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ScienceDirect

Ceramics International 41 (2015) 2735–2743

CERAMICS INTERNATIONAL

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Tailoring of the electrical properties of carbon black–silica coatings for de-icing applications

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Received 17 September 2014; received in revised form 14 October 2014; accepted 14 October 2014
Available online 22 October 2014

Abstract

Electrical heating properties of carbon black–silica composite coatings have been studied for de-icing devices. Coatings are consolidated by spray deposition onto tile substrates and thermal treated in the temperature range of 300–500 °C in air atmosphere. The electrical properties of the coatings depend on the thermal treatment as for higher sintering temperatures the coatings are more resistive, and consequently the heating produced by the Joule effect is more significant. Therefore, it is possible to tailor the heating properties of the coatings by means the thermal treatment, achieving thin coatings with low electrical resistance ($\sim 5\times10^{-4}\ \Omega$ m), or coatings with higher resistivity ($\sim 5\times10^{-3}\ \Omega$ m) which produce thermal heating >70 °C with heating rate >0.13 °C s^{-1}. This coating heating is determined using DC intensity–voltage curves and thermal infrared camera. The relationship between electrical power and temperature increasing showed a nearly linear dependence. The experimental results for these coatings allow their use in a wide range of functional applications, such as de-icing devices for aeronautics.
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Keywords: C. Thermal properties; Conductive thin films; de-icing applications; Multifunctional

1. Introduction

Electrically conductive coatings results of great interest in different fields of new energy and transport technologies, such as transparent electrodes (ITO, ATO) for photovoltaic and electroluminescent applications, or as electrodes in batteries or electroanalysis [1–3]. The addition of conductive filler into a polymer coating is widely used for conductive paints in the automotive industry, as anti-static protections, electromagnetic and radio frequency interference (EMI/RFI) shielding [4,5]. However, few works are focused in coatings with heating applications, such as aircraft paints with de-icing properties in wings [6] or for transmission lines in the telecommunications field [7]. Carbon nanotubes or carbon nanofibers are widely proposed as conductive fillers in these electrical composites, but both the dispersion problems and the material cost limit their use [8,9]. On the other hand, Carbon black (CB) is a preferred conductive filler because is easily available, low cost, and possesses high conductivity values ($\rho\sim10^{-5}\ \Omega$ m) [10]. CB is produced by incomplete combustion of gaseous and liquid hydrocarbons, and it is characterized by a wide particle size distribution, from 10 nm to 100 μm [11,12]. The addition of CB to polymer or epoxy based composites, applied in thick coatings, allows obtaining different ranges of resistivity, 10^{-1}–$10^{-3}\ \Omega$ m [13,14]. Recently submicronic coatings by the sol–gel process allows to consolidate CB in a silica matrix having the lowest resistivity values ($\rho\sim10^{-5}\ \Omega$ m) reported in the literature for CB coatings [15]. Silica provides an appropriate matrix to carry out the preparation of homogenous hybrid coatings due to its chemical stability and its low temperature ceramic processing [16–18]. Thus hybrid CB-silica coatings are potential candidates for integrated electrical heating applications based on their submicronic configuration. These thin coatings could provide advantages as de-icing heater over conventional heaters that use metal foil, due to

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http://dx.doi.org/10.1016/j.ceramint.2014.10.088
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its low thickness which does not increase the device weight. Moreover, the silica matrix prevents corrosion wear and passives the conductive filler [6]. These composites withstand high and low temperature requirements of some applications as in the aerospace industry. In addition, the low coating thickness allows to obtain transverse electrical conduction through the composite despite a surface voltage is applied [19].

The aim of this work is to study the electrical response and the electrical heating of the CB-silica composite coatings obtained by the sol–gel process. Electrical heating properties based on Joule effect are correlated with the coatings nanostructure in which resistive interfaces play a major role. The resistivity of the coatings depends on the sintering process and thus the electrical heating response may be tailored based on coating properties. Finally, the efficiency of the conductive coatings is proved by means of the design of a de-icing prototype with heating properties by applying an adequate voltage.

