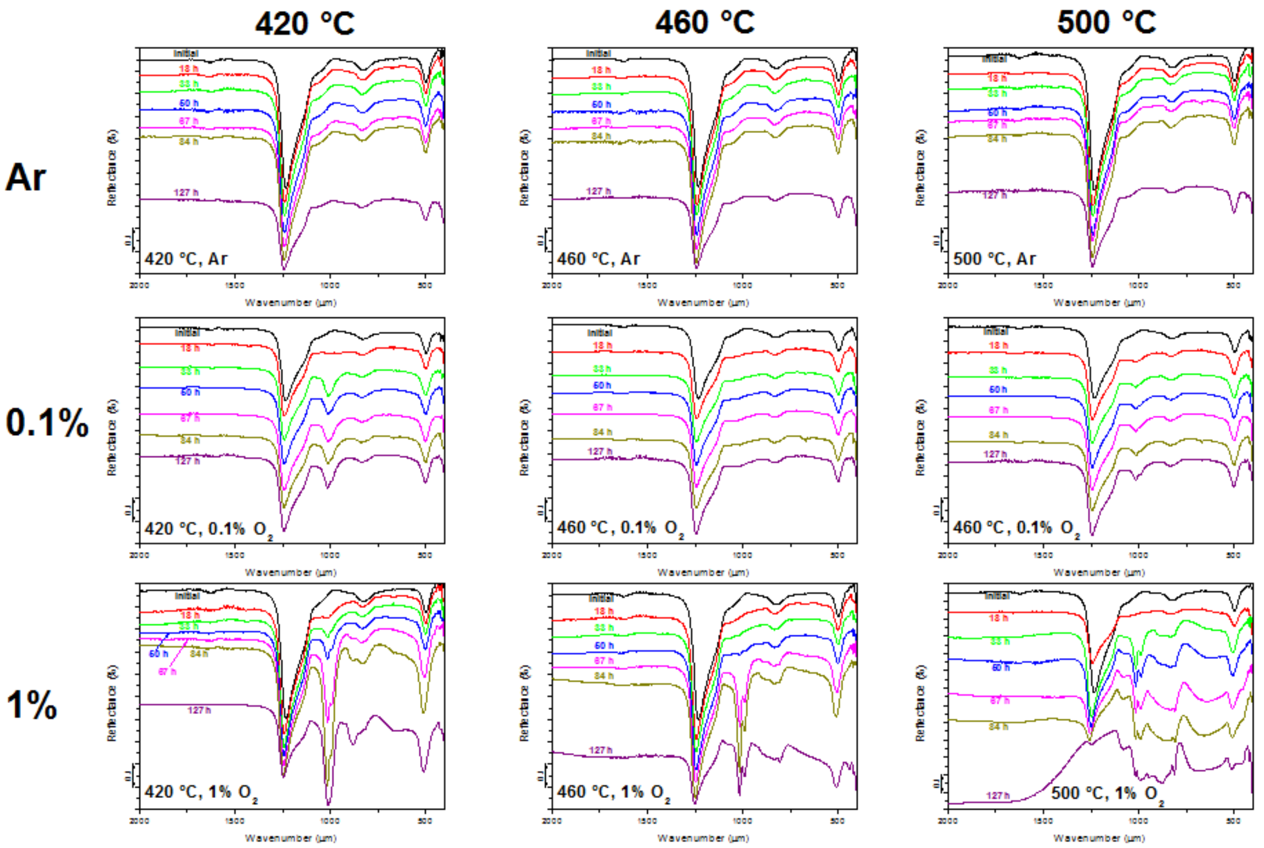


Figure 17. Changes on SiO_2 layer measured by NGIA IR RA

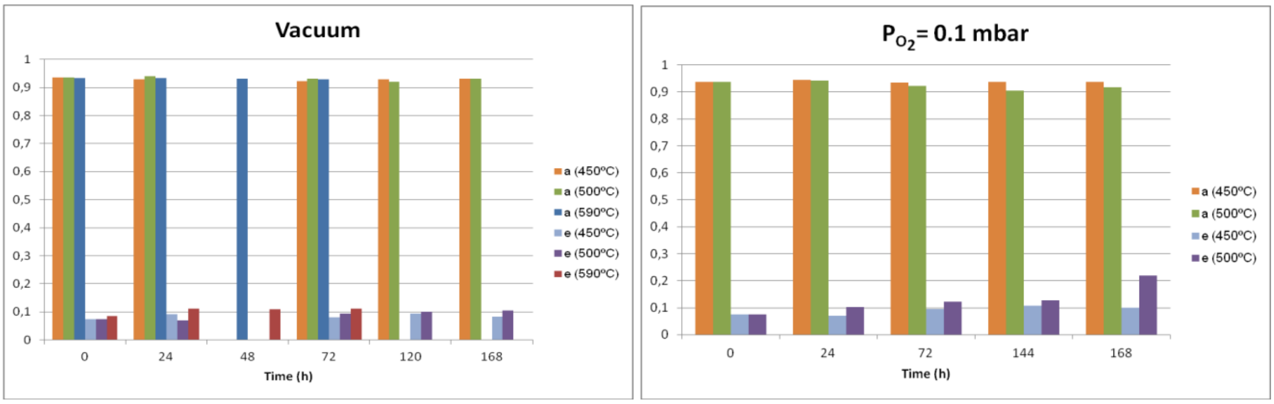


Figure 18. Examples of results obtained on ageing tests in TECNOVAC chamber showing how the coatings are stable up to 500ºC in vacuum and how when O_2 is present the emissivity increase indicating the oxidation of the Mo IRR layer.

