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SolarPACES 2014Towards standard testing materials for high temperature solar
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Abstract

Solar thermal technology for the production on electricity is one of the current technological challenges. In concentrating solar power (CSP) plants, in order to achieve high power it is required to use a high operating temperature to reach high conversion efficiencies. The majority of today's commercial solar thermal power plants are based on the parabolic trough collector technology with operating temperature around 400°C. However, the technology of solar tower is used in order to maximize the efficiency of the CSP plants. This technology reaches an operating temperature higher than 1000 °C and the development of high temperature receivers that work in this temperature ranges is still in its early stages. The fundamental problems observed are related to materials durability and reliability.

The main objective of this paper has been to develop testing methods for solar receivers which guarantee their reliability and durability under demanding working conditions of high solar concentrating technology. Based on a revision of published or draft Standards, a qualification test methodology for durability tests has been developed.

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1. Introduction

Due to the high investment costs of new component and material for solar thermal concentration technologies, investors are asking for guaranties for their power plant economical feasibility studies. But nowadays, there is no International Standard for component and material characterization and durability test methods due to the few solar tower power plant commercial experiences. The International committee for Standardization of CSP plants components, IEC TC 117, has just started to work on a testing standardization for solar plants and in particular for central tower plants.

One of the key aspects for solar power concentration materials is to determine the degradation during life-time under operating conditions through accelerated ageing test procedures. This analysis of testing methodologies for receiver is part of the Spanish project “MIRASOL” for fundamental research on materials for solar tower receiver. In this paper, different possible agents processes will be contemplated, and some possible testing methodologies will be defined.

Nomenclature

v	heating and cooling rate
t	time
t _{ac}	time accumulated
T	temperature
T _f	final temperature
T _i	initial temperature

2. Background

This paper gives a review of the state of the art of the possible accelerated ageing test that could be applied to receiver material candidates.

2.1. State-of-the-art of Standard for solar thermal components

Relevant existing test methodologies from Standard have also been revised for solar components.

In Table 1 the tests performed to solar thermal collector for domestic hot water applications are summarized.

Table 1. Test methods for solar thermal collector

Standard	Test	Variables	Main instrumentation	Description
ISO 9806 [1]	High-temperature resistance	Absorber Temp.	Temp. sensors	Determination whether a collector can withstand high temperature and irradiance levels without failure and under non-operation conditions
		Global irradiance	Pyranometer	
		Ambient Temp.	Anemometer	
		Wind speed	Data logger	
ISO 9806 [1]	Exposure test	Global irradiance	Pyranometer	Consists in exposing the collector until a minimum of days having passed a minimum irradiation and a minimum of hours to a minimum irradiance level.
		Ambient Temp.	Temp. sensors	
		Rainfall	Rain gage	
			Data logger	
ISO 9806 [1]	External thermal shock test	Global irradiance	Pyranometer	Consists in exposing the collector having passed a minimum irradiance and then cooled by a water spray.
		Ambient Temp.	Temp. sensors	
		Water spray Temp. and flow rate	Flowmeter	
			Water spray device	
			Data logger	

From all those tests, both the thermal shock test and the high temperature test could be adapted but with different testing conditions for the receiver to resist temperature up to 1200°C.

In Table 2 the durability tests performed to solar reflector are summarized.

Table 2. Test methods for Reflector

Reference	Test	Variables	Main instrumentation	Description
ISO 6270-2CH [2]	Thermal Cycling	Temperature Relative humidity	Temperature sensor Relative humidity sensor Power supply Data logger	Determination of the ability of the solar component to withstand thermal mismatch, fatigue and other stresses caused by repeated changes of temperature. The samples are subjected to thermal cycling within a climatic chamber
DIN 50018 [3] / ISO 6988 [4]	Humidity Freeze Test	Temperature Relative humidity	Temperature sensor Relative humidity sensor Power supply Data logger	Determination of the ability of the solar component to withstand the effects of high temperature and humidity followed by sub-zero temperatures.

