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ScienceDirect

Procedia Manufacturing 10 (2017) 634 – 640

Procedia MANUFACTURING

45th SME North American Manufacturing Research Conference, NAMRC 45, LA, USA

Scalable manufacturing of 10 nm TiC nanoparticles through molten salt reaction

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Abstract

Titanium carbide (TiC) nanoparticles have great potential for strengthening metals as TiC has high hardness, high Young's modulus, good conductivity and excellent wear resistance. To enable effective Orowan strengthening effect, smaller TiC nanoparticles (e.g. < 10 nm) are highly preferred. However, small TiC nanoparticles (< 10 nm) are not available in market. Current production methods either cannot produce such small TiC nanoparticles or cannot manufacture nanoparticles in large scale. Here we explored a molten salt based reaction method that can manufacture TiC nanoparticles below 10 nm. Diamond nanoparticles were used as the carbon source and reaction template. Titanium powders dissolve in molten salt and deposit on the diamond nanoparticles and then reacts with diamond to form TiC nanoparticles. This method provides a simple and inexpensive pathway to manufacture TiC nanoparticles below 10 nm and opens up the opportunity for making high performance metal matrix nanocomposites (MMNCs) reinforced by TiC nanoparticles.

Peer-review under responsibility of the organizing committee of the 45th SME North American Manufacturing Research Conference

Keywords: molten salt reaction; TiC nanoparticles; nanomanufacturing; metal matrix nanocomposites

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doi:10.1016/j.promfg.2017.07.066

1. Introduction

TiC offers high hardness, high strength, high Young's modulus, and good electrical conductivity due to its partial metallic bonds, and thus it is considered as a good reinforcement for metal matrix nanocomposites (MMNCs) [1-4]. There are numerous research activities on using TiC micro/nano particles as reinforcements for strengthening metals such as aluminum [4] and magnesium [3]. Recently, we have incorporated as high as 9 volume percent (vol.%) 60 nm TiC into Al by using a self-incorporation method with the assistance of $KAlF_4$ flux [4]. However, the strengthening effect of resultant MMNC samples was mainly from the load bearing effect [5] instead of Orowan strengthening which involves bowing dislocation between particles during plastic deformation. To active effective Orowan strengthening in metal matrix nanocomposites, shorter distance between particles is preferred. Thus to achieve a better strengthening effect, smaller nanoparticles (below 10 nm) is desired. Base on Orowan strengthening theory, if same volume percent of 10 nm and 60 nm is incorporated in metals such as aluminium, the strength and ductility improvement in case of 10 nm is much higher than 60 nm. Therefore, there is a strong motivation to incorporate small TiC nanoparticles (e.g. less than 10 nm) into metal matrix for ultra-strong MMNCs.

Nevertheless, TiC nanoparticles below 10 nm are not readily available in the market. Several methods including carbonthermal reduction process, Sol-Gel process, plasma method and combustion synthesis have been developed [6-9]. The most popular and commercialized method is the carbonthermal reduction method [10-12]. However, these methods at least will bring one of the following problems: (1) formation of coarse particles due to a high temperature reaction, (2) the formation of a non-stoichiometric powder ($TiC_{0.7\sim0.9}$), (3) time consuming and expensive milling process. Therefore, a new scalable and inexpensive method which can produce small size (below 10 nm) TiC nanoparticles is needed.

Molten salt reaction to produce nanoparticles is a relatively new, simple and low cost method. There are several research activities using molten salt systems for metal deposition especially titanium on different types of ceramic material such as diamond, TiN, Al_2O_3, SiC, CNTs and carbon fibers [13-17]. While other deposition techniques such as Physical vapor deposition (PVD), chemical vapor deposition (CVD) are mainly applied to flat surface, molten salt based reaction enables us to coat different surfaces include particles.

Inspired by molten salt assisted coating method, we deposited a titanium layer on the 10 nm diamond nanoparticles, and then allowed titanium to react with diamond to synthesize TiC nanoparticles. As titanium dissolves in the molten salt, the final size of the TiC nanoparticles is determined by the size of the diamond nanoparticles. Moreover, it is shown that other carbon source such as carbon black, which is significantly cheaper than diamond, can also be used for molten salt based reaction [18]. Thus this molten salt reaction method opens up a new pathway to manufacture TiC nanoparticles (< 10 nm) for fabrication high performance MMNCs in industrial scale.

2. Materials and methods

Diamond nanoparticles (3-10 nm, US Research Nanomaterials, Inc.) were mixed with 40 weight percent (wt.%) Potassium chloride (KCl) (99.1%, Fisher Scientific), 40 wt.% Sodium chloride (NaCl) (99.5%, Fisher Scientific), and 20 wt.% Potassium hexafluorotitanate (K_2TiF_6) (Sigma Aldrich) in ethanol with an ultrasonic processing for 2 hours. For the ultrasonic processing, a titanium probe was inserted about 6 mm deep into the liquid mixture. The probe was attached to a booster (Misonix Sonicator 3000). A peak-to-peak vibration amplitude of 60 μm and a frequency of 20 kHz were used [19]. The volume fraction of diamond nanoparticles in the salt mixture is 5 vol.%. Then ethanol was evaporated and the powder mixture was dehydrated in a vacuum furnace at 150 °C for 12 hours. Titanium powders (44 μm, 99%, Alfa Aesar) were added and mixed with the dehydrated powder mixture by a mechanical shaker (molar ratio between carbon and titanium was designed to be 1:2.5 and 2:1). The schematic of the experiment setup is shown in Figure 1. The powder mixture were put inside a graphite crucible with an inner diameter of 100 mm and a height of 120 mm and covered by an aluminium oxide lid. Then the graphite crucible with lid was sealed in a stainless steel autoclave for high temperature reaction. Under argon gas protection, the autoclave was heated up by an electrical resistance furnace to 900 °C and held for 1 hour. Then the autoclave was cooled down in air and synthesized product was removed.