6.3. Development of life-time assessment calculation procedure

The procedure for calculation of life-time assessment was developed on the basis of 3x3 matrices of measured/estimated times required to reach critical degradation. The critical degradation was set to 4% jump in e_T or 4% drop in a_s, while a 10% decrease in integral intensity of LO modes was used for NGIA IR RA measurements. The calculation procedure includes temperature and oxidation driven degradation, which can be given by a general expression (Eq. 1):

$$\frac{d\alpha}{dt} = \kappa(T,p) f(\alpha)$$

(Eq. 1)

In which α denotes the extent of conversion (i.e. a_s, e_T, LO mode). Degradation reaction mechanism is described by the functional form f(α) and κ(T,p) is the temperature and the pressure dependent rate constant. If we explicitly postulate that the rate constant depends only on the temperature and the pressure, while we focus on the time at which the fixed extent of degradation (i.e. relative change by 4% - or 10% - relative to the initial value) takes place, we can make predictions of the life time of the coating at various conditions without knowing the exact form f of the degradation reaction mechanism. This fact is known as the isoconversional principle.

The details of the development of the model will not be shown here but once model parameters are known, we can make predictions of the time needed to reach the same critical degradation (i.e. 4% change) at operating conditions (T_0 and p_0) by using reference degradation time tr at reference temperature T_r and p_r. Given that we have 9 different reference conditions (3 x 3 = 9 matrix), we calculate the final prediction as the average of predictions with respect to all N = 9 reference conditions.

The prediction of life-time used in the coating was 958±153 h from a_s measurements and 914±153 h from e_T measurements for a working temperature. The proposed calculation led to quite similar prediction on the basis of both regarded quantities, i.e. a_s and e_T. On the other hand, the prediction of life-time based on the basis of on longitudinal optical (LO) modes in the spectra on the most intense SiO_2 mode was 742±151 h. The proposed calculation led to somewhat smaller prediction of time on the basis of LO modes than was obtained for both other regarded quantities, i.e. a_s and e_T. Better agreement would be achieved when in addition to considering the decrease in integral intensity of SiO_2 mode also the increase in oxidation bands of Mo is taken into account. On the other hand, the estimation of lifetime calculated with the ageing tests performed by TECNOVAC a lifetime of 4543 ± 2746 h was obtained for predictiosn based on 4% drop on the value of absorptance while the estimation is 2018 ± 544 h when the calculation is done considering the drop of emissivity. These values are significantly higher that the ones obtained with the ageing data from the 9- chamber system. In these sense there are a couple of important remarks to be considered. The level of vacuum and the gas content is better controlled on TECNOVAC ageing system while in the case of the 9-chamber oven the partial pressure of O_2 is controlled using Ar flow, an inert gas. Even when using high purity Ar there are residual oxygen that can start the oxidation of the coating. This result is aligned with the experiments performed by ECP-CRSA using He gas that showed that the gas flow has an influence on the oxidation of the Mo layer due to residual O_2 content. Thus, in terms of control of the ageing TECNOVAC ageing system is better. On the other hand the 9-chamber oven allows to perform more tests in a shorter time including a number of samples that allow obtain good statistics that reduce de variability of the final results. Thus, from these results the main conclusion is that both accuracy and high control of gas content and vacuum and having enough number of samples to obtain good statistics are key issues to take into account when ageing tests are performed to be used for lifetime prediction calculations.

9. Fundamental analysis of degradation

Besides the interest of ageing protocols for plant builders to ensure the reliability of the coatings during its expected lifetime, NECSO project also aimed to understand the mechanisms behind degradation in order to help selective coatings developers to improve their coatings. Thus, a fundamental analysis of the mechanisms of degradation have been performed.

9.1. Chemical degradation

According to the testing performed in for the development of protocols which showed that the main degradation of the coatings comes from the oxidation of the Mo, further testing and analysis was focused on the analysis of the mechanisms of degradation by studying the molybdenum oxides formed under different loads. A model to predict the oxidation of Mo is also proposed on basis of XRD analysis.

Combining with scanning electron microscopy (SEM), interferometric microscopy (IM), Auger electron spectroscopy (AES) and Glow Discharge Optical Emission Spectroscopy (GDOES) profiling and gracing incident X-ray diffraction (GIXRD), the thermal dilatation tests were used to study the **oxidation of molybdenum compounds in selective coatings**.

Three major factors, including ageing temperature, ageing time and partial pressure of O2, can influence the oxidation of Mo compounds causing the optical performance fading with no doubt of the coating degradation. Generally speaking, the multilayer stack shows a thermal stability in order of 400 °C > 500 °C > 600 °C. SEM images show the formation of crystalline structure, and pin-holes in the coating surfaces after 240 h heating at 400 °C and 500 °C, respectively. It is reported that the major oxide products of Mo are MoO_2 and MoO_3.The oxidation of Mo begins at 250 °C, firstly forms a brownish-black oxide of MoO_2 directly on the substrate, then a yellowish-white oxide of MoO_3 on the top of MoO_2 film . Furthermore, MoO_3 is not stable at high temperature that volatilize at 475 °C under vacuum, at 550 °C in 10.1 kPa of oxygen, at 650 °C in 101.3 kPa of oxygen. This can explain the formation of pinholes at 500 °C which may result from the volatilization of MoO_3. Obviously, the mircro-porosity of the upper SiO_2 layer is not small enough that cannot well protect the inner layers from the oxidation. In case of 400 °C, after 240h heating the roughness of the multilayer stack surface was quite stable no matter which He flowing velocity, 2.34 or 5.30 l/h, was used. However, in case of 500 °C, the interferometric images show a remarkable decrease of surface roughness when O_2 partial pressure fall down that may be related to the volatilization rate of MoO_3.

