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A dedicated electric oven for characterization of thermoresistive polymer nanocomposites

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Abstract

The construction, characterization and control of an electric oven dedicated to the study of thermoresistive polymer nanocomposites is presented. The oven is designed with a heating plate capable of reaching 300 °C with a resolution of 0.3 °C and an area of uniform temperature of 3.8 cm × 2.5 cm. The temperature is regulated by means of a discrete proportional–integral–derivative controller. A heat transfer model comprising three coupled non-linear differential equations is proposed to predict the thermal profiles of the oven during heating and cooling, which are experimentally verified. The oven is used for thermoresistive characterization of polymer nanocomposites manufactured from a polysulfone polymer and multiwall carbon nanotubes.

Keywords: Electric oven; Discrete PID controller; Polymer nanocomposite; Thermoresistive

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1. Introduction

Laboratory and commercial ovens are used in a broad variety of applications, from cooking to industrial processing (Mullinger & Jenkings, 2008). By considering their energy source they can be broadly classified into two groups, fuel-based and electric ovens (Mullinger & Jenkings, 2008; Trinks, Mawhinney, Shannon, Reed, & Garvey, 2004). Electric resistance heating has various advantages over systems based on fuel combustion, such as increased control accuracy and heating speed. Thus, electrical heating constitutes a suitable choice for developing laboratory instruments, especially those demanding small heating volumes and precise temperature control (Corona, Maldonado, & Oliva, 2007; Devaraju, Suresha, Ramani Radhakrishnam, 2011; Gam, 1996; Merlone, Iacomini, Tiziani, & Marcarino, 2007). As a general rule, laboratory ovens must operate at established temperatures and avoid temperatures that may damage the samples or their components. Uniform temperature at prescribed zones is also a frequently desired feature, which may be difficult to achieve. All these requirements demand a rational mechanical design, thermodynamic (mathematical) simulations, and the implementation of a suitable control system (Mullinger & Jenkings, 2008). At the industrial level, the majority of the feedback controllers comprise a form of proportional–integral–derivative (PID) loop, mainly because of its simplicity and the vast literature available on PIDs (Johnson & Moradi, 2005; Ogata, 2010). Analog PID controllers work through pneumatic, electronic, electrical or combinations thereof, although with the use of microprocessors, digital versions have been created for these controllers (Ogata, 2010). The major advantages of digital systems are the flexibility and decision making features. The availability of inexpensive digital computers and the benefit of working with digital signals have promoted the current trend towards digital

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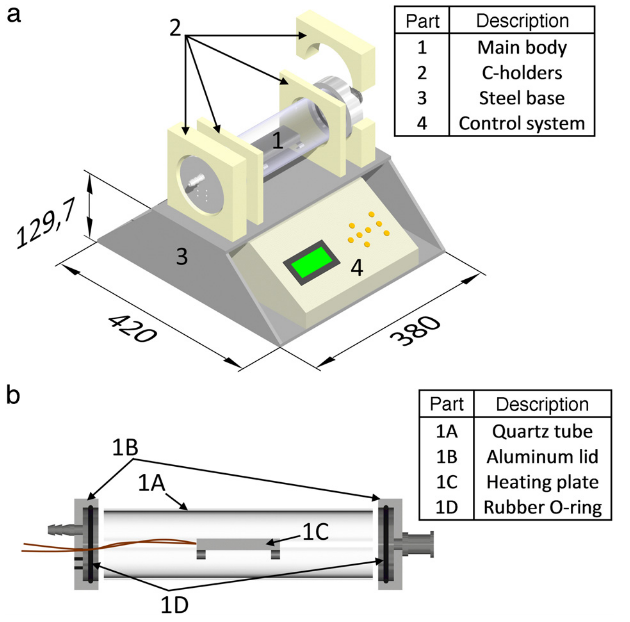
control systems (Ogata, 1995). During the design processes, thermodynamic modeling may assist in the design of the oven (Abraham & Sparrow, 2004; Mistry, Ganapathi, Dey, Bishnoi, & Castillo, 2006). Modeling may assist in the selection of appropriate dimensions and design parameters, saving important amount of resources which would be otherwise wasted in expensive trial and error. Besides simpler analytical efforts (Abraham & Sparrow, 2004), numerical models such as those based on the finite element method have allowed the study of rather complex systems (Depree et al., 2010; Mistry et al., 2006; Najib, Abdullah, Khor, & Saad, 2015; Ploteau, Nicolas, & Glouannec, 2012). Nevertheless, a numerical approach is often very particular for the oven in question, and thus difficult to utilize as a general design guide. Another way to approach the simulation of ovens is the use of the lumped parameter method, which consists in a simplification of the system of equations as a network of discrete elements with reduced number of nodes (Ramallo-González, Eames, & Coley, 2013; Ramirez-Laboreo, Sagues, & Llorente, 2016; Underwood, 2014); although this method reduces the computational time, a set of appropriated parameters should be known to calibrate the model.

