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Evaluation of thermal shock resistance of silicon oxycarbide materials for high-temperature receiver applications

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ABSTRACT

Materials used as solar receivers in concentrated solar power technology must withstand severe operational conditions caused by concentrated solar radiation. For this solar technology several ceramic material candidates (silicon carbide, porous and dense silicon oxycarbide) have been subjected to thermal shock resistance test by using Fresnel lens, the equipment available, that concentrates the solar radiation more than 2600 times. Fast heating (37 °C s^{-1}) and cooling rates (28 °C s^{-1}) from 100 to 1200 °C and dwelling time of 10 min are employed. The evolution of materials surface has been evaluated during test by spectroscopic methods and both confocal and electronic microscopies. It has been obtained the surface map of each analyzed sample in order to evaluate the effect of the concentrated solar radiation on the surface and the relationship with their durability. The absorptance values were also determined before and after the ageing test using normal direction between 400 and 1100 nm. Concentrated solar radiation facilitates the decomposition of tested materials producing the formation of gaseous species (mainly CO and CO_2) and a dense SiO_2 layer which is formed over the material surface. SiC and porous silicon oxycarbide materials fail the thermal shock resistance test. SiC experiences a catastrophic break and in the case of porous silicon oxycarbide, gases generated can evolve easily through the pores producing a severe degradation of the material surface. However, dense silicon oxycarbide resists 100 cycles at 1200 °C due to the formation of a protective SiO_2 layer over the material surface and its dense microstructure that slow down the diffusion of the gases preventing bulk material from being degraded. The surface studies confirm the formation of a crystalline SiO_2 phase all over the surface. Furthermore, the very similar coefficients of thermal expansion of silicon oxycarbide and silica ($\approx 0.4 \times 10^{-6}$ °C), protects material against catastrophic failure. Finally, the absorptance values remain fairly constant before and after thermal shock test (94.78 to 95.96–96.09%). The high resistance of dense silicon oxycarbide materials to thermal shock under concentrated solar radiation makes these materials suitable candidates of being used as high temperature solar receivers.

1. Introduction

Solar energy needs the development of durable novel materials which can be employed at very high temperature in concentrated solar power (CSP) systems for increasing efficiency in electrical power generation. The receiver is the key component of CSP plants as it is planned to absorb a maximum of concentrated sunlight and transfer it to the heat-transfer fluid with the highest efficiency (Rojas-Morín and Fernández-Reche, 2011; Boubault et al., 2012). It is exposed to highly concentrated solar fluxed and high temperatures. For example, every day the receiver is exposed to the thermal shock produced by the temperature variation between day and night and even by the sudden appearance of clouds during solar radiation exposure, all these are the principal ageing factors (Boubault et al., 2014). In these sense, receiver materials need to be carefully selected and designed taking into account the specific requirements of a given engineering environment such as working temperature, mechanical properties (resistance to fracture, elastic modulus, creep, fatigue), thermal properties (thermal-fatigue, thermal stress, thermal resistance, resistance against oxidation, thermal conductivity, coefficient of thermal expansion), optical properties (absorptance and reflectance) and external parameters (geometry, size, thickness, texture, channels) among others. Currently approaches are

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focused on metallic and ceramic materials (Kennedy, 2002) and there are several studies of the experimental, theoretical and simulated behavior of different materials under concentrated solar radiation (Fend et al., 2004; Rojas-Morín and Fernández-Reche, 2011; Boubault et al., 2012, 2014; Capeillêre et al., 2014; Morris et al., 2015; Charpentier and Caliot, 2017; Lalaua et al., 2017). Metallic materials (i.e. refractory alloy, INCONEL) are basically limited by their melting temperature and, on the other hand, ceramic materials (i.e. SiC) although increase their working temperature and life-time they are limited by their fragility. In these sense, SiC meets the specific requirements of solar receiver systems (Fend et al., 2004), however the thermomechanical behavior during service need to be carefully optimized by the design of material composition, geometry and texturation of the surface, because is the major responsible of system failure (Fend et al., 2004; Capeillêre et al., 2014). Furthermore, SiC-Si (silicon infiltrated SiC) materials are widely used for solar receiver applications due to their exceptional properties (good resistance to oxidation and corrosion, excellent thermal conductivity and high mechanical strength up to 1300 °C) (Agrafiotis et al., 2007; Rodríguez-Sánchez et al., 2016). Recently, ultrahigh temperature ceramics which include borides and carbides of early transition metals are also being studied for solar receivers applications (Sani et al., 2011, 2016a, 2016b).

In a previous paper (Sallaberry et al., 2015) based on a revision of standards and papers published, we studied the development of testing methods for solar receivers which guarantee their reliability and durability under demanding working conditions of high solar concentrating technology. This methodology was proposed after a careful revision of the most relevant and appropriate literature in order to select proper candidates as receivers that could resist high temperatures and high solar concentrated radiation. Generally, these studies employ very extreme conditions and, in this sense Rojas-Morín and Fernández-Reche (Rojas-Morín and Fernández-Reche, 2011) use a parabolic dish facility (DISTAL-1, Plataforma Solar de Almeria, Spain) to study the response of a central solar receiver material (INCONEL 625LCF®) under normal and critical operational conditions during different thermal cycles. Boubault et al. (2012, 2014) use a solar accelerated aging facility at PROMES laboratory (Odeillo, France) to test different materials (INCONEL 625 + Pyromark ® 2500) used in receivers of concentrated solar power plants. In this sense, several tests were adjusted in base to the requirements of selected materials that could be used as high temperature receivers (> 1000 °C) and the available facilities. In particular, the proposed-adapted studies were: high temperature (oven with air circulation at 1000 °C), thermal shock (Concentrated solar radiation by Fresnel lens; Temperature from 1000 to 1200 °C, at least 10 cycles of 10 min), salt mist (Temperature of 35 °C, humidity of 100% and sprayed solution of 5% of NaCl) and high humidity and temperature tests (Temperature ranged from −40 to 85 °C and humidity from ambient to 100%) (Sallaberry et al., 2015).