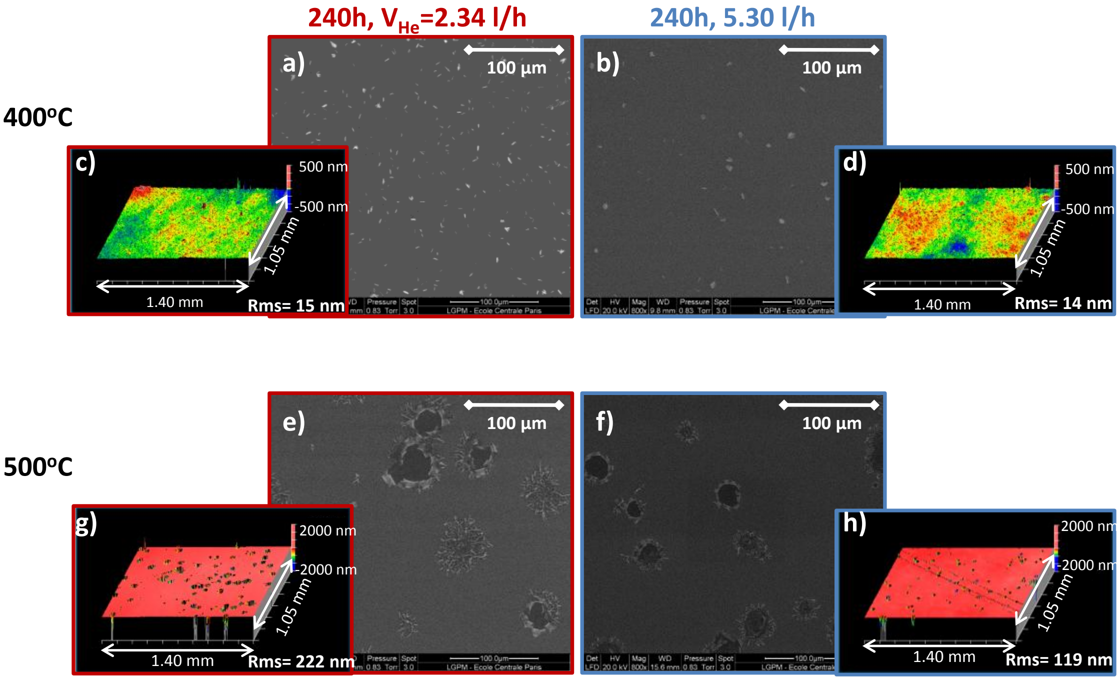


Figure 19. SEM imagies of multilayer stacks (3rd batch) after 240 h heating at 400 °C under pure He flowing of a) 2.34 l/h and b) 5.30 l/h, at 500 °C under pure He flowing of e) 2.34 l/h and f) 5.30 l/h; and relevant 3D surface topography of multilayer stacks after 240 h heating at 400 °C under pure He flowing of c) 2.34 l/h and d) 5.30 l/h, at 500 °C under pure He flowing of g) 2.34 l/h and h) 5.30 l/h.

To understand the oxidation level of coatings, AES profiling was performed before and after 240 h ageing at 400 ^{o}C. The increase of oxygen atomic percentage in the cermet layer indicates the penetration of O_2 from SiO_2 to sublayers. The large plateau of oxygen in the cermet layer suggests a homogeneous Mo oxidation along the cermet leading to its thickness enlargment. Hence, the oxidation of Mo compounds happens mostly along the cermet and possibly also in the cermet/Mo interface.

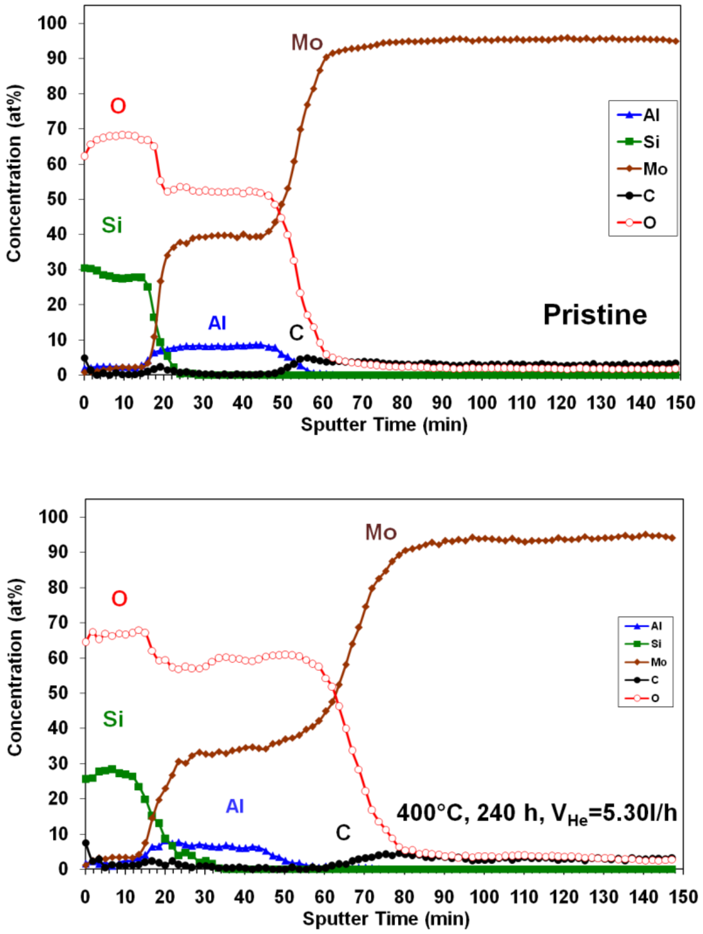


Figure 20. AES depth profilings of pristine and 400^{o}C 240 h aged samples.

