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The properties of nanodispersed carbon black particles after thermal treatment

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Abstract

The effect of thermal treatment at the temperature ranging from 800 to 2800°C on electrical resistance, specific adsorption surface and Raman spectrum of nanodispersed carbon black samples was investigated. It was determined that during the temperature increase, at first, the sample electrical resistance decreases (till the temperature of 1100 - 1600°C), and then rises. Specific adsorption surface decreases to geometrical one with the increase of the treatment temperature. New band appears within the range of ~ 2570 cm^{-1} in Raman spectra after the thermal treatment at the temperature of 2000°C and higher.

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Keywords: nanodispersed carbon black; thermal treatment; electrical resistance; specific adsorption surface; Raman spectroscopy

1. Introduction

Among the existing diversity of various nano-size carbon materials, nanodispersed carbon black (NDCB) has the widest range of applications. Its conductive brands used in industry for antistatic polymeric composites production (tires, cable jackets, hoses for pumping explosive and flammable fluid, conveyor belts, etc.) are of particular interest [1-3], those brands being used for chemical power source electrode paste production as well [4-6]. Nowadays the main synthesis process of conductive NDCB is liquid hydrocarbon high temperature oxidative pyrolysis (furnace method) [7]. This method, however, allows to obtain the end-product with properties conforming not to all consumer requirements. Moreover, the choice of industrial synthesis conditions is mostly of semi-empirical nature,

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and this is primarily connected to the complexity of the processes involved in oxidative pyrolysis as well as limited range of means promoting NDCB electrical conductivity increase.

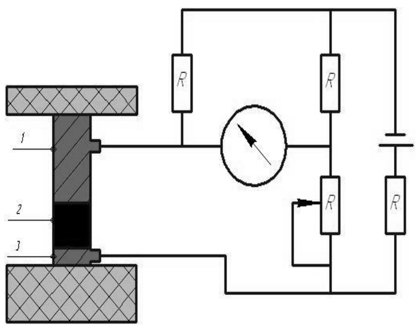
Various kinds of modifying treatment (thermal, thermal oxidative, and thermal-gas-chemical) are mostly used for detailed research and modeling of nanodispersed carbon black properties in simplified controlled conditions. The findings of investigation on effect of steam thermal oxidative treatment on NDCB particles structure and properties are reported in [8]. Also thermal oxidative treatment of non-conductive NDCB samples results in electrical resistance decrease till the values typical of highly conductive brands of nanodispersed carbon black [8]. The same paper specified that the nature of NDCB particles structural parameters changes after thermal oxidative treatment is similar to that of structural changes observed in the particles after high temperature treatment under vacuum induction heating [9]. Herein the results of investigating the thermal modifying treatment effect on NDCB properties, namely on conductive properties, are presented.

2. Experimental

Thermal treatment was performed in Tamman laboratory furnace of electrical resistance at the temperature range of 800 - 2800°C. Thermal treatment subject was heated in a closed graphite crucible till the specified temperature and was held for 20 minutes at the given temperature value. The heating and cooling of the crucible was conducted in N_2 medium.

The study subjects were NDCB samples with different specific surface by nitrogen adsorption obtained in experimental industrial equipment for NDCB synthesis: P 514 (S_{sp} = 50 m^2/g; GOST 7885-86), P 234 (S_{sp} = 103 m^2/g; GOST 7885-86), P 267-E (S_{sp} = 246 m^2/g; TOR 38 11574-86), PO 1 (S_{sp} = 545 m^2/g) and PO 2 (S_{sp} = 755 m^2/g). PO 1 and PO 2 thermal treatment subjects are experimental samples of thermal oxidative pyrolysis.

Conductive properties of heat-treated samples were measured with the equipment consisting of a dielectric cylinder with inside diameter of 17 mm and height of 95 mm, two steel cylindrical electrodes, and a universal measuring bridge. Schematic electrical diagram of the equipment is shown in Fig. 1a. Powder column with weight of 1 g was compressed till the minimum height of 7 mm, and staged measuring of electrical resistance (R) was performed per each millimeter in the column height range from 10 to 7 mm (Fig. 1b). At minimal height (7 mm) the powder column was put into the standard bridge four-terminal circuit branch [10]. Measurement uncertainty was no more than ± 6 %. Battery voltage in the bridge circuit equaled to 1 V.



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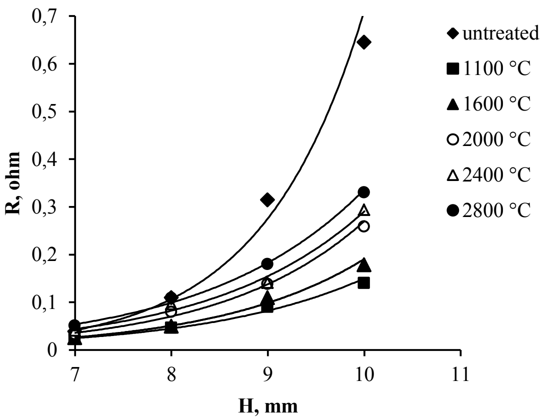

b

Fig. 1. (a) Schematic electrical diagram of the equipment for measuring bulk electrical resistance: 1, 3 are steel electrodes; 2 is NDCB sample. (b) The dependence of electrical resistance of the untreated and heat-treated NDCB samples on powder column height.