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Synthesis, thermal stability, and photocatalytic activity of nanocrystalline titanium carbide

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ABSTRACT

Titanium carbide (TiC) was prepared via one simple route by the reaction of metallic magnesium powders with titanium dioxide (TiO_2) and potassium acetate (CH_3COOK) in an autoclave at 600 °C and 8 h. Phase structure and morphology were characterized by X-ray powder diffraction (XRD) and Scanning electron microscopy (SEM). The results indicated that the product was cubic TiC, which consisted of particles with an average size of about 100 nm in diameter. The product was also studied by the thermogravimetric analysis (TGA) and its photocatalysis. It had good thermal stability and oxidation resistance below 350 °C in air. In addition, we discovered that the cubic TiC powders exhibited photocatalytic activity in degradation of Rhodamine-B (RhB) under 500 W mercury lamp light irradiation.

1. Introduction

Among the transition metal carbides, TiC is particularly important for industrial applications due to its promising physical and chemical properties, such as high melting temperature, high hardness, low density and high resistance to corrosion and oxidation, high Young's modulus, high Vickers hardness, high abrasion resistance, good thermal conductivity and high thermal shock resistance [1–4]. Therefore, it is widely used for cutting materials, abrasive and anti-wear materials, aerospace materials, a substitute for tungsten carbide in cermets and so on. Because some properties of the materials are obviously affected by their size, it is meaningful to develop a facile and easy-going route to synthesize nanocrystalline TiC.

Traditionally, TiC powders were commercially synthesized via the carbothermal reaction of titanium dioxide and carbon in the temperature of approximately 2200 °C and 24 h. Hassine et al. [5] have reported that TiC powders were synthesized via microwave carbothermal reduction. This route has some obvious advantages. First, the reaction temperature is lower than the conventional method. Second, the total reaction time is shorter than the conventional method. However, the size of the products is with micro scale, which cannot satisfy the demands of nano-TiC for modern industry. Hence, it is desired to develop new techniques to prepare uniform TiC nanoparticles to meet the demands of industrial applications. Recently, attentions have been paid to synthesize nanocrystalline TiC at mild conditions. Some teams have reported to obtain nanocrystalline TiC at lower temperature than the conventional industrial method. Chandra et al. [6] have synthesized several different morphologies of nano-TiC using titanium gel precursor and nano-carbon particles under different conditions. Qi et al. [7] have reported that TiC nanowires were successfully synthesized by the reaction of TiO gas with methane in the presence of a catalyst containing Fe. Tong and Reddy [8] have obtained TiC nano-powders via the thermal plasma method. Lee and Kim [9] have reported that TiC nano-powders with the average size of 50 nm were synthesized by liquid–magnesium reduction of vaporized $TiCl_4 + CCl_4$ solution above 1000 °C. Bai et al. [10] have reported that nanocrystalline TiC hollow polyhedrons were prepared at 500 °C and 8 h, however, the reactants ($TiCl_4$, CaC_2) have some defects, such as hydrolysis, volatility and toxicity, which make the operation more complicated.

In this paper, we have developed a new convenient route to synthesize nanocrystalline TiC by the reaction of metallic magnesium powders with TiO_2 and CH_3COOK in an autoclave at 600 °C. In this route, TiO_2 and CH_3COOK were lower toxic and much easier to operate than other titanium sources (e.g. $TiCl_4$) and other carbon sources (e.g. CCl_4, CaC_2). Because the whole synthesis

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route was carried out in a sealed autoclave, it could be obtained that all manipulations were rather safe and convenient.

2. Experimental

All the chemicals were analytical grade and used without further purification. Initially, 0.01 mol TiO_2, 0.02 mol CH_3COOK and 0.05 mol metallic magnesium powders (excessive) were put into a mortar, followed by mixing these powders thoroughly. Then the mixture was transferred to a stainless steel autoclave under argon atmosphere. The autoclave was heated at 600 °C and then cooled to room temperature in the furnace naturally. The obtained product from the autoclave was washed several times with absolute ethanol, dilute HCl aqueous solution, distilled water to remove the impurities. Finally, the product was washed three times with absolute ethanol to remove water. The final product was dried in vacuum at 60 °C for 8 h. Black powders were obtained.

The obtained samples were analyzed by powder X-ray diffraction (XRD) on a Bruker D8 Advance X-ray powder diffractometer using Cu K-α radiation (wavelength λ = 1.54178 Å). 2-theta angles were from 15° to 90°. The morphologies of the samples were observed on a JEOL JSM-6700F scanning electron microscope, and the operating voltage was 10 kV. The thermogravimetric analysis was performed on a thermal analyzer (Model: Q600) below 1000 °C in air at a rate of

