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Accelerated Aging Tests on ENEA-ASE Solar Coating for Receiver Tube Suitable to Operate Up to 550 °C

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Abstract. A patented solar coating for evacuated receiver, based on innovative graded WN-AlN cermet layer, has been optically designed and optimized to operate at high temperature with high performance and high thermal stability. This solar coating, being designed to operate in solar field with molten salt as heat transfer fluid, has to be thermally stable up to the maximum temperature of 550 °C. With the aim of determining degradation behaviour and lifetime prediction of the solar coating, we chose to monitor the variation of the solar absorptance α_s after each thermal annealing cycle carried out at accelerated temperatures under vacuum. This prediction method was coupled with a preliminary Differential Thermal Analysis (DTA) in order to give evidence for any chemical-physical coating modification in the temperature range of interest before performing accelerated aging tests. In the accelerated aging tests we assumed that the temperature dependence of the degradation processes could be described by Arrhenius behaviour and we hypothesized that a linear correlation occurs between optical parameter variation rate (specifically, $\Delta \alpha_s/\Delta t$) and degradation process rate. Starting from $\Delta \alpha_s/\Delta t$ values evaluated at 650 and 690 °C, Arrhenius plot gave an activation energy of 325 kJ mol⁻¹ for the degradation phenomenon, where the prediction on the coating degradation gave a solar absorptance decrease of only 1.65 % after 25 years at 550 °C. This very low α_s decrease gave evidence for an excellent stability of our solar coating, also when employed at the maximum temperature (550 °C) of a solar field operating with molten salt as heat transfer fluid.

INTRODUCTION

In "parabolic trough" technology for concentrating solar plants, optimization and fabrication of effective and thermally stable solar coatings for evacuated receiver tubes represent the most challenging technological goal in order to maximize the photo-thermal conversion efficiency of a solar field operating at high temperature [1,2,3,4,5]. Specifically, for solar plants utilizing molten salts entering at 290°C in the solar field and coming out at 550°C, it is essential to assure a very low thermal degradation rate of the solar coating inside the glass envelop under vacuum. At this purpose, since 2005 ENEA laboratories developed and patented solar coatings suitable to very high temperature applications [6,7]. In particular, starting from the CERMET-based thin film technology, an innovative solar coating was designed, fabricated and characterized [8]. This coating consists of the following thin film stratification: Tungsten (α phase) / Graded CERMET WN-AlN / AlN / SiO₂. Solar absorptance (α_s) higher than 95% and thermal emittance (ε_{th} evaluated at 550 °C) lower than 13% were obtained by means of a very careful optimization procedure in modelling and fabricating the above described solar coating. This coating, subjected to thermal annealing cycles under vacuum (10⁻² Pa) at the temperature of 580 °C for a total duration of three months, did not show any appreciable modification in terms of photo-thermal parameters (α_s and ε_{th}). Archimede Solar Energy (ASE) company has been utilising this ENEA patented solar coating for producing receiver tubes suitable to high temperature employment.

However, starting from the consideration that the durability of this strategic component strongly determines the cost-performance ratio of a solar thermal power plant, accelerated aging tests of the coated steel tubes at elevated temperatures, higher than the operative temperature in service mode, have to be carry out to predict its lifetime.

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Furthermore, a more substantial aim consists in determining the degradation behaviour where a time dependent prediction of the photo-thermal parameters is to be given.

Two different investigation approaches can be taken into account for determining degradation behaviour and lifetime prediction of a solar coating operating at high temperature under vacuum : i) a direct method where chemical, physical and structural variations have to be monitored by means of specific characterization techniques (SEM, TEM, AFM, XRD, RAMAN, SIMS, etc.) after each annealing cycle; ii) an indirect method where variations of some solar coating parameters (α_s , ε_s , electrical resistivity, strength adhesion, etc.) testify to chemical, physical and structural variations of the materials constituting the solar coating and can be correlated to degradation phenomena.

Generally, standard procedures have been codified for aging tests on thermal panels [9]. However, starting from the consideration that solar coatings for receiver tube are very complex thin film stratifications and they have to operate at very high temperatures, the choice of a correct standard procedure to perform aging tests represents a critical point in durability evaluation. Some approaches have been proposed to assess coating stability for high temperature employment, utilizing different aging test procedures [10,11]. In practice, these methods consist in obtaining prediction of photothermal parameter degradation at a specific aging time (e.g. 20 years) starting from measurements at higher temperatures (accelerated tests) and utilizing an activation energy obtained from Arrhenius law.