From all those tests to thermal cycling test and the humidity test could be used but with different testing boundary conditions as we want the receiver to be submit to temperature up to 1000°C.

In Table 3 the durability tests performed to solar absorber with selective coating are summarized.

Table 3. Test methods for Reflector

Reference	Test	Variables	Main instrumentation	Description
ISO/DIS 22975-3 [5]	Thermal stability of absorber surface	Absorber absorptance Absorber emittance Temperature Relative humidity	Spectrophotometers (for optical properties) Temperature sensor Relative humidity sensor	Consist of placing the test components in a testing chamber at a specified temperature during a specified time period and then decrease the temperature down to room temperature before measuring optical properties.
ISO/DIS 22975-3 [5]	Resistance to condensed water of absorber surface	Absorber absorptance Absorber emittance Temperature Relative humidity	Spectrophotometers (for optical properties) Temperature sensor Relative humidity sensor	Consists of placing the test components in a testing chamber at a specified temperature and high relative humidity until condensation of water is first observed and then keep the sample at room conditions for 2 hours before measuring optical properties.
ISO/DIS 22975-3 [5]	Corrosion resistance to high humidity air containing sulphur dioxide of absorber surface	Optical properties of the solar absorber (absorptance and emittance), temperature and relative humidity, flow rate, concentration of sulphur dioxide in the air flow at the outlet	Climatic chamber with inert materials air flow and sulphur dioxide injection system temperature and relative humidity sensor gas analyzer	Consist of placing the test panels in a testing chamber at a specified temperature a high-humidity and sulphur dioxide - air composition, then keep it at those conditions for the specified time period and then keep the sample at room conditions for 2 hours before measuring optical properties.

Those tests make sense for a selective coating but in our case the receiver will suffer temperature much higher. The resistance to condensed water test is similar to humidity test. So we will not use this Standard.

In Table 4 is summarized the durability tests performed to receiver tubes for parabolic trough based on the first draft of Standard written by the Spanish committee AENCTN 206SC117/GT2 – Solar thermal plants components in the Spanish accreditation body AENOR.

Table 4. Test methods for PT Receiver tube

Reference	Test	Variables	Main instrumentation	Description
AENOR committee (Draft)	External thermal shock test	Absorber tube Temp. Water Temp.	Electrical heating elements	Determination of the resistance of the receiver tube to external thermal shock. Consists of several cycles. First, heating the receiver up to the nominal working temperature using electrical heating elements the inside of the receiver. Then, let stabilize during a 30 minutes period. Then cooling the receiver spraying it with deionized water. Finally, drying the receiver with a microfiber cloth.
			Watering system with deionized water with temperature control system.	
			Temperature sensors Electrical power transducers Data logger	
AENOR committee (Draft)	Internal thermal shock test	Absorber tube Temp. Glass envelope tube Temp. Ambient Temp. Surrounding air Temp.	Temperature sensors Electrical power transducers	Determination of the resistance of the receiver tube to internal thermal shock. Consists of several cycles. First, heating the receiver up to the nominal working temperature using electrical heating elements the inside of the receiver. Then, let stabilize during a 30 minutes period. Then cooling the receiver with pressurized air. Finally, let stabilize during a 30 minutes period
			Data logger	

Those tests make sense to the receiver absorber as it has a selective coating and for the glass-metal junctions but our case the receiver will not suffer temperature much higher. So we will not use this reference.

2.2. State-of-the-art of receiver testing

Relevant existing test methodologies have been revised in order to find the more relevant tests and the more useful in order to select an appropriate material candidate as a receiver that could resist high temperature and high solar concentrated radiation.

In the studied references some extreme conditions have been defined for the receiver material. In Boubault et al. (2014) [6] a testing methodology has been defined for material ageing for the following ageing mechanism: corrosion, oxidation, aerosol/water/biofilm deposition, mechanical stresses, or cracks. The degradation of the characteristics of solar radiation absorption and heat transfer to a heat transfer fluid have been studied.