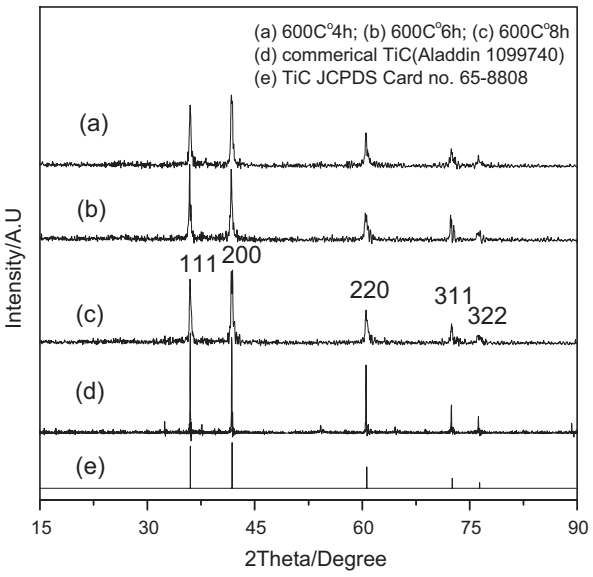

Fig. 1. The XRD patterns of the as-prepared TiC samples under different reaction conditions: (a) 600 °C, 4 h; (b) 600 °C, 6 h; (c) 600 °C, 8 h; (d) the commercial TiC and TiC (JCPDS Card no. 65-8808).

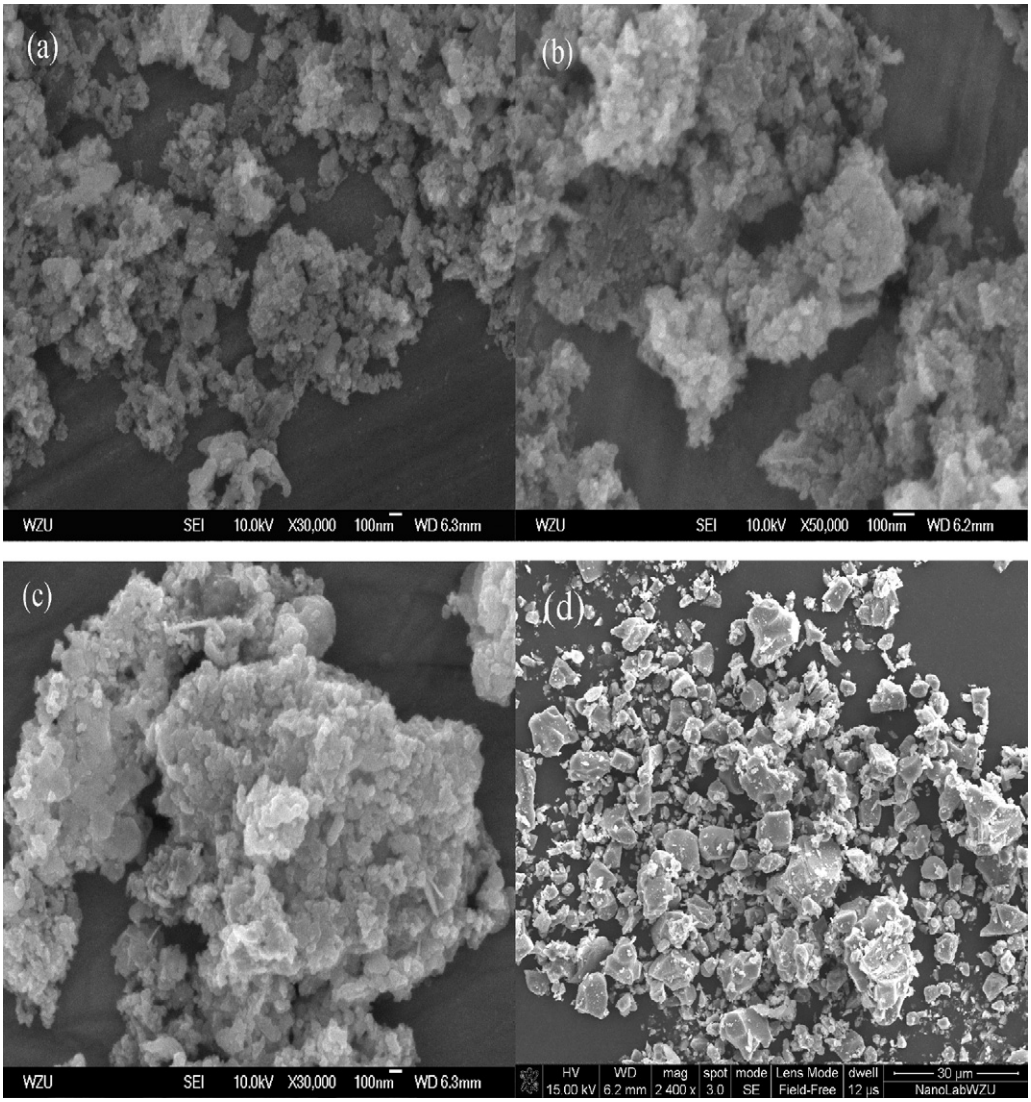

Fig. 2. SEM images of the as-prepared samples prepared under different reaction conditions: (a) 600 °C, 4 h; (b) 600 °C, 6 h; (c) 600 °C, 8 h and (d) the commercial TiC.