2. Experimental procedure

2.1. Materials

Carbon black (CB) PUREX® HS 55 (powder, Degussa) (density 0.335 g/cm^3, agglomerate size of 1.4–1.7 μm and a high specific surface area of 40 m^2/g) is used as starting material. The reagents used to prepare the sol was tetraethyl orthosilicate 99% (TEOS, Sigma-Aldrich) as silica precursor, ethanol absolute 99.9% (MERCK) as solvent, deionized water to produce the hydrolysis and hydrochloric acid 37% (MERCK) as catalysts. The surfactant used to disperse CB nanoparticles in water (20 wt% solid content) was Triton X-100 (T) (Sigma-Aldrich) which is a non-ionic surfactant with a hydrophilic polyethylene oxide group, $-(OCH_2CH_2)_nOH$, a phenyl group and a hydrocarbon hydrophobic chain (C_8H_{17}). Triton X-100 has an "average of $n=9.5$" ethylene oxide units per molecule [12,20]. The proportion of Triton X-100 used to disperse the CB nanoparticles was 2.75 wt%. since this is the amount that provides the highest viscosity value, and therefore, the larger dispersion of nanoparticles, as explained in previous work [15]. The dispersion was carried out in a high shear disperser (SL-1 ADF Disperser Lleal) that works at 50 Hz, 220 V and 5.5 A. The solution was dispersed at 2500 rpm during 10 min.

2.2. Sample preparation

The sol was prepared with the following reagents proportion: 1TEOS:16EtOH:4H_2O:0.01HCl and 50 wt% of CB respect to the theoretical formation of SiO_2 from the complete hydrolysis of the precursor, TEOS. This is the adequate percentage of CB to achieve the percolation limit, as demonstrated in previous work [15]. The sol was prepared as in previous works, and it was deposited onto the tile ceramic substrates provided by Roca Sanitario S.A., which composition is well known [21], by air-spray deposition method using a spray gun with a pressure of 2.5 bars in air. The coatings were dried to gel consolidate and then thermally treated in air at 300 °C during 5 h, 400 °C 1 h and 500 °C 1 h. The heating and cooling rate were 1 °C/min and 3 °C/min, respectively.

2.3. Characterization

Microstructural characterization was studied by means of Field Emission Scanning Electron Microscopy (FESEM) by using a Hitachi S-4700 microscope and the Conductive Atomic Force Microscopy (C-AFM) by using an NT-MDT Solver SPM instrument. C-AFM provides both topography and surface conductivity mapping of the sample with a nitrogen-doped diamond tip for conductivity for DC contact mode (in the spreading resistance mode) and Au reflective side and resonant frequency 190–255 kHz. FESEM micrographs were studied by means of the Leica Q-Win software in order to determine the agglomerate size distribution. Electrical characterization was followed using an electrode configuration located at the surface of the coating as seen in the scheme presented in Fig. 1a. Fig. 1b shows a photograph of the coating composed by 50 wt% CB/SiO_2 with 2.75 wt% of Triton X-100, with the copper electrodes on the surface, and of a one euro coin that shows the size of the sample.

The electrical measurements of submicronic coatings were carried out with a Unilab DC HT power supply, AC 6 · 3 V

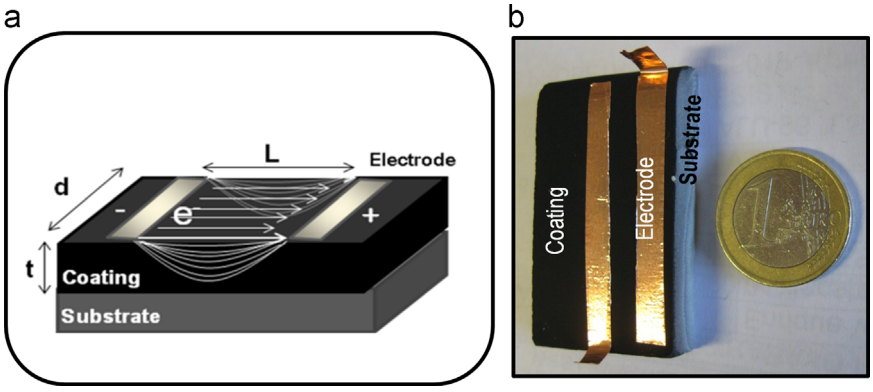

Fig. 1. a) Scheme of hybrid CB-silica coatings deposited onto tile ceramic substrates, with two-probe method and surface electrode configuration ($L=5$ mm, $d=25$ mm and $t\sim500$ nm [15]) and b) Photograph of a hybrid 50 wt% CB-silica coating onto ceramic tile having copper surface electrodes and of one euro coin that shows the size of the sample.

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