In Boubault et al. (2012) [7], a degradation model has been defined, and different cycles of ageing using a shutter and a solar oven in Promes-Odeillo laboratory. The main degradation considered mechanisms were: irradiance and temperature.

In Rojas-Morin and Fernandez-Reche (2011) [8], a parabolic dish is used to performed degradation cycles.

In the following Table 5 are summarized the testing conditions of durability test for solar receivers.

Table 5 Test methods for solar furnace

Reference	Component tested	Variables measured	Main instrumentation	Boundary Conditions
A. Boubault et al 2014 [6]	Durability of materials	Solar absorptance, thermal diffusivity, effusivity, conductivity of the paint coating, and thermal contact resistance between the coating and the substrate	Solar oven in Odeillo Concentrated radiation up to 1500kW/m ²	Cycles from 104 to 346kW/m ² Duration cycle: 10s to 30s

A. Boubault et al. 2012 [7]	Solar absorber material	Solar bidirectional reflectance and material bulk proprieties (conductivity diffusivity, thermal contact resistance)	Solar oven in Odeillo	Cyclic irradiance of average 250kW/m ² amplitude 50kW/m ² Period: 30s
Rojas-Morin and Fernandez-Reche 2011 [8]	INCONEL	Thermal tensile and compressive stresses	Parabolic dish	500 cycle at a rate of 120 cycles/h Max. temperature: 1025°C
Setien et al. 2013 [9]	Durability and surface heat treatment of materials	Incident solar power, temperature, pressure	Flux sensor Temperature sensor Pressure sensor	Ageing at 700°C Duration: up to 4000 hours
Guillot et al. 2013 [10]	Radiometers	Direct Normal Irradiance (DNI) Temperature Water flow Flux distribution	Heat Flux Gauges Pyrheliometer Thermocouples Coriolis flow meter Data logger	Clear sky conditions: Diffuse < 60W/m ² Stable DNI ± 5W/m ² Wind speed ≤ 18km/h

3. Methodology

From the revision of all the existing testing methodology a selection and an adaptation have been done. This analysis allowed selecting the test methodology that will be used in order to check if a material could be used as a receiver. The testing conditions has been chosen for the requirement of the receiver to resist temperature higher than 1000°C and also taking into account the limitation of equipment available in this project.

A summary of the tests chosen is shown in Table 6.

Table 6 Test methods chosen for the receiver materials

Test	Equipment	Ambient conditions	Measurement	Cycle conditions or duration
High temperature	Tubular oven with air circulation	Oven with constant temperature at 1000°C Temp. 35 ± 2°C Relative Humidity: constant 100%	temperature in the oven, superficial temperature of sample	More of 10 cycles of 10 min
Salt mist	Salt mist chamber	Sprayed solution: demineralized water + 50g/l NaCl (pH 6.5 – 7.2) Condensation rate: 1.5 ± 0.5ml/h on a surface of 80cm ²	temperature, content of NaCl	480 – 3500 hours
Thermal shock	Fresnel lens, thermocouples	Minimum of 10 cycles of 10 min	superficial temperature of sample	If no degradation 10 cycles more, and repeat the same process
High humidity and temperature	Temperature and humidity chamber	Temperatures: -40°C to +85°C Humidity: ambient to 100%	superficial temperature of sample, humidity sensor	Exposition to more than 20 cycles with a 24h

In parts 3.1 to 3.5 are explained the tests to be performed

3.1. High temperature

The resistance to high temperature is tested by exposing the sample to 1000°C during 1, 2, 5 and 10 cycles. The temperatures, dwelling time at 1000°C, and heating and cooling rates of a high temperature cycle are given in Table 7. The equipment available during this project is a small tubular oven (Fig. 1) with a maximum temperature up to 1400°C. The cycles of 10 minutes have been defined to simulate the irradiance change due to clouds on a CSP plant.

