



Nanocomposites derived from silica and carbon for low temperature photothermal conversion



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ABSTRACT

Carbon-oxide nanocomposite films have been fabricated using a sol-gel synthesis of hybrid precursors i.e. tetraethyl orthosilicate (TEOS) + methyl- β -cyclodextrin (M β CD) or colloidal zirconia + M β CD, followed by carbonization in an inert atmosphere. Reflectance measurements were performed on these samples to determine solar absorptance (α) and thermal emittance (ε). The ratio α/ε was used to quantify the selectivity of the samples, the bigger this value the better the selectivity. The selectivity of the films was optimized according to various parameters such as the amount of M β CD in the precursor, the thickness of the film or the surface roughness. The best value of α/ε was obtained for a TEOS + M β CD film coated on a copper substrate.

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1. Introduction

The basic principle of photothermal conversion is to transfer the collected incident solar energy to a heat-transfer fluid through an absorber. However, part of the incident radiation is lost by reflection at the surface of the absorber, which constitutes the optical losses. In an effort to minimize the optical losses, spectrally selective absorber surfaces are widely used for the photothermal conversion of solar energy. These surfaces are characterized by high solar absorptance (low reflectance) in the visible and near-infrared regions (0.3–2.5 μm), and a low emittance (high reflectance) in the long wavelength (infrared) region (2.5–20 μm). No intrinsic material is capable of this selectivity. It can however be achieved by the use of a combination of various compounds which tailor the optical properties of the surface [1–4]. A solution implemented to achieve this goal is to make thin solar absorbing coatings on a reflecting metallic substrate. Many examples [5] deal with this topic, leading to an important set of solution. Sputtering and electrolysis of environmentally hazardous elements such as Cr or Ni were used for the fabrication of these selective films. Many studies [6–12] have been carried out to try to obtain selective coatings by green chemistry processes (sol-gel) using eco-friendly and low cost compounds (silica, carbon); such work has not lead to any industrial applications.

In this present work, selective surfaces were obtained by a process inspired by the work of Mastai [11]. It is based on coating a metallic substrate (Cu, Al) with a film containing “nanocarbon” grains embedded in an oxide matrix. Our purpose was to modify the nature of the oxide matrix to improve the selectivity of the coatings. For that, silica matrix was partially or completely substituted by zirconia.

This film is obtained by the carbonization of a hybrid precursor oxide + methyl- β -cyclodextrin (M β CD) obtained using a sol-gel process. In order to optimize the selectivity of the coating, various parameters such as the carbohydrate concentration, the thickness of the deposited layer and the nature of the oxides used (i.e. silica, zirconia or a mixture silica + zirconia) were studied. The evolution of the optical properties of the coatings on the efficiency of the solar collector is also discussed.

2. Experimental and measurements

2.1. Substrate preparation

Smooth aluminum or copper plates of 25 \times 50 mm² were used as substrates. These substrates were cleaned by electrolysis treatment at 5 V for 1 min in a commercial solution of UniClean® 248 concentrated at 10 wt.%. A platinum electrode was used as the cathode. In a second step, the substrate is dipped in a sulfuric acid bath (10 wt.%) for 2 min, to remove the oxide layer. Lastly, the substrate is rinsed thoroughly in distilled water and then dried by blowing with nitrogen gas.

2.2. Nanocoating preparation

Different hybrid precursors (oxide + M β CD) based on silica, zirconia or a mixture of silica + zirconia, were obtained by a sol-gel process. Briefly, a stock solution was first prepared using 8.815 g tetraethyl orthosilicate (TEOS), 5 g EtOH and 6.285 g H₂O pH 2 (HCl). This solution was then mechanically shaken for 1 h. Simultaneously, M β CD was dissolved in 25 g EtOH, and to which 3.63 g of the solution stock was added. The resulting solution (S1) was stirred continuously at room temperature for 2 h. The metallic substrate

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was dip-coated onto the solution S1 to obtain the M β CD + TEOS precursor samples.

Acetate stabilized colloidal zirconia was purchased from Nyacol® (particle size 10 nm). The addition of these colloidal particles to the solution S1 was done to obtain the slurry S2. Acid acetic (pH = 3) was added to the slurry S2 to correct its viscosity. The slurry S2 was sonicated to prevent the agglomeration of the particles. The metallic substrate was dip-coated onto the slurry S2 to obtain the M β CD + colloidal ZrO₂ and M β CD + a mixture of TEOS and colloidal ZrO₂ samples.

The dip-coating was performed initially at rates of 1, 2, and 6 mm/s. An optimum speed of 2 ± 0.3 mm/s was used for the rest of the experiment. The coating was performed in a glove box at 50% humidity. The samples were dried under vacuum for 12 h and were placed in a furnace to carbonize the M β CD. The carbonization process was carried out under flowing argon at temperatures ranging from 400 to 540 °C for 1 h. The furnace was raised to this temperature at $1 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$, and then cooled to 30 °C at $20 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$.

In the work presented below, the samples referred as C/SiO₂, C/ZrO₂, and C/SiO₂ + ZrO₂ were respectively prepared from M β CD + TEOS precursor, M β CD + colloidal ZrO₂ and M β CD + a mixture of TEOS and colloidal zirconia and carbonized at 500 °C under argon atmosphere.