GIXRD results of complete 4-layer stack after 24h ageing at 600 ^{o}C revelas the presence of $Al_2(MoO_4)_3$ and MoO_2, MoO_3 is not detected. But SEM shows the formation of holes which suggests the vaporization of MoO_3 for this sample. The presence of aluminium molybdate, suggests that in cermet the freshly formed MoO_3 could react with Al_2O_3 to convert to aluminium molybdate. The possible oxidation mechanism of Mo could be:

$$Mo + O_2 \rightarrow MoO_2 \qquad (1)$$

$$2MoO_2 + O_2 \rightarrow 2MoO_3 \qquad (2)$$

$$3MoO_3 + Al_2O_3 \rightarrow Al_2(MoO_4)_3 \qquad (3)$$

On the basis of XRD measurements during thermal cycling there were also calculated the degradation profiles

The assumption that was used: amount of Mo in the sample is proportional to the integrated XRD peak intensity. The points in the graph correspond to the results of the XRD measurements that were normalised to the initial integrated peak intensity.

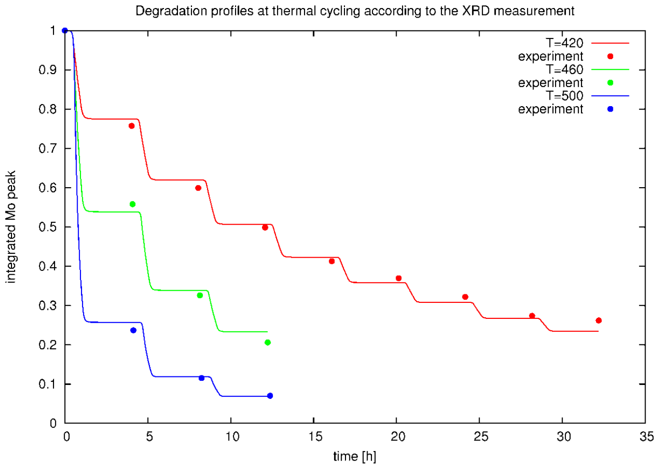


Figure 21. Degradation profiles at thermal cycling according to the XRD measurements.

Trends in Fig. 21 revealed one-way reaction mechanism:

$$\frac{dS(t)}{dt} = -k(T)S(t)^p \qquad \text{(Eq. 2)}$$

Where S(t) represents normalised integrated intensity and K(T) temperature dependent rate constant. After comparison of solutions of equation (Eq. 9) and the measured values, as well the variation of the rate constant function and power p, the rate constant dependence on temperature was best described by the exponential function:

$$k(T) = \alpha \exp(\beta T) \qquad \text{(Eq. 3)}$$

By using this model a prediction of the oxidation with time can be made for different temperatures (figure 22)

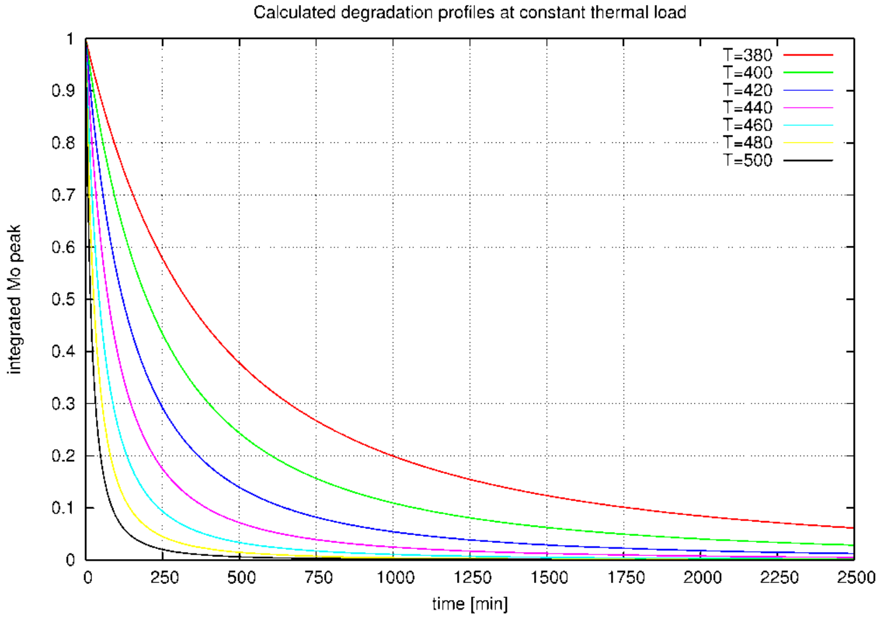


Figure 22. calculation on degradation profiles for the oxidation of Mo.

9.2. Nanomechanical degradation

Based on the protocol previously developed for tribological conditions loads on the meso load and macroload range were applied. By using HertzWin software and applying the mechanical characteristics (Hardness, Young's modulus) which were provided by the NECSO partners (ECP, CSM) the contact pressure under both macro- and micro- load scales was calculated.