Table 7. High temperature test cycle (T_i , T_f : initial and final temperature; v : heating and cooling rate; t : time; t_{ac} : accumulated time).

T_i (°C)	T_f (°C)	v (°C/s)	t (s)	t_{ac} (s)
25	800	70	11	11
800	1000	5	40	51
1000	1000	0	600	651
1000	600	11	36	687
600	400	4	50	737
400	200	1	200	937

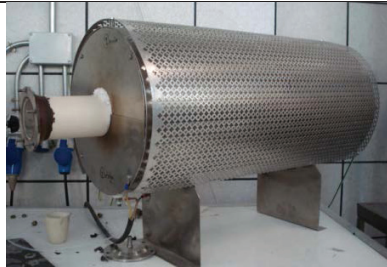


Fig. 1. Tubular oven used for high temperature test at CSIC-ICV

3.2. Salt mist

The resistance to salt mist is tested by exposing the sample to an extreme salty environment that simulates coast location. The test of salt mist corrosion test, based on the European standard UNE-EN 61701:1999, consists on placing the sample in a salt spray chamber (Fig. 2) with a tilt between 15 and 30 degrees during 96 hours and determining the resistance of the sample to the corrosion under salt mist. This test is useful to assess the compatibility of materials, quality and uniformity of protective coatings.



Fig. 2. Picture of the chamber used for salt mist test at CENER.

The sample is introduced in the salt spray chamber at a temperature of 35°C, during a time of 96 hours and in a saline environment created from a dissolution in which the concentration must be 5% of Chloride Sodium NaCl. After completing the test, each sample is clarified and cleaned with distilled water and a final inspection is realized to verify the level of sample degradation.

3.3. Thermal shock

The equipment available during this project is a Fresnel lens mounted on a solar tracker (Fig. 3). The lens is positioned on an aluminum frame with a polar axis orientation. The lens movement from east to west is controlled automatically by a computer and the solar height is hand positioned. The physical characteristic of the lens were previously reported [13, 14]. As the solar irradiance levels are different from day to day it is necessary to install a solar irradiance attenuation device. By this way, a constant solar irradiance value can be achieved for most of the year except the days where solar irradiance is under the established level. It is then possible to work at a constant average irradiance and which would result on the controlled application of constant power density on the sample.

The study considers the use of a wide range of ferrous and nickel-base refractory alloys, as well as some intermetallic alloys for high-temperature receiver applications. The materials may be classified into different groups as: a) refractory steel; b) iron aluminides, molybdenum silicide or boro-silicide intermetallic, and c) nickel-base super alloys.

Samples of 1 cm square section and 5 mm thick were subjected to thermal shock receiving concentrated solar radiation during heating/ cooling cycles. Sample temperature while cycling was registered with a chromel/alumel thermocouple introduced in a side hole in the sample. Discovering the lens initiates the shock cycle by concentrating solar radiation fast heating up to 1200°C; after maintaining 10 min at this temperature shock cycle ends by fast cooling down to 200°C covering the lens to interrupt solar radiation on the sample. A small specimen undergoes much faster heating/cooling than a real device. This one, because of its larger size, spreads the energy applied over its larger mass and so heating/cooling speeds will always be lower. Any material that exceeds the thermal shock test conducted on small specimens will demonstrate its ability to be employed at full scale



Fig. 3. Pictures of the Fresnel lenses used for thermal shock test (a) un-mounted in CENER (b) on solar tracker in CSIC-CENIM

3.4. High humidity and temperature

The purpose is to determine the capacity of the sample to withstand the long term effects of humidity penetration. The sample is placed at ambient temperature inside the chamber (Fig. 4) without any preconditioning.



Fig. 4. Pictures of relative humidity climatic chambers in CENER

Chamber conditions are placed to a temperature of $85^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and relative humidity of $85\% \pm 5\%$ during 1000 hours.