In this paper we chose to follow the indirect method and, in particular, we monitored the variation of the photothermal parameters (α_s and ε_{th}) after each thermal annealing cycle carried out at accelerated temperatures under vacuum. Variation of these parameters during accelerated thermal cycles is very effective in indicating the seriousness of the chemical-physical degradation phenomena and, if opportunely handled, it can give long-term degradation trend with good approximation on the lifetime prediction of the solar coating. However, in this paper we strongly emphasize that this prediction method has to be coupled with a preliminary thermal analysis in order to give evidence for any chemical-physical coating modification in the temperature range of interest before performing accelerated aging tests. This innovative approach can be considered very effective to avoid rough mistakes in evaluating degradation behaviours.

EXPERIMENTAL

Metallic, ceramic and cermet films of the spectrally selective solar absorbers were deposited by sputtering technique using a proprietary planar magnetron sputtering system equipped with process chamber and load lock chamber [6].

Tungsten sample were deposited with thickness of about 130 nm by DC sputtering mode on SS and glass substrate at Ar pressure of 0.3 Pa and with 3.7 W/cm^2 power density applied to the cathode. The sweep and spin velocity of the tube-holder were 60 cm/min and 60 rpm, respectively.

WN-AlN cermet samples were deposited by reactive sputtering under an atmosphere containing nitrogen and argon, moving and rotating the tube-holder back and forth respect to Al target and W target arranged in two opposite sides (co-sputtering mode). The sweep and spin velocities of the tube-holder were 400 cm/min and 60 rpm, respectively. In these cermet materials, WN represents nitride with metallic behaviour and AlN represents nitride with ceramic behaviour. In order to obtain cermet layers with different metal content, samples were deposited applying a constant cathode power density of 7.4 W/cm² to the Al target, whereas the WN component was deposited at decreasing cathode power density applied to the W target. Four cermet layers were deposited at the following power densities: 2.23W/cm², 1.67W/cm², 1.11W/cm² and 0.56W/cm². The thicknesses of these samples were 20, 40, 60 and 80 nm, respectively.

The AlN layer was grown at $Ar+N_2$ pressure of 0.43 Pa and with power density of 5.55 W/cm² applied to Al cathode. The sweep and spin velocities of the tube-holder were 400 cm/min and 60 rpm, respectively.

Similarly, a SiO₂ layer was deposited at $Ar+O_2$ pressure of 0.43 Pa and with power density of 3.7 W/cm² supplied to Si cathode. The sweep and spin velocities of the tube-holder were 400 cm/min and 60 rpm, respectively.

Refraction index (n) and extinction coefficient (k) of each deposited layer were estimated by ellipsometric technique using the Jobin-Yvon mod. UVISELTM spectroscopic phase modulated ellipsometer. This instrument allows to operate in the wavelength range $0.25 - 1.65 \mu m$.

Reflectance measurements in the range $0.2 - 2.5 \mu m$ were carried out by UV-Visible-NIR spectrophotometer, using a double-beam Perkin-Elmer mod. Lambda 900 instrument equipped with a 10 cm diameter integrating sphere.

Infrared reflectance measurements were carried out by a Fourier Transform spectrophotometer in the wavelength range $1.7 - 17 \mu m$, using a BRUKER mod. EQUINOX 55 equipped with an MCT detector and a 7.5 cm diameter integrating sphere, coated with diffuse reflecting gold, where the reference mirror was a gold film evaporated on very flat stainless steel plate.

The thickness of each layer was mechanically measured on films grown on glass substrate using a Tencor P-10 surface profiler.

Differential Thermal Analysis (DTA) coupled with Thermogravimetric Analysis (TGA) was performed with a TA Instruments SDT Q600 in order to study the thermal behaviour of the solar coating structure and the thermal decomposition process of the material. The measurements were carried out under argon atmosphere on powders of the material at a heating rate of 20 °C/min from room temperature to 1000 °C

Annealing cycles under vacuum were performed at a pressure value of 10^{-2} Pa by using a Linn programmable oven equipped with a mechanical-turbomolecular pumping group.

RESULTS AND DISCUSSIONS

The innovative solar coating utilized for the annealing tests was composed by a tungsten nitride-aluminum nitride (WN-AlN) graded cermet layer (absorbing layer) deposited between a back reflector of W and an antireflection filter, formed by materials with high transparence and appropriate refraction index such as silica (SiO₂), aluminum nitride (AlN) and WN-AlN cermet with very low metal volume fraction (VLMVF).