Given this background, this work aims to develop and characterize an electric laboratory oven to achieve uniform temperatures for studying small (1 cm × 1 cm) polymer nanocomposite samples heated below 300 °C, by controlling the heating temperature with a minimum precision of 0.3 °C by means of a PID control. Thermodynamic balance renders a set of differential equations that are used to predict the temperature evolution in the major components of the oven. As an application example, the oven is used to provide uniform, precise and controlled heating to small carbon nanotube/polymer composites, for their use as thermoresistive sensors. The design approach used here, the governing differential equations proposed and the characterization methods employed may assist other researchers and engineers in the design of dedicated, low temperature, high precision controlled ovens.

2. Oven design

2.1. Mechanical design

The main body of the oven consists of a 300 mm long quartz tube with 80 mm outer diameter and 2.5 mm wall thickness. The quartz tube has two solid aluminum lids made of 102 mm diameter circular plates. The oven is supported by a trapezoid-shaped steel base, which encloses the control system. A couple of internal supports for the body of the oven and C-shaped holders for fixing the tube and lids were manufactured from Nylamid plates, see Figure 1a. C-holders at the end of the body of the oven hinge into two sections to allow the removal of the lids. Ceramic fiber-cloth was used to thermally isolate the supports from the oven body. Sealing of the oven is achieved by Viton® O-rings hermetically adjusted between the quartz tube and aluminum lids. The aluminum lids include feedthroughs for electrical wires, which connect the heating element contained inside the quartz tube to the power supply, as well as for thermocouples and wires. K-type thermocouples were used to measure the temperature inside the oven, with the reference one located typically over the heating plate or directly over the sample (depending on the experiment), see Figure 1b. The aluminum lids also have connectors for potential gas supply, although this feature was not used herein. The heating element was fabricated from a 12.7 mm thick rectangular aluminum plate with in-plane dimensions of 95 mm by 38 mm, containing an electric cartridge heater (electrical resistance) supplied with 120 V AC at a nominal frequency of 60 Hz. The heating plate also contains a small hole which receives a thermocouple for temperature monitoring. The oven lids can be removed to introduce the samples, placing thermocouples or simply to access the heating plate. To prevent samples from sticking to the heating plate, a Teflon-coated fiber glass fabric cover (∼200 μm) was used.



Part	Description
1	Main body
2	C-holders
3	Steel base
4	Control system

Part	Description
1A	Quartz tube
1B	Aluminum lid
1C	Heating plate
1D	Rubber O-ring

Fig. 1. Components of the electric oven: (a) major components, (b) detail of the sub-components of the main body. Dimensions in mm.

2.2. Electric and control system

The oven was designed to operate with a power supply of 120 V (AC) at 60 Hz. Electrical current/voltage measurements showed that the mean power demanded by the electrical resistance (cartridge heater) was 60 W. The control system uses a microcontroller implemented on a specially designed electronic board, similar to the control system used in other devices reported in the literature (Affanni, 2013; Ahlers & Ammermuller, 2013; Devaraju et al., 2011; Oliveira, Freire, Deep, & Barros, 1998). A schematic of the electronic components and the interconnections of the control system are shown in Figure 2. The microcontroller chosen for the control system is the PIC18F4550 (Microchip Technology, Chandler, USA), mainly because it includes a serial communication module which assists in the interconnection with other integrated circuits via an I^2C protocol (Predko, 2008). Other relevant components used are an analog-to-digital converter (ADC) MAX6675 for K-type thermocouples (Maxim Integrated, San Jose, USA), a