Silicon oxycarbide (SiOC) materials are very interesting and promising candidates due to their intrinsic properties as high temperature resistance, resistance against oxidation at harsh environments, high creep, moderate high mechanical properties and tuneable electrical and thermal properties among others (Pantano et al., 1999; Colombo et al., 2010; Wang et al., 2011; Ionescu et al., 2012; Mazo et al., 2013, 2017). Silicon oxycarbide materials are composed by a SiOC glassy matrix and a well-dispersed embedded graphite-like carbon phase (C_{free}) which confers to these materials their characteristic properties (Pantano et al., 1999). The oxidative degradation of silicon oxycarbide materials depends on several factors such as annealing temperature, thermal history (i.e. pyrolysis temperature) (Bois et al., 1995), composition (amount of carbon, both $C_{oxycarbide}$ and C_{free}) (Brewer et al., 1999), microstructure (Sorarù and Suttor, 1999), etc. but, of course, the main factor is the annealing temperature (Xu et al., 2011). Several authors (Bois et al., 1995; Chollon, 2000; Parmentier et al., 2001) established the temperature ranges and the chemical bonds involved during the oxidative degradation of silicon oxycarbide and related materials.

For the SiOC materials, in the 400–800 °C range, the main process is the degradation of C_{free} (Reaction (1)) and a weight loss is generally observed,

$$C_{free} + O_2 \rightarrow CO_2 + H_2O \qquad \text{(Reaction 1)}$$

At higher temperatures (800 °C), SiOC (i.e. Si-C) experiences a slight weight gain associated to the oxidative degradation of the matrix according with Reaction (2), where CO_x means CO and CO_2,

$$SiOC + O_2 \rightarrow SiO_2 + CO_x \qquad \text{(Reaction 2)}$$

Finally, at temperatures higher than 1200 °C SiC can be oxidized in relation to Reaction (3),

$$SiC + O_2 \rightarrow SiO_2 + CO_x \qquad \text{(Reaction 3)}$$

Previous results of high temperature cycling test (100 cycles at 1000 °C) indicated the suitability of silicon oxycarbide materials as high temperature receivers (Zaversky et al., 2014). Here we present the results of these potential candidates exposed to thermal shock tests. Thus, the experimental conditions such as temperature, heating, cooling rates and number of cycles were established taken into account our facilities and previous works. In this way, 1200 °C is the highest temperature reached by the size of our Fresnel lens in our solar radiation conditions which can be maintained during all the exposure time; the heating and cooling rates are the highest available in our installation in order to subject samples to a thermal shock; and the number of cycles were estimated based on previous studies (Zaversky et al., 2014; Sallaberry et al., 2015).

In this sense the aim of this work is to study and compare the suitability of SiC and two types of SiOC materials one porous and the other one well-densified to be employed as high temperature receivers. These materials have been studied under an accelerated ageing test consist on a thermal shock under concentrated solar radiation at a maximum temperature of 1200 °C. The surface evolution of these materials has been studied through spectroscopic methods (infrared, Raman and UV–Vis spectroscopies), confocal microscopy, roughness and scanning electron microscopy.

2. Experimental procedure

Three samples were investigated: two dense materials, SiC and $SiOC_d$, and a porous one, $SiOC_p$. Dense SiC was obtained by sintering fine SiC powders (generously supplied by Advanced Thermal Devices, S.L., Madrid, Spain; surface area = 33 $m^2 g^{-1}$; mean particle size d_{50} = 0.86 μm and composition: SiC = 97.85%; SiO_2 = 1.60%; C = 0.30%). The sintering process was carried out without addition of any additive by using a Spark Plasma Sintering equipment (SPS, SPS-510CE, SPS Syntex Inc., Kanagawa, Japan) working at 1800 °C, 50 MPa and holding time of 5 min. The dense $SiOC_d$ material was obtained from fine SiOC powders (surface area = 5.83 $m^2 g^{-1}$, mean particle size D_{50} = 3.42 μm and composition $SiO_{1.69}C_{0.43}$) that were also sintered employing the SPS equipment working at 1500 °C, 40 MPa and 5 min of holding time. A detailed description of the experimental procedure can be found elsewhere (Oteo et al., 2011; Mazo et al., 2012). Finally, the porous $SiOC_p$ material was obtained in one-only step process from the pyrolysis at 1100 °C of a hybrid preceramic material prepared from Tetraethylortosilicate (TEOS)/Polydimethylsiloxane (PDMS) (Mazo et al., 2015). The chemical composition of the $SiOC_p$ sample was $SiO_{1.67}C_{0.50}$. For SiC and $SiOC_d$ materials all samples were cylindrical of approximately 20 mm × 3 mm, however for the $SiOC_p$ samples they were cylinders of 30 mm × 7 mm. The initial porosities of SiC, $SiOC_d$ and $SiOC_p$ materials were 19%, 2% and 81%, respectively and the thermal conductivities were 6.82, 1.43 and ≈0.1 $W m^{-1} K^{-1}$, respectively.

Thermal shock tests were carried out under concentrated solar radiation by means of a Fresnel lens mounted on a solar tracker located at