A significant difference between the friction and wear of the solar selective coatings in the meso- and macro- load scale is observed. This is mainly because when a normal force is applied on a material, elastic or plastic deformation of its surface occurs. For an elastic ball-on-flat contact, as used in this series of tests, the maximum Hertzian elastic deformation depth δ, is given by the following equation:

$$\delta = a^2 / R \approx x\,(Fn^{2/3}/R^{1/3}) \qquad (1)$$

where a is the contact radius, R is the radius of the counterbody, Fn is the applied load and x is a constant.

At low normal forces, the deformation depth δ is of the order of a few nanometers and thus the influence of surface properties (SiO_2 layer of the solar selective coating) are more pronounced. In addition, at higher applied loads and deformation depths, the bulk characteristics of the coating and of underlying layers (Mo cermet layer) also play a vital role. Indeed, by plotting the coefficient of friction (vs corundum) and wear depth per distance of the solar selective coatings as a function of deformation depth, it can be clearly seen that the lower the deformation depth lower the friction and wear damage. This is possibly attributed to higher impact of the top SiO_2 layer. In addition, in the areas where an overlap between meso- and macro- load scale is observed, the calculated deformation depth is similar.

In order to get a better insight on the wear mechanisms under the different motions, contacting conditions and enviroments, used in the previous sections SEM and EDS analysis of the wear tracks on the solar selective coatings was performed. The results are categorized per type of test.

- Reciprocating sliding at different load scales

In to investigate the effect of load scale on the wear mechanisms, SEM analysis of the wear tracks on the surface of the solar selective coatings was performed. From the experimental results three different loading regimes were found. The first one corresponds to low contact pressures in the range of 20 up to 50 MPa (10 up to 50 mN). In this regime, no measurable wear is observed, whereas the main wear mechanisms are mild abrasion and in some localized areas rupturing of the top SiO_2 layer was observed.

The second one corresponds to average contact pressures in the range of 95 up to 150 MPa. The wear mechanisms observed are mild abrasion of the coating (abrasive lines in Fig.7 23a), localized areas rupturing of the top SiO_2 layer and formation of an oxide based tribo-layer (as indicated in Fig. 7.23b). Thus in this loading regime Quinn's oxidation model can be successfully applied. In addition, the tribological phenomena are similar but accelerated in comparison to those observed in the low contact pressure regime. This means that higher contact pressure can be effectively used to accelerate the testing procedure/protocol, while maintaining the same wear phenomena.

In the third regime, which corresponds to high contact pressures in the range of 300 up to 550 MPa more severe wear damage was observed. The main mechanisms were abrasion of the coating and rupturing, leading to the formation of debris at the contacting interface.

- Fretting mode II

For the lowest oscillating contact loads of 1 N and 2± 1 N (≈ 272 and 343 MPa) no damage was observed on the surface of the solar selective coatings. This indicates that for minor vibrations the risk of failure is extremely small. Only in the case of applied loads above 5 N and contact pressures above 400 MPa, can wear damage be generated due to vibrations.

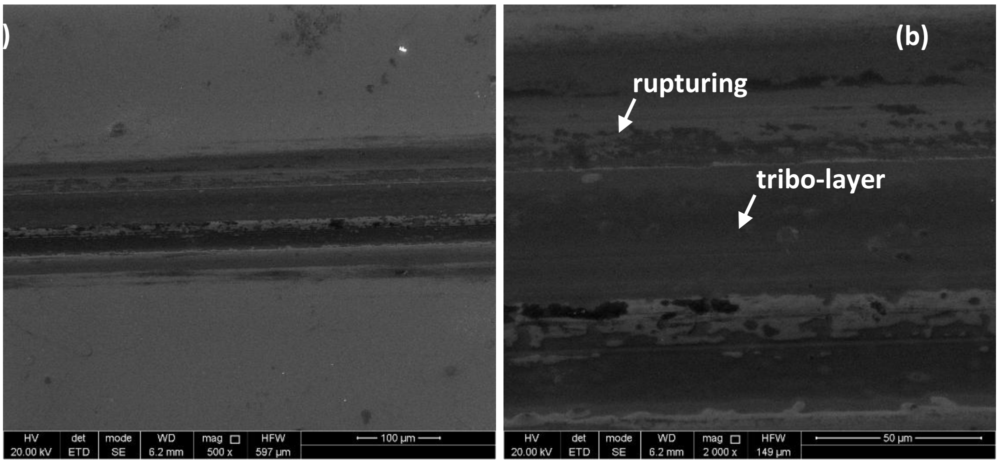


Figure 23: Wear track on solar selective coatings after reciprocating sliding at 95 up to 150 MPa.

