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Original Research Paper

Investigation of template effect on zirconium carbide synthesis process in carbothermal method at low temperature condition

Mohammad Mahdavi [a,*], Mazaher Ramazani [b], Zahra Darvishi [a]

[a] Department of Chemistry, Malek-ashtar University of Technology, Shahin-shahr, P.O. Box 83145/115, Islamic Republic of Iran
[b] Department of Materials Engineering, Malek-ashtar University of Technology, Shahin-shahr, P.O. Box 83145/115, Islamic Republic of Iran

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ABSTRACT

The template effect of carbon source precursors on ZrC synthesis at low temperatures were investigated by carbothermal reduction of $ZrCl_4$ using metallic sodium. The polymerization of naphthalene or toluene in the presence of $ZrCl_4$ was directed to formation of two ZrC products including nanorods and nanoparticles. The polymerization behavior of naphthalene or toluene had template effects on ZrC shaping. The ZrC nanorods/nanoparticles were characterized by X-ray and SEM. The results showed that $ZrCl_4$ in both condition was completely transformed to ZrC, after heat treatment in argon at temperatures above 600 °C. The SEM study revealed that the nucleation and growth of ZrC nanorods/nanoparticles occurred on the surface of derived polymer from naphthalene/toluene. There was no carbon residue from naphthalene/toluene in the products. The obtained ZrC particles exhibited face-centered cubic lattice structure and narrow size distributions.

1. Introduction

Zirconium carbide (ZrC) is one of the interesting members of refractory interstitial compounds with the NaCl-type structure [1]. ZrC has attracted considerable interest due to various unique properties, such as high hardness, high melting point, high chemical stability and high resistance to radiation, which makes it suitable for many applications, such as field emitters, coating of nuclear fuels and cutting tools [2,3]. Several different synthetic methods have been developed for the synthesis of ZrC, such as carbothermal reduction of ZrO_2 at elevated temperatures [4], solution-based processing at low temperature [5], mechanochemical reaction of zirconium metal by carbon [6,7], solid state metathesis [8], organometal preceramic method [9], Mg-thermite method [10], and co-reduction methods [11,12]. Wong and coworkers prepared metal carbide nanorods using carbon nanotubes [13]. Metal Carbide nanorods/nanoparticles play a noteworthy role in leading scientific and industrial research of nanotechnology [14]. Nowadays these nano-materials are being studied widely, due to their improved mechanical and physical properties [15].

Polymerization of toluene by Lewis acid catalysts; tend to create nanospheres of poly-toluene, since the reaction takes place in Meta position of toluene [16]. While the naphthalene polymerization turn into nanorods and linear chains of poly-naphthalene, due to polymerization in para position of naphthalene [17].

In this study, we attempted to produce ZrC nanospheres/nanorods using polymerization manners of toluene and naphthalene. For the first a simple method was developed for synthesis of ZrC nanorods at low-temperature, using $ZrCl_4$ and naphthalene.

2. Materials and methods

All chemicals including sodium metal (99.7%), naphthalene (99.98%), Zirconium (IV) chloride anhydrous (99.99%), and toluene (99.8) were purchased from Sigma–Aldrich). The composition of ZrC nanomaterials was characterized by X-ray diffraction pattern (XRD) on Bruker D8 ADVANCE instrument. The field emission scanning electron microscopy (FE-SEM) and energy dispersive X-ray analysis (EDAX) studies were done on SEM Philips XL30.

2.1. Preparation of zirconium carbide using naphthalene

All the reactions were carried out under argon atmosphere. In a typical experimental procedure, appropriate amounts of naphthalene (9.6 g) and $ZrCl_4$ (4.82 g) were mixed in a steel reactor under argon atmosphere. The reagents were stirred at 100°° and slightly excessive of Na (1.95 g) was added into the solution and

* Corresponding author. Tel.: +98 314 522 0520; fax: +98 314 522 0420.
E-mail addresses: mahdavi.mut@gmail.com, m.mahdavi@mut-es.ac.ir (M. Mahdavi).

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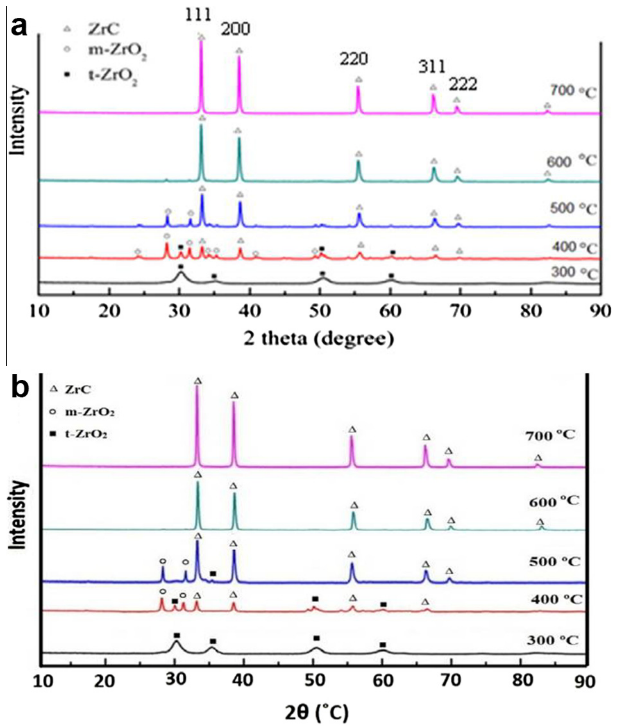

Fig. 1. XRD patterns of (a) $ZrCl_4$-naphthalene and (b) $ZrCl_4$-toluene reactions at different temperatures.

stirring was continued to evaporate excess naphthalene and remained a brown solid. The brown solid was heated in an electric furnace at various temperatures (300, 400, 500, 600 and 700 °C) under argon atmosphere for 1 h, then cooled to room temperature.

