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Structure and supercapacitive performance of hierarchical porous carbon obtained by catalyzing microporous carbide-derived carbon

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ABSTRACT

Microporous carbide-derived carbon (CDC) with both high SSA and extremely narrow pore size distribution is synthesized by chlorination of niobium carbide powder. The produced microporous CDC is catalyzed at 1000 °C by using nickel nitrate as the catalyst. This treatment leads to the formation of a small amount of mesopores and slight decrease in the SSA. Electrochemical investigations show that the specific capacitance of the CDC catalyzed by nickel nitrate is almost as high as that of the pristine CDC in 6M KOH electrolyte. Furthermore, its cyclic voltammogram curves can keep a rectangular-like shape even at a scan rate of 500 mV s⁻¹, a significant improvement compared with that of the pristine CDC, indicating that the catalyzed CDC as an electrode material for supercapacitor exhibits superior specific capacitance and rate performance. Therefore, catalyzing CDC may be regarded as a facile and effective strategy to tune the CDC pore structure to match the applications of supercapacitor or some others.

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1. Introduction

Carbide-derived carbon (CDC) is a type of carbon produced by selective removal the metal or metalloid atoms from carbides [1]. It shows several outstanding advantages over traditional carbon materials, such as high specific surface area (SSA) and tunable pore size with a narrow distribution [2]. Because of the unique micro-structure, the CDC exhibits potential application in the fields of electric double-layer capacitors (EDLCs) [3,4], hydrogen and methane storage [5], catalyst supports [6] and so on.

It has been recognized that the CDC structure can be influenced by the catalyst incorporated during the CDC formation process. Kormann et al. [7] found that the introduction of transition metal catalysts during chlorination led to the increase of the degree of graphitization. Some other carbon structures (e.g. carbon onions [8], barrel-like carbon [9]) can also be formed due to the effect of catalyst. Besides, Käärik et al. [10] and Xu et al. [11] reported that, during the chlorination, the presence of catalyst caused the formation of a large amount of mesopores and consequently a great decrease in SSA. However, up to now, few work concerns the effect of catalyst on the produced CDC.

In this work, we investigated the structural evolution of a microporous CDC due to the introduction of nickel nitrate as

catalyst and the effect of incorporated catalyst on the pore characteristics and electrochemical properties of the CDC.

2. Experimental

2.1. Sample preparation

The NbC powder (99.5%, ~2 μm, Changsha Xinlan Cemented Carbide Co. Ltd, P.R. China) was placed in a horizontal quartz tube furnace and heated to 800 °C under argon at atmospheric pressure. The argon flow was stopped when the temperature reached 800 °C, and the pure chlorine was passed through the quartz tube at a flow rate of 20 ml min⁻¹. The chlorination lasted for 2.5 h. The produced chloride gas was blown away by the chlorine flow immediately during the chlorination. At last, the quartz tube was purged with argon for 10 min at a rate of 1.5 L min⁻¹ and cooled down to room temperature. The waste chlorine and chloride gas were adsorbed by 1 M NaOH solution.

The produced CDC (denoted as NbC-CDC) and nickel nitrate (1:0.3 in weight) were added in ethanol, thoroughly mixed by ultrasonic stirring for 10 min, and dried in an oven at 50 °C overnight. Thereafter, the dried mixture was placed in a horizontal quartz tube furnace and heated to 1000 °C at a rate of 15 °C min⁻¹ under argon at atmospheric pressure. The treatment lasted for 4 h and cooled down to room temperature at argon atmosphere. The catalyzed sample (denoted as NbC-CDC/Ni) was washed by

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hydrochloric acid and then deionized water to pH=7 to ensure the sufficient removal of residual metals.

2.2. Characterization

The microstructure of the samples was observed using a JEM-2010 transmission electron microscopy (TEM) at 200 kV. X-ray

diffraction (XRD) patterns between 10 (2θ) and 70 (2θ) degrees were collected using Rigaku D/MAX-2500 powder diffractometer with Cu-K α radiation ($\lambda=0.154$ nm) operated at 40 kV and 200 mA. Gas adsorption/desorption analysis was done in the V-Sorb 2800TP surface area and pore distribution analyzer (Gold APP Instruments Corporation, China) with N₂ as adsorbent at 77 K. Prior to analysis, samples were degassed in vacuum at 200 °C for