Tungsten back reflector was deposited on SS for optical parameter estimation and on glass substrate for an accurate thickness measurement. In the same way, the cermet with highest metallic volume fraction (HMVF) was deposited both on glass for an accurate thickness measurement and on the SS/W structure for the estimation of the optical parameters. Following the same approach, each new cermet layer with lower metallic power density was deposited on glass for an accurate thickness measurement and on the preexisting structure with cermet layers at higher metallic content for the estimation of the optical parameters. This procedure allowed to estimate n and k parameters in the actual growth condition of the cermet layers during the solar coating fabrication, taking into account the influence of the underlying layers on structure and morphology of the cermet layer. This procedure proved very accurate in determining the complex refraction index of each single cermet layer [8]. The same procedure was adopted to estimate the optical parameters of AlN and SiO₂ deposited in sequence on SS/W/Graded CERMET structure.



FIGURE 1. Ellipsometrically determined (a) spectral refractive index and (b) spectral extinction coefficient for four WN-AIN cermet layers having different metal contents. The metal content (WN) decreases moving from cermet#1 towards cermet#4.

The optical parameters ellipsometrically determined for each layer (see Fig. 1) were organized in a database and utilized to design and fabricate a solar coating optimized for the operating temperature of 550°C. A homemade program was utilized to generate hundreds and hundreds of reflectance curves related to different thin film

stratifications by varying thickness of absorbing layer, shape of metallic depth profile inside this layer and thickness of the two layers constituting the antireflection filter. The coating design, with the most promising simulated spectral reflectance for operating at temperature up to 550°C, was chosen and the optical parameters of this design were imported in Macleod to further improve the performance. The improvements were obtained by means of the following modifications: i) introducing a four-layer antireflection filter, formed by AlN, VLMVF cermet, AlN and SiO₂, in place of the bi-layer filter included in the homemade program; ii) modifying the depth profile of the metallic content inside the graded cermet to obtain a better optical matching between the graded cermet layer and the new antireflection filter.



FIGURE 2. Spectral global reflectance of the optimized solar coating, in the wavelength range $0.2 - 17 \mu m$, obtained by spectrophotometric measurements. AM1.5 solar spectrum and black body irradiance at 550 °C are also reported.

The design output of this second refinement was transformed in a "recipe" for the sputtering apparatus and N samples of the optimized solar coating were produced for the annealing tests. In Fig. 2 the spectral reflectance of the optimized solar coating is reported.

Solar absorptance (α_s) and thermal emittance (ϵ_{th}) of the optimized solar coating, "as grown" and after each annealing cycle, were determined starting from the spectrophotometric spectral reflectance performed in the range $0.2 - 2.5 \mu m$ and in the range $0.2 - 17 \mu m$, respectively.

$$\alpha(\theta) = \frac{\int_{0.2}^{2.5} Gb(\lambda) [1 - R(\lambda, \theta)] d\lambda}{\int_{0.2}^{2.5} Gb(\lambda) d\lambda}$$
(1)

$$\varepsilon(\theta, T) = \frac{\int_{0.2}^{17} E(T,\lambda) [1 - R(\lambda, \theta)] d\lambda}{\int_{0.2}^{17} E(T,\lambda) d\lambda}$$
(2)

With the aim of determining degradation behaviour and lifetime prediction of a solar coating operating at high temperature under vacuum, in this paper we chose to follow the indirect method and, in particular, we monitored the variation of α_s and ε_{th} after each thermal annealing cycle carried out at accelerated temperatures under vacuum. In the accelerated aging tests we assumed that the temperature dependence of the degradation processes could be described by Arrhenius behaviour and we hypothesized that a linear correlation occurs between optical parameter variation rate (specifically, $\Delta \alpha_s / \Delta t$) and degradation process rate.

For diffusion or other thermally activated chemical-physical phenomenon, the degradation process rate \mathbf{r} can be expressed as:

$$r = A * e^{\left(-\frac{E_a}{RT}\right)} \tag{3}$$

where E_a is the activation energy, A is the characteristic parameter of the process (for diffusion, A is D_0 the maximum diffusion coefficient at infinite temperature expressed as $m^2 s^{-1}$) and T is the temperature utilized for accelerated annealing test.