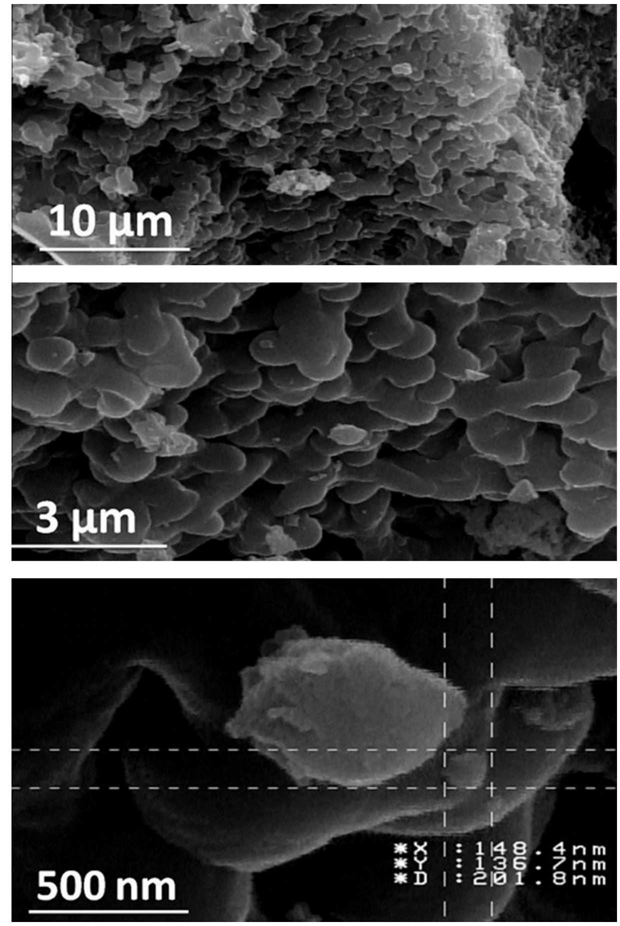

Fig. 3. FE-SEM pictures of ZrC nanoparticles.

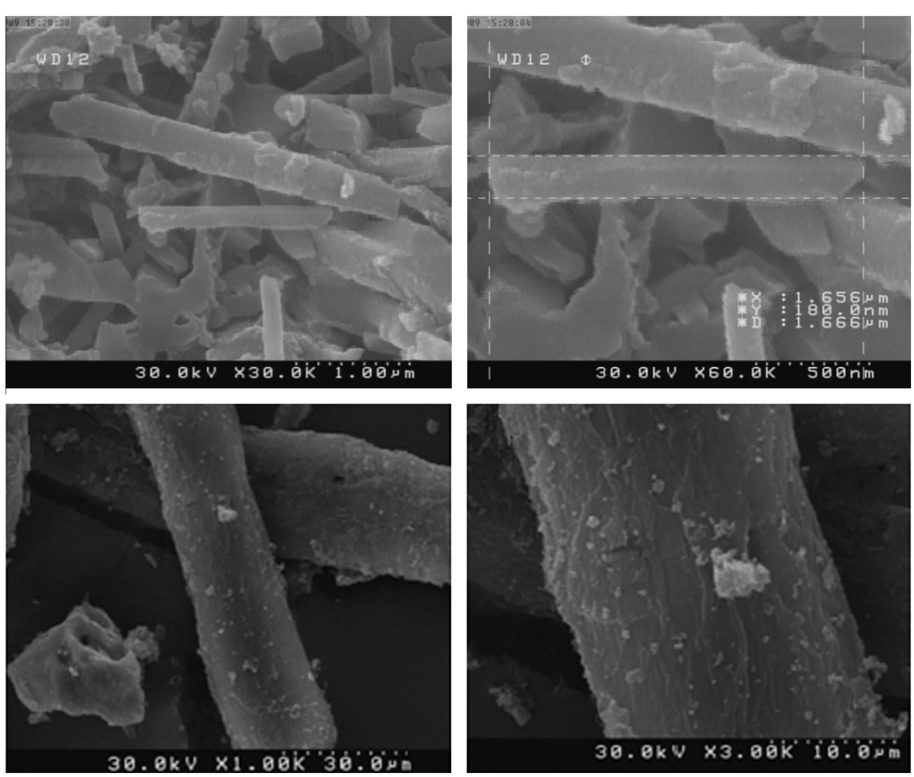

Fig. 2. FE-SEM pictures of ZrC nanorods.