3.5. Checking before and after

The quality control to check if the material “passes” or “fails” the tests is based on the measurement of some optical and mechanical properties.

The spectral reflectance curve is measured before and after the durability tests, based on the Standard ASTM E 424 – 71 [11] using a spectroradiometer with integration sphere. The solar absorptance was computed integrating the solar spectrum based on the Standard ISO 9845-1 [12].

For the samples tested, the weight loss and the mechanical proprieties will also be analyzed. The degradation or change of other properties of the material will be also analyzed such as: the weight, the conductivity, the dilatation coefficient of the sample.

4. Results

This article presents the test results of innovative materials that fulfill the requirements for high temperature solar receivers, with optical as well as mechanical properties, based on the durability tests of Table 6

4.1. High temperature tests

The high temperature experiments were performed with three different samples: SiC (used as reference: Ref), and a porous (S1) or a dense (S2) laboratory prepared materials. Figure 5 shows how the samples are placed within tubular furnace and the appearance of the samples during the high temperature treatment. As the dwelling time at maximum temperature increases the samples turn to red (Figure 5 (b) and (c)).

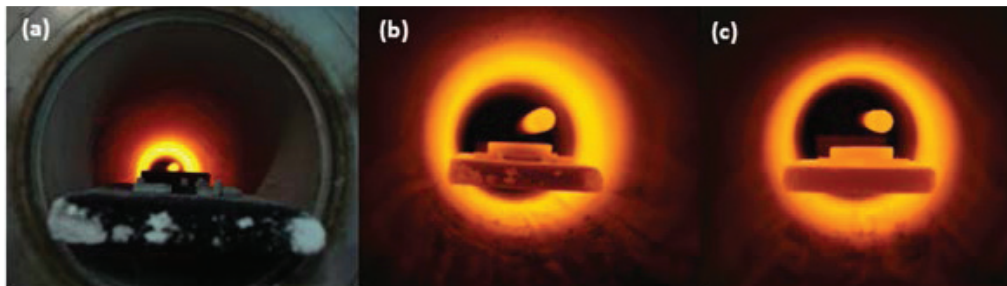


Fig. 5. Details of tubular furnace during the high temperature tests. (a) Starting the test. (b and c) Sample at 1000°C for 0 and 600s.

Figure 6 collects the photographs of samples before and after 1, 2, 5 and 10 high temperature cycles. It is observed that both porous and dense samples (S1 and S2) remain unchanged during the 10 cycles however, Ref sample crushed after the fifth cycle.