We have to suppose that the degradation process advancement, inducing optical modification of the solar coating, is proportional to the α_{solar} variation:

$$r \sim \frac{\Delta \alpha_s}{\Delta t} \tag{4}$$

$$\frac{\Delta \alpha_s}{\Delta t} \sim e^{\left(-\frac{E_a}{RT}\right)} \tag{5}$$

$$\ln\left(\frac{\Delta\alpha_s}{\Delta t}\right) \sim -\frac{E_a}{R} * \frac{1}{T} \tag{6}$$

So, Arrhenius plot consists in graphing Ln ($\Delta \alpha_s / \Delta t$) vs 1/T were $\mathbf{E}_a / \mathbf{R}$ is the slop of the linear fit.

However, to pursue the aim of correctly performing accelerated thermal tests at temperatures from 550 up to 700 °C, it is fundamental to verify if chemical-physical processes start and occur in this temperature range. The presence of chemical-physical processes starting at a temperature higher than the maximum operating one (550 °C) and lying inside the range of interest (550 to 700 °C) utilised for accelerated thermal tests should lead to two rough evaluation mistakes : i) A degradation process, not occurring at normal operation temperatures (< 550 °C) of the receiver in the solar field, will be erroneously monitored and evaluated, whereas only the acceleration of degradation processes having a starting point at temperatures lower than 550 °C have relevance for our aim and have to be monitored and evaluated; ii) A degradation process (for example, crystallization of a component constituting the solar coating) having a starting point at a very high temperature (for example, 670 °C) should give a degradation behaviour at the accelerated temperature of 690 °C not comparable with the degradation behaviour obtained at a lower accelerated temperature of 650 °C (no crystallization), making not more applicable Arrhenius method for determination of a well-defined activation energy and related aging behaviour. The above described observations lead us to consider indisputable and absolutely necessary the implementation of a preliminary thermal analysis on the solar coating, able to give us any information on all the thermally activated chemical-physical modifications occurring in the temperature range of interest. As preliminary investigation before performing accelerated thermal tests, Differential Thermal Analysis (DTA) coupled with Thermo-Gravimetric Analysis (TGA) was carried out to give evidence for any exothermic or endothermic process such as: melting, sublimation, crystallization, crystalline rearrangement, chemical reaction, etc. In Fig. 3 DTA-TG thermograms are reported for the structure W/WN-AlN/AlN/SiO₂. The DTA thermogram shows some endothermic and exothermic transitions. It was found a transition at 100 °C attributable to removal of adsorbed water, where the corresponding mass loss percentage in the TG curve is about 0.4%. Furthermore, a small exothermic transition in DTA curve appears at 208 °C. As there is not any corresponding mass loss of the sample in TG curve, it can be attributable to structure rearrangement or phase transformation in the solid, very probably the crystalline rearrangement of a little amount of β -W inside the predominant α -W structure. DTA and TGA behaviours do not show any chemical-physical process start in the temperature range of interest (550 to 700 °C, yellow region in the graph). Only at temperature higher than 800 °C a strong pyrolysis phenomenon occurs where tungsten nitride decomposes with gaseous nitrogen loss.



FIGURE 3. Differential Thermal Analysis (DTA) coupled with Thermo-Gravimetric Analysis (TGA) carried out on ENEA-ASE solar coating . The yellow region in the graph represents the temperature range utilized for performing accelerated thermal aging tests. In this temperature range there is not any chemical-physical process start.

Ultimately, DTA characterization results give us the useful information that 650 °C and 690 °C are two appropriate temperatures to perform accelerated thermal tests without rough evaluation mistakes. In the accelerated aging tests at the temperatures of 650 and 690 °C, higher than the maximum temperature (550 °C) utilized in the operative mode of the receiver, we assumed that the temperature dependence of the degradation processes could be described by Arrhenius behaviour and we hypothesized that a linear correlation occurs between optical parameter variation rate (specifically, $\Delta \alpha_s / \Delta t$) and degradation process rate.

Solar coating samples deposited on stainless steel sheet were subjected to thermal annealing cycles inside a programmable oven operating under vacuum: i) at 650 °C for a total duration of 550 hours; ii) at 690 °C for a total duration of 105 hours. Two samples of the optimized solar coating have been tested for each annealing temperature. At 650 °C the degradation rate $\Delta \alpha_s / \Delta t$ resulted about 3.5×10^{-3} hr⁻¹ and at 690 °C about 2.3×10^{-2} hr⁻¹ (see Fig.4).



FIGURE 4. Solar absorptance variation as a function of annealing time for thermal cycles carried out at 690 °C under vacuum. Two samples of the optimized solar coating have been tested and related results are reported.