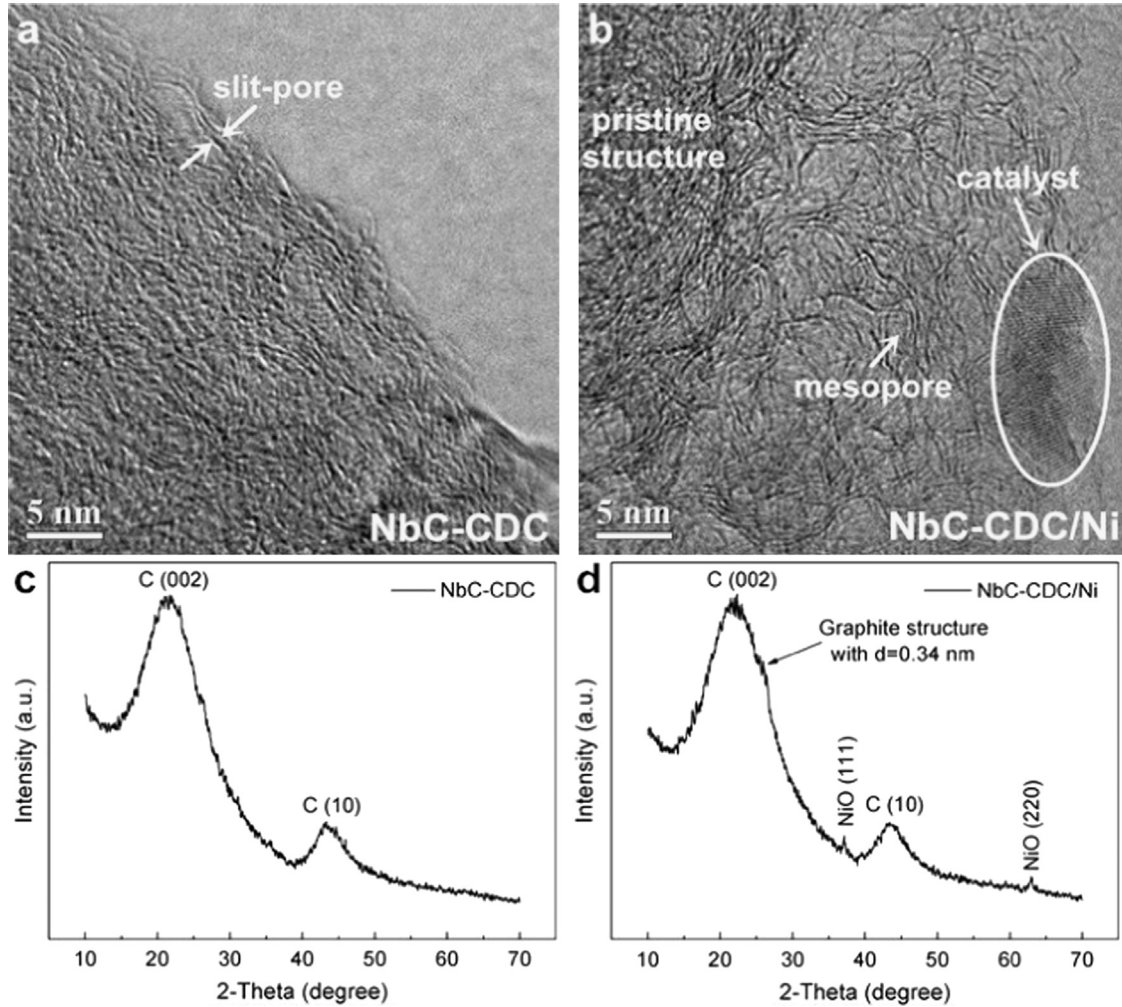


Fig. 1. The HRTEM images (a, b) and XRD patterns (c, d) of both NbC-CDC and NbC-CDC/Ni.

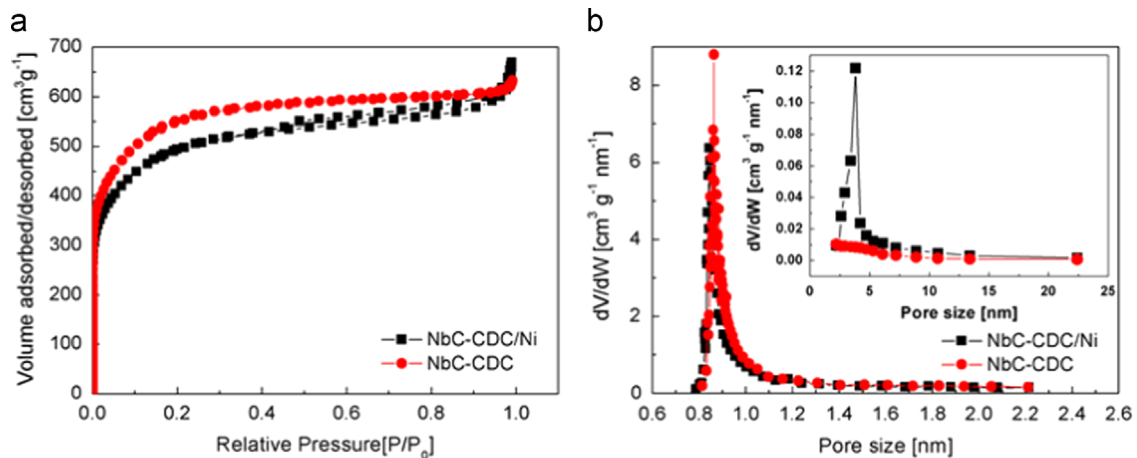


Fig. 2. Low-temperature N₂ adsorption/desorption isotherms (a) and pore size distributions (b) of both NbC-CDC and NbC-CDC/Ni. The inset in (b) is the mesopore size distribution.

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