2.3. Characterization methods

The hemispherical reflectance *R* of the samples was measured in the wavelength range of 0.28–16 μm . A Shimadzu UV-3600 UV-VIS-NIR spectrophotometer equipped with an integrating sphere of 50 mm diameter, circular beam entrance and sample ports of 20 mm diameter, was used in the wavelength range of 0.28–2.5 μm . The infrared wavelength range of 2.5–16 μm was covered with a PerkinElmer Spectrum 100 spectrophotometer with an integrating sphere of 80 mm diameter and a circular beam entrance and sample port of 20 mm diameter.

$$\alpha = \frac{\int_{0.28}^{2.5} I_{\text{sol}}(\lambda) \times (1-R(\lambda)) d\lambda}{\int_{0.28}^{2.5} I_{\text{sol}}(\lambda) d\lambda} \quad (1)$$

$$\varepsilon = \frac{\int_{2.5}^{16} I_{\text{p}}(\lambda) \times (1-R(\lambda)) d\lambda}{\int_{2.5}^{16} I_{\text{p}}(\lambda) d\lambda} \quad (2)$$

The measurements were combined to create one spectrum and the absorptance α and emittance ε were calculated using formulas Eqs. (1) and (2), where *R* is the total reflectance, *I*_{sol} the solar spectrum (AM1.5) and *I*_p is the Planck black body power system. The comparison of the selectivity of the surface is based on the α/ε ratio (α : the solar absorptance, ε : the thermal emittance), the higher the value of α/ε the better the selectivity. Typical spectra of C/SiO₂ or C/ZrO₂ films deposited on aluminum substrate are presented in Fig. 1. The comparison of these measurements with those obtained on a metallic substrate of aluminum highlights the selectivity achieved by the deposition of a carbon + silica or carbon + zirconia layer. Characteristic chemical bonds of silica were identified from the reflectance spectrum (see Fig. 1).

Environmental scanning electron microscopy (ESEM) analyses were obtained with a FEI Quanta FEG 200 ESEM coupled with an energy dispersive X-ray microanalyzer (EDX) Bruker XFlash 4010 SDD. ESEM observations and EDX analyses were performed at an operating voltage of 15 or 30 kV under a H₂O gaseous partial pressure of 10 Pa.

Thicknesses were evaluated with X-film® software provided by Bruker Company. It enables to measure the thickness of a film from EDX analyses. For three primary energies *E*₀ (5, 10 and 15 kV), the *k*-ratio of the elements were measured with the same current beam and acquisition time. The values of *k*-ratios and the composition of the

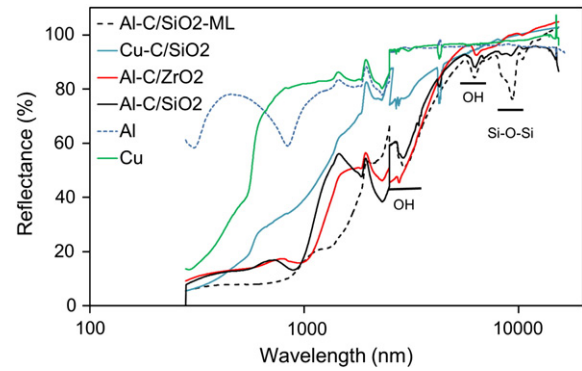


Fig. 1. Hemispherical reflectance spectrum of C/SiO₂, C/ZrO₂ nanocomposite deposited on aluminum substrates and C/SiO₂ deposited on copper substrate compared to the values obtained for aluminum and copper. The chemical bonds of silica are identified in the absorber composite layer Al-C/SiO₂. ML refers to the deposition of multilayers onto the sample.

analyzed layer were used as input for the X-film® software to calculate the thickness (Table 1).

The conversion of the M β CD + TEOS or M β CD + colloidal ZrO₂ precursors was carried out in a tubular furnace and in the thermogravimetric analyzer (TGA) apparatus. A flow of argon over 30 L/h was used in the tubular furnace to evacuate the gaseous species produced during the thermal treatment. TGA analysis was performed using a Setaram SETSYS analyzer. Approximately, 50 mg of M β CD + TEOS precursor was placed in an open 120 μL alumina pan and heated at a heating rate of $10 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ to 850 °C under argon flowing gas ($20 \text{ mL}\cdot\text{min}^{-1}$).

3. Results and discussion

Fig. 2 shows a typical TGA curve of TEOS + M β CD precursor. The weight loss observed on this curve can clearly be attributed to the decomposition of the M β CD precursor into carbon composite particles. This decomposition is achieved at a temperature of about 500 °C. Preliminary measurements of the absorptance indicate that this property increases with the temperature of carbonization of the precursor. However, for temperatures around 500 °C, the aluminum substrate becomes soft (*T*_{melting} = 660 °C). In order to optimize the ideal temperature of the heat treatment, absorptance measurements have been

Table 1
Characteristics of the samples investigated in this work.

Samples	Number of layers	M β CD (wt.%)	Thickness (nm)	α (%)	ε (%)	α/ε
Al				26.2	4.1	6.4
Cu				36.2	3.1	11.6
Al-C/SiO ₂	1	14	320	59.2	2.7	21.9
		24.6	550	73.9	4.5	16.4
		32.9	770	77.2	7.4	10.4
		39.5	900	82.3	12.6	6.5
Al-C/SiO ₂	2	44.9		82.0	22.6	3.6
		14	680	80.5	5.6	14.3
		14	1020	85.5	6.0	14.2
		14	1360	86.8	9.4	9.2
		14	1680	89.7	18.0	4.9
Cu-C/SiO ₂	1	14	400	67.4	2.1	32
		14	700	81.2	3.5	23.2
		14	1020	88.8	12.9	6.8
		14	1360	89.6	22.4	4
Al-C/SiO ₂ + ZrO ₂	1	39.1		82.0	10.3	7.9
		38.7		82.3	8.7	9.4
		38.4		82.1	8.2	10
		38		81.9	7.6	10.7
Al-C/ZrO ₂	1	7.4		80.3	6	13.3
		12.5		79.7	3.9	20.4
		17.6		78.1	7.4	10.5
		23.1		81.6	16.8	4.8

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