- High temperature sliding

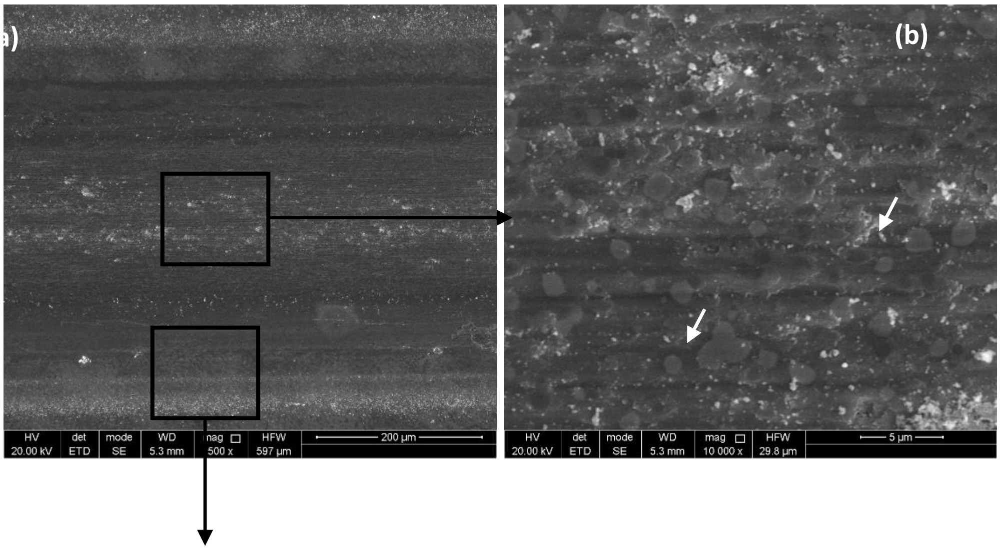
Analysis of the wear track on solar selective coating after isothermal wear testing at 150 °C are presented in Fig. 24. Ploughing lines indicate the existence of an abrasion wear mechanism, Fig.

7.24a. Analysis in the middle of the track at higher magnifications showed localised oxidation of the coating (as indicated in Fig. 24b). Indeed according to Quinn’s model, an oxide film can grow on the surface of the material, until it reaches a critical thickness (around 70 nm as calculated by FIB microscopy) After reaching this thickness the oxide film ruptures and oxide particles are generated. Some debris can be observed in Fig. 24b. Analysis on the edges of the track showed the formation of micropores (Fig. 24c,d). These micropores are attributed to the diffusion of oxygen through localized defects of the SiO_2 layer. Similar observations were also made by ECP.

Analysis of the wear track on solar selective coating after isothermal wear testing at 400 °C was performed. Ploughing lines indicate the existence of an abrasion wear mechanism. Analysis in the middle of the track at higher magnifications showed extensive oxidation and the formation of a tribolayer. Deformation and rupturing of the tribolayer can also be observed. Furthermore delamination of the coating can be observed at the edges of the track). The reason that more intense damage is observed during isothermal testing at 400 °C is actually twofold:

1. Increase of heating leads to a significant decrease of the mechanical strength of molybdenum based materials.

2. Above 460 °C the oxidation mechanism of molybdenum changes from parabolic to linear. In our case despite the fact that testing was performed at 400 °C, at the contacting interface higher temperatures can be reached due to the frictional forces.



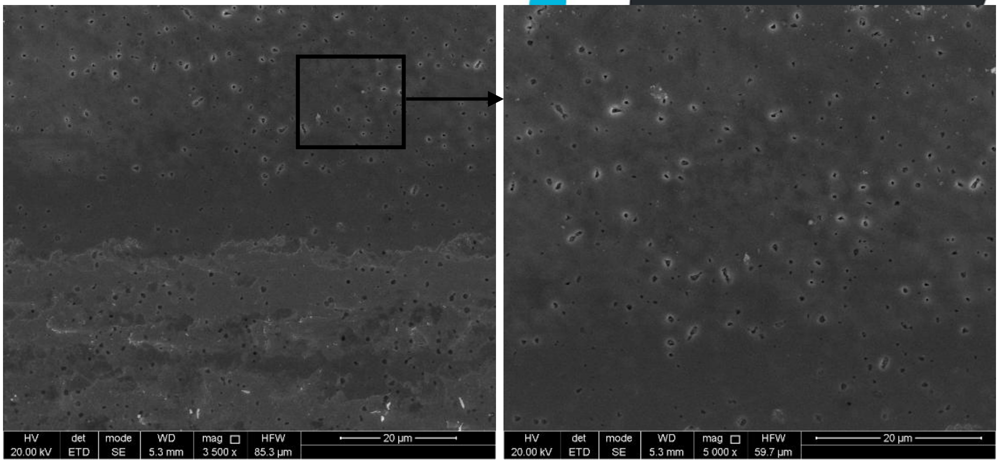


Figure 24: SEM images of wear track on solar selective coating after isothermal wear testing at 150 °C

Finally the tests performed allowed to obtain the wear rate of the coatings by analyzing the dissipation of energy. The wear rate of the solar selective coatings was calculated to be 8 10^{-6} µm/cycle or is 8 10^{-6} µm/s. A linear relationship between the dissipated energy and wear on solar selective coatings was observed. From the dissipated energy versus wear loss diagram the wear per unit of dissipated energy can be derived. The wear rate of these coatings calculated is 0.485 µm/J as long as the wear is confined within the coatings. That slope is of large practical interest since it opens a way to a quantifiable evolution and comparison of the wear resistance of different materials under sliding conditions.

Potential impact

The Strategic Energy Technology Plan (SET-Plan) is a strategic plan from the European Commission to accelerate the development and deployment of cost-effective low carbon technologies. This plan comprises measures relating to planning, implementation, resources and international cooperation in the field of energy technology. The SET-Plan identified a series of key challenges that need to be addressed in the next years, not only to meet the 2020 targets, but also to ensure that the EU is on track to address the 2050 ambition of reducing green house gas emissions by 60-80%. In order to be able to achieve the 2020 targets, the EC has defined several key EU technology challenges for the next years, which include the demonstration of commercial readiness of large-scale Photovoltaic (PV) and **Concentrated Solar Power**. Furthermore, the SET-Plan Roadmap on low carbon energy technologies states that up to 15% of the EU electricity will be generated by solar energy in 2020. In order to achieve all these challenging goals, the parabolic trough technology has to be developed with projects like the NECSO project.