FIGURE 5. (a) Arrhenius plot Ln ($\Delta \alpha / \Delta t$) vs 1/T where the slope allows to determine the activation energy E_a of the degradation process. Two samples of the optimized solar coating have been tested for each annealing temperature and related results are reported (b) Decrease prediction of the solar absorption vs years operated at 550 °C for a degradation phenomenon with an assigned activation energy of 325 kJ mol⁻¹.

Previously, we supposed that the optical degradation rate could be treated by the following Arrhenius behavior

$$\frac{\Delta \alpha_s}{\Delta t} \sim e^{\left(-\frac{E_a}{RT}\right)} \tag{7}$$

where $(\Delta \alpha_s / \Delta t)$ is the optical degradation rate and E_a is the activation energy of the chemical-physical degradation process associated to observed optical modifications. Starting from optical degradation rate $(\Delta \alpha_s / \Delta t)$ values evaluated at 650 and 690 °C, Arrhenius plot (see Fig. 5a) gives an activation energy of 325 kJ mol⁻¹ for the degradation phenomenon, with a pre-exponential A value of $4*10^{16}$. The calculated E_a value well agrees with selfdiffusion phenomenon of tungsten at the grain boundary ($E_a = 300 \text{ kJ mol}^{-1}$). If we hypothesize that the receiver will operate for 8 hours/day at the maximum temperature with a solar absorptance degradation rate determined by Arrhenius law (with $E_a = 325 \text{ kJ mol}^{-1}$), it is possible to simulate 25-years aging at the operating temperature of 550 °C, where the prediction on the coating degradation gives a solar absorptance decrease of only 1.65 % (see Fig. 5b). This very low decrease gives evidence for an excellent stability of our solar coating employed at high temperature.

CONCLUSIONS

By means of a very careful optimization procedure suitable to model optical filters, an innovative solar coating for high temperature employment has been optimized and realized with the following layer sequence: Tungsten (α phase) / Graded CERMET WN-AIN / AIN / SiO₂. Solar absorptance (α _s) higher than 95 % and thermal emittance (ε _{th} evaluated at 550 °C) lower than 13 % were obtained.

With the aim of performing accelerated thermal tests at temperatures higher than the maximum operating one (550 °C), in this paper we chose to follow the indirect method and, in particular, we monitored the variation of the solar absorptance α_s after each thermal annealing cycle carried out at accelerated temperatures (650 and 690 °C) under vacuum. However, this method for long-term aging prediction was coupled with a preliminary thermal analysis in order to give evidence for any chemical-physical coating modification in the temperature range of interest before performing accelerated aging tests. Differential Thermal Analysis (DTA) coupled with Thermo-Gravimetric Analysis (TGA) was carried out to give evidence for any esothermic or endothermic process. DTA and TGA behaviours did not show any chemical-physical process start in the temperature range of interest (550 to 700 °C). This information was very useful to assure that the results of the accelerated tests, carried out at 650 °C and 690 °C, could be handled without serious evaluation mistakes, as potential degradation phenomena started at

temperatures lower than the maximum operating temperature (550 °C). In the accelerated aging tests we assumed that the temperature dependence of the degradation processes could be described by Arrhenius behaviour and we hypothesized that a linear correlation occurs between optical parameter variation rate (specifically, $\Delta \alpha_s / \Delta t$) and degradation process rate. Solar coating samples deposited on stainless steel sheet were subjected to thermal annealing cycles inside a programmable oven operating under vacuum: i) at 650 °C for a total duration of 550 hours; ii) at 690 °C for a total duration of 105 hours. At 650 °C the degradation rate $\Delta \alpha_s / \Delta t$ resulted about $3.5*10^{-3}$ hr⁻¹ and at 690 °C about $2.3*10^{-2}$ hr⁻¹.

Starting from optical degradation rate ($\Delta \alpha_s / \Delta t$) values evaluated at 650 and 690 °C, Arrhenius plot gave an activation energy of 325 kJ mol⁻¹ for the degradation phenomenon. This value well agreed with self-diffusion phenomenon of tungsten at the grain boundary ($E_a = 300 \text{ kJ mol}^{-1}$) and it can be utilised to simulate 25 years aging at the operating temperature of 550 °C, where the prediction on the coating degradation gave a solar absorptance decrease of only 1.65 %. This very low α_s decrease gave evidence for an excellent stability of our solar coating, also when employed at the maximum temperature (550 °C) of a solar field operating with molten salt as heat transfer fluid.

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