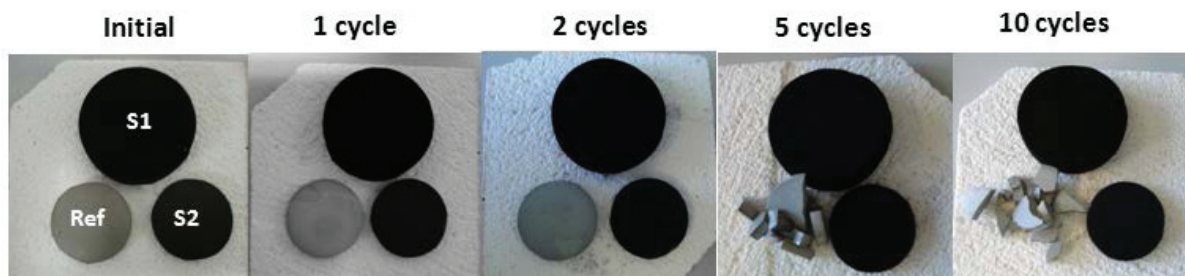
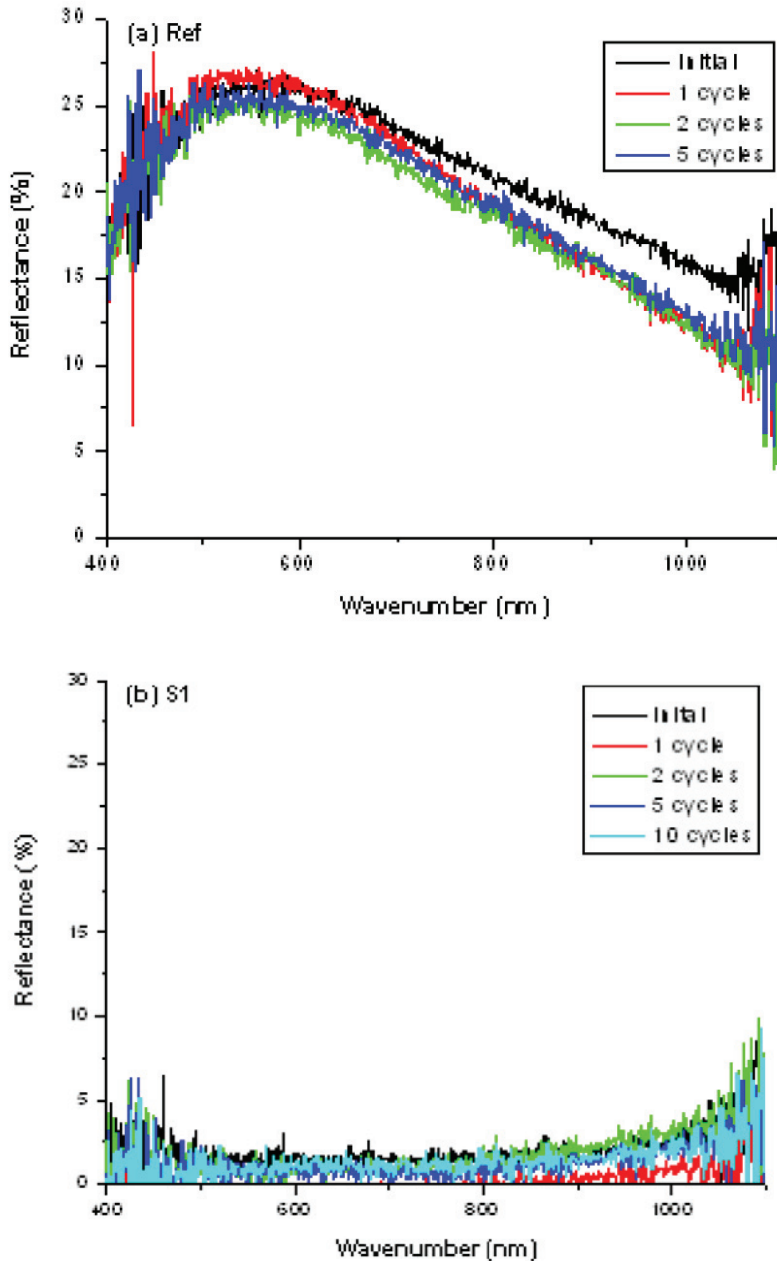


Fig. 6. Photographs of materials before and after high temperature tests (cycle of Table 7).

During these tests it was observed that after 10 cycles, weights and dimensions of both S1 and S2 samples did not change, however for the Ref sample and after the second cycle this material suffered an oxidation because its weight increased $\approx 3\%$ and it broke into several pieces.

On the other hand, and as can be seen in Figure 7 sample reflectance values practically do not vary after temperature test cycles. In the case of Ref and S2 samples the reflectance shows a small decrease, but for the S1 sample it presents the same reflectance values before and after 10 thermal cycles.