According to “Concentrating Solar Power Global Outlook 09”, a joint report published by Greenpeace International, the European Solar Thermal Electricity Association (ESTELA) and IEA SolarPACES, with advanced industry development and high levels of energy efficiency, **concentrated solar power could meet up to 7% of the world’s power needs by 2030** and fully one quarter by 2050. Globally, the CSP industry could employ as many as 2 million people by 2050, which will help save the climate. This is a truly inspiring vision. In Europe, there are already around 1.000 MW connected to the grid, but in order to reach the 30.000 MW needed to meet the 20% target in the year 2020, research and development will be needed.

Under just a moderate scenario, the countries with the most sun resources could together (source: HITECO project):

- Create €17.5 billion investment in 2015, peaking at €92.5 billion in 2050,
- **Create more than 200,000 jobs by 2020**, and about 1.187 million in 2050,
- Save 148 million tonnes of CO_2 annually in 2020; rising to 2.1 billion tonnes in 2050.

However, in order to meet not only the most ambitious goals that fall into an advanced scenario, intensive RTD activities like the ones that the NECSO Project suggests are needed. Nowadays the operation temperature range of CSP systems is limited by the lack of long term thermal resistance of the absorber selective coatings. The successful implementation of this project will allow to reach operating temperatures beyond the current state-of-the-art 400ºC, therefore increasing the efficiency and thus the power level for a given plant size. The performance of a CSP could be multiplied by 1.5 or more.

In addition, a better selection of coatings will allow enlarging the service life of absorber collector tubes. Since ageing and deterioration of the selective absorber coatings is critical on maintenance costs, it is expected that the new characterization methods and standard protocols developed in NECSO will guarantee the performance and durability of absorber collector tubes through the appropriate quality control tests.

As stated in the FP7 NMP Work Programme 2012, this project will improve the competitiveness of European industry and generate knowledge to ensure its transformation from a resource-intensive to a knowledge-intensive industry. Europe is the World leader in the CSP technology, mainly in parabolic trough receivers, and NECSO project will push towards a better understanding of the selective coatings necessary to absorb and convert the solar energy. **Therefore, this project will collaborate to maintain this leadership, to create new jobs, to decrease CO_2 emissions and to reduce our dependence of fossil fuels.**

The results of NECSO project will **lead to innovation in the design of selective coatings and their associated production processes and to improving the performance of these coatings**. In the long term, this will lead to enhanced operational performance of solar receivers. It has to be considered that a typical CSP plant of 50 MW supposes an initial investment of around 200-300 M€. And some sources points that 10 GW will be operating in a few years (i.e. around 50000 M€ of initial investment). And the collector tube is the heart of the receiver. For these figures we are talking of around **12000-16000 km of coated tubes**, depending on the technology. Nowadays each 4 m tube is sold by approximately 700 €. Making the calculation, we are dealing with a business of around 3000 M€ in less than 5 years only for the tubes, but being a key element of the whole cylinder parabolic thermosolar plant and its proper performance.

Impact for the SMEs involved in the project

The project has the potential to produce significant scientific and technical impact as its main focus is on the development of testing protocols for solar absorber coatings for high temperature operation in CSP plants. Currently, there is a lack of knowledge on the degradation mechanisms of the solar absorber coatings working at high temperatures. Moreover, there are no standards established for accelerated testing that guarantee the performance of the coatings during the expected lifetime of the coatings (25 years). In this sense the project has produced interesting scientific and technical knowledge on the degradation mechanisms. This new knowledge, together with the testing protocols developed in the project, will also have an impact on the development of new improved materials for this application in order to test their suitability before qualifying for commercial use. These issues are of key importance for new developments that work at higher temperatures than current operating technologies (from current 400ºC to temperatures around 600ºC). The NECSO project is strongly linked to previous HITECO project in which the partners ARIES and TEKNIKER are also involved in the development of a new concept of tube for high temperature CSP collectors. In this sense the outputs of NECSO are of key importance in order to validate the HITECO concepts in relation to the absorber coatings and thus to develop its full commercial potential.

The results of NECSO Project will also create new product opportunities for European instrumentation industry, involved in the project. The successful definition of testing protocols, equipments and standards, as well as the growth of CSP plants during next years will also provoke and increase in the need of analysis of selective coatings. Characterization equipments are necessary for researching entities interested in the development and basic research of selective coatings: universities and research centres. Receiver tubes developers will also need the acquisition of instruments for quality control as well as further developments in the multilayered nanostructured coatings. Finally, parabolic trough plant promoters should also be benefited by the advanced characterisation resulting from the NECSO project, having tools to compare among different suppliers of collector tubes and being able to guarantee a proper performance of the solar receiver during the whole life of the system. With this in mind it is foreseen a great increasing demand for this instrumentation industry mainly dominated by SMEs. In this sense, **The results of NECSO Project will also create new product opportunities for European instrumentation industry**, involved in the project. The successful definition of testing protocols, equipments and standards, as well as the growth of CSP plants during next years will also provoke and increase in the need of analysis of selective coatings. Characterization equipments are necessary for researching entities interested in the development and basic research of selective coatings: universities and research centres. Receiver tubes developers will also need the acquisition of instruments for quality control as well as further developments in the multilayered nanostructured coatings. Finally, parabolic trough plant promoters should also be benefited by the advanced characterisation resulting from the NECSO project, having tools to compare among different suppliers of collector tubes and being able to guarantee a proper performance of the solar receiver during the whole life of the system. With this in mind it is foreseen a great increasing demand for this instrumentation industry mainly dominated by SMEs.