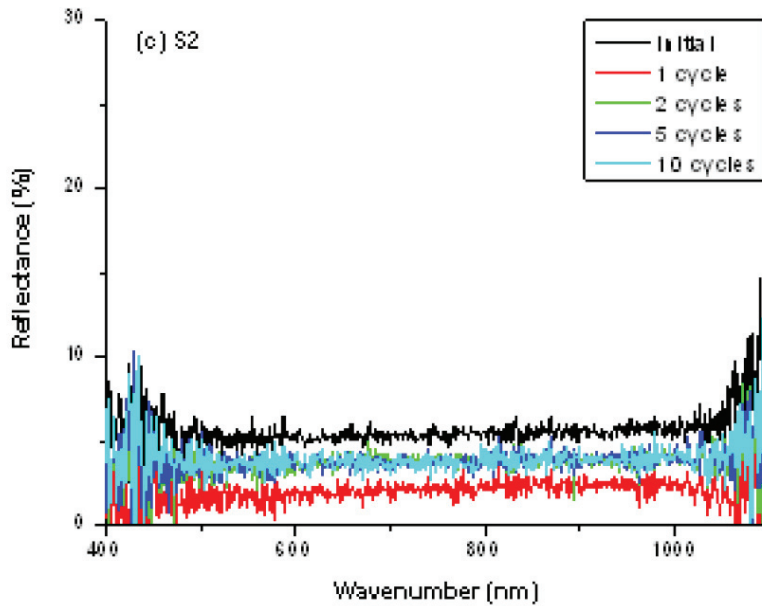


Fig. 7. Reflectance results (a) Ref (b) S1 (c) S2

4.2. Thermal shock

An example of the variation of temperature during the thermal shocks using the Fresnel lens is shown in Figure 8. As can be observed both the heating rate and the cooling rate vary depending on the temperature range selected. So, from ambient temperature to 800°C, heating rate of 50-70°C/s was registered. This is the maximum value of heating rate, because for higher temperature, from 800 to 1000°C the heating rate decreases at values of 3-5°C/s. Concerning the cooling, the highest rate occurred from high temperature to 600°C, with cooling rate of 9-11°C/s. The cooling rate decreases from 600-400°C to 2-4°C/s and for the range 400-200°C to 1°C/s. The cooling rate is much lower than the heating rate; when lens is covered, heat is lost only by the thermal diffusivity of the metallic sample at environment temperature. In order to increase the thermal shock, the cooling rate should be increased by external supply of cool air or wet cool air.

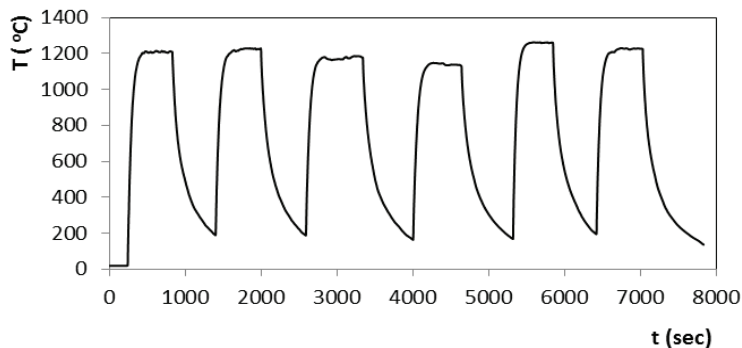


Fig. 8: Example of temperature (T) cycle with the Fresnel lens

Metallic samples were examined by optical and scanning electron microscopy at the end of the thermal shocks experiments. The iron aluminides with low Al content suffered severe oxidation with extensive oxide spalling, while

the iron aluminides with high Al content withstand oxidation. These results confirm previous papers showing a significant reduction in oxidation as the Al content increases from about 25% to about 40% [15].

The others tests (high humidity and temperature and salt mist) have not been performed yet in the project development.

5. Conclusions

This article presents a state-of-the-art of standard testing methods for solar components and of bibliography of tests used on central tower receiver.

A series of testing methods have been defined based on this first analysis and considering the equipment available within this project.

Some test results for materials that fulfill the requirements for high temperature solar receivers have been presented.

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