Thus, the SMEs TECNOVAC and CSM, which are not involved directly in the solar business, have delivered testing devices such as the ageing system (TECNOVAC) and the tooling to perform nanomechanical testing on cylindrical samples. Thus, the impact of the project for them is directly linked to the potential use and exploitation of the new developed testing devices and tooling for CSP applications but also in other applications that have similar requirements. in the case of CSM, the tool to measure surface mechanical properties can be used on any application using cylindrical geometry while Thus the ageing system developed by TECNOVAC has possibilities of use and exploitation in other applications such as space applications: Simulating, in small samples, the same conditions (vacuum, gas composition, radiation …) as in different space atmosphere and in thermosolar to study hydrogen tubes permeation. In this sense, the equipment has been promoted by TECNOVAC between companies in CSP field (plant and tube manufacturers and technological centers) as well as between companies in the aerospace market that showed their interest in the ageing system to test components from the plane wings and turbine segments.

Dissemination

Different dissemination actions were carried out during the project targeting different audiences from general public to specialized public. A web page of project and LinkedIn group were set up and different press releases were launched for both general and specialized public with a worldwide diffusion.

Dissemination of results of the project have also been done in scientific forums such as the Solarpaces conferences 2013 and 2014, the most important conference in the CSP field as well as in the ICMCTF 2015 Congress (one of the main ones about thin films) and Local Mechanical Properties (LMP) 2015.

Two scientific papers have been published so far:

- Cachafeiro, H., de Arevalo, L. F., Vinuesa, R., Goikoetxea, J., & Barriga, J. (2015). Impact of Solar Selective Coating Ageing on Energy Cost. *Energy Procedia*, *69*, 299-309. **(ARIES and TEKNIKER)**
- Barriga, J., Ruiz-de-Gopegui, U., Goikoetxea, J., Coto, B., & Cachafeiro, H. (2014). Selective coatings for new concepts of parabolic trough collectors.*Energy Procedia*, *49*, 30-39. **(TEKNIKER and ARIES)**

And after the termination of the project at least 4 more scientific publications in collaboration between the different partners are expected in relation to the mechanical properties of the coatings, the lifetime assessment and the mechanisms of degradation of the coatings.

Other dissemination actions

The results of the project were also disseminated in the following specific forums were the main players and stakeholders in the CSP field are represented

- **Spanish Standardization Association AENOR for tube collectors: AEN/CTN 206/SC/GT2 "Components of the thermosolar electric plant" – Subgroup: collector tube.** Two meetings. A standard procedure for testing solar collector tubes is under development. TEKNIKER is actively involved in the standardization commitee and the results of the project are being considered for the development of the standard. The work on the standardization group is especially relevant taking into current lack of standards on coatings for CSP and the advances obtained in the NECSO project have been very positively received by the working group.

- **STE-STAGE - EURPEAN ENERGY RESEARCH ALLIANCE (EERA).**

- **TECNOVAC** has organized visits for companies and technological centers involved in the CSP business and in aerospace in order to show the ageing system 3 and its potential to be used in these applications.

NECSO Workshop

The most important dissemination action carried out in the project was the organization of a final workshop of the project with presentations from TEKNIKER, ECP-CRSA, NIC and CSM on the results of the project. 3 additional presentations from outside the consortium as invited talks carried out by important players in the industry including solar plant builders (ABENGOA), collector manufacturers (ARCHIMIDE) and Technological centers (DLR). All the main players in Europe were represented with attendees in the workshop.

Figure 25. Attendees to the NECSO Workshop

Exploitation

The different results of the project were analyzed in order to study their potential exploitation in the next future. 12 results have been identified to have a potential of exploitation.

All the exploitable results are included in the Use and dissemination of Foreground section. The main exploitable results include: New testing devices (Sample holder for cylindrical samples, ageing chamber for high temperature and controlled atmosphere), coatings and know-how on is development, and the different services associated to the characterization of the coatings including oxidation protocols, mechanical properties, assessment, tribological protocols, methodology to measure solar selectivity in curved samples and lifetime assessment of the coatings. The perspectives of exploitation of the coatings and characterization methods are good according to interest showed by the attendees to the NECSO workshop in the results obtained and the characterization methods and protocols developed. Nevertheless in order to be properly exploited the promotion of these methodologies and protocols to become standards. In this sense the involvement of IK4-TEKNIKER in the **Spanish Standardization Association AENOR for tube collectors: AEN/CTN 206/SC/GT2 "Components of the thermosolar electric plant" – Subgroup: collector tube** is very important to promote the results.

On the other hand, it is worth to point that the dissemination actions carried out targeting the main players in the CSP industry has already paid off as some of the results are already under exploitation before the termination of the project. IK4-TEKNIKER has already started to exploit 2 results:

- **Know-how on processing solar selective coatings:** Using the know-how generated in the project IK4-TEKNIKER has developed and deposited solar selective coatings for a company in CSP sector to be used in an experimental collector. 11 tubes were produced.

- **New protocols to measure solar selectivity values in curved samples:** The new protocols developed to measure the solar selectivity values have generated the interest of both solar plant builders (to assess the properties of their potential suppliers) and tube developers. In that sense, services of charaectirisation on curved samples to both plant builders and tube manufacturers have been provided by IK4-TEKNIKER nad are expected to continue in the next future.

The fact that some of the results are already under exploitation (that proves the interests of the CSP industry on the results obtained in NECSO) together with the positive feed received from the main players during the NECSO workshop and the involvement of NECSO partners in key standardization groups allows to have an optimistic view on the exploitation of NECSO results in the short-mid term.

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