Int. Journal of Refractory Metals and Hard Materials 56 (2016) 96–103

Contents lists available at ScienceDirect

Int. Journal of Refractory Metals and Hard Materials

journal homepage: www.elsevier.com/locate/IJRMHM

Effects of dispersants on dispersibility of titanium carbide aqueous suspension

H. Foratirad [a,*], H.R. Baharvandi [a], M.G. Maragheh [b]

[a] Department of Materials Science and Engineering, MUT, Tehran, Iran
[b] Nuclear Science Research School, Iran

ARTICLE INFO

Article history:
Received 1 November 2015
Received in revised form 6 December 2015
Accepted 21 December 2015
Available online 23 December 2015

Keywords:
Titanium carbide
Suspension
Dispersibility
Zeta potential
Sedimentation test

ABSTRACT

In this study, the dispersing phenomenon of titanium carbide suspensions has been investigated using various dispersants. The effect of pH and dispersant concentration on the dispersibility of the powder has been studied via sedimentation and zeta potential test. To optimize the pH range for the best dispersibility, the sedimentation test has been carried out in various dispersant concentrations in wide pH range. The zeta potential of TiC suspensions, both with and without polyelectrolyte addition, is examined as a function of pH. Zeta potential studies show that the isoelectric point of TiC powder is at pH 3.1. The use of an anionic polyelectrolyte, tetramethylammonium hydroxide in the optimum concentration, significantly increased the stability of suspension. The maximum value of the zeta potential −60 mV is obtained in 0.4 wt.% at pH 8. The addition of a cationic dispersant, polyethylenimine, significantly alters the isoelectric point and shifts to the basic pH. The maximum stability of suspension was achieved in 2 wt.% at pH 8. The result showed that nonionic dispersant polyethylene glycol 400 is not a good dispersant for TiC suspension. The surface charge and potential do not change in the presence of this dispersant.

1. Introduction

The titanium carbide with the high modulus, hardness, strength retention at elevated temperatures, good thermal stress resistance, excellent wear and oxidation resistance and electro conductivity has been widely used in a number of industrial applications, such as heat engines and gas turbines [1]. Also, its excellent combination of the thermal properties (low coefficient of thermal expansion and high thermal conductivity) and availability has made it liable to be in an important class of materials as reinforcement for high volume fraction metal matrix composites (MMCs) for metal-cutting tools and wear-resistance applications [2]. At the same time, porous titanium carbide ceramics possess an attractive set of properties such as low weight, variable porosity, high temperature stability, low thermal conductivity, high permeability and low specific heat capacity. Thus, it has been widely used in many applications [3–7]. For many of these applications, slurry processing is widely used because the ceramic powder itself is inherently difficult to handle and shape into the desired components [8].

However, an important aspect of fabrication sequence in slurry processing for production of porous ceramics or ceramic composites which can improve subsequent sintering and final properties of bodies, is obtaining a homogenous structure together with the serviceable packing of powders at the consolidation stage of the green body. It is imperative to have slurry with good dispersion and a desirable rheological behavior, because the quality of dispersion controls the casting behavior and the resulting green-body properties [9–10]. Inhomogeneous dispersion, such as flocculation or agglomeration, is harmful to the sintering of composite materials or for the properties of porous ceramic and always introduces processing flaws [11]. However, powder dispersion characteristics and the flow properties of the resulting suspensions are controlled by the powder surface charge, which varies with the type and even the source of powder. Due to this surface property variation it is often necessary to use a specific pH or a certain dispersing agent for each individual powder [12–13].

In order to disperse a powder in water, the surface charge properties of the powder have to be controlled. The dispersion of colloidal particles in an aqueous medium by electrostatic repulsion and/or steric hindrance relies on the control of surface charge of ceramic particles by dispersant [14]. The former mechanism is managed by both attractive and repulsive forces between those particles. The net effect of these forces determines the state of dispersion of particulate suspensions. The repulsive forces between particles dominating the attractive forces result in satisfactory dispersions. In general, an electrostatic stabilization of suspension can be achieved by manipulating electrostatic charges on the particle surface with controlling pH slurry away from the isoelectric point pH (pH_{IEP}) of the powder (electrostatic stabilization) and by

* Corresponding author.
E-mail address: hforatirad@aeoi.org.ir (H. Foratirad).

http://dx.doi.org/10.1016/j.ijrmhm.2015.12.007

using polyelectrolytes as dispersing agents (electrosteric stabilization), which is absorbed onto the surface of the particles, thus increasing the repulsive force [15–18].

In this study, the effect of various dispersants on TiC powders was investigated in order to achieve the desired dispersion of the powder in water. Tetramethylammonium hydroxide (TMAH), Polyethylene glycol 400 (PEG 400) and Polyethylenimine (PEI) dispersants were conducted to disperse TiC particulates in aqueous solution. Then the effect of the type and dispersant content on the stability of TiC aqueous suspension was also studied via measuring the suspension sedimentation and zeta potential in order to optimize the use of dispersant.

2. Experimental

2.1. Material

The TiC powder used in the experiments performed was purchased from Pacific Particulate Materials Ltd. (TiC 2012 Batch# 20125339) with a quoted average particle size of 3–5 μm. and 0.11% retained free carbon. The TiC powder was first analyzed as received to verify the particle size distribution and to identify the particle morphology which were scrutinized using scanning electron microscopy (Philips SEM 515). The specific surface area of the TiC powder was measured by means of a single point BET (Quantachrome instrument, Nova model). Fig.1a shows the grain size distribution of TiC powders. It can be seen that a mean particle size (d50) is characterized below 5 μm and a specific surface area (BET) is $<2.2\ m^2/g$. A scanning electron microscopy image of the sample is shown in Fig. 1b. The SEM image revealed irregular particles with slightly plate-like shape. The observed morphology can be explained by the powder production procedure, as the TiC is milled by the manufacturer to produce the desired particle distribution. This would explain the fractured irregular particles and the abundance of fine fragments.

To conduct a comparative study, three types of dispersants were used. The first one consisted of tetramethylammonium hydroxide (25% aqueous solution, Sigma-Aldrich Co) having the molecular weight of 91.153 g/mol. TMAH is a quaternary ammonium salt with the molecular formula $N(CH_3)_4^+ OH^-$. The second dispersant was polyethylenimine (PEI) with a molecular weight of 25,000 g/mol (Sigma-Aldrich Co). PEI is a polymer with repeated units composed of the amine group and two carbon aliphatic CH_2CH_2 spacers. It is positively charged in a low pH range and provides a steric repulsion force via its branch. The last dispersant was polyethylene glycol 400 (Sigma-Aldrich Co), a non-ionic polymer with the molecular formula $H(OCH_2CH_2)_nOH$. A representative diagram of the structures of TMAH, PEG and PEI is shown in Fig. 2.

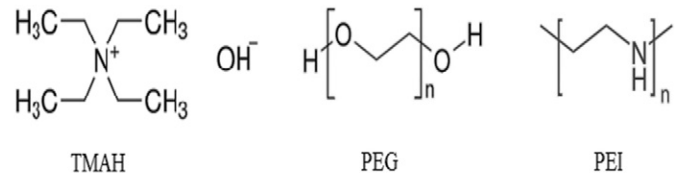

Fig. 2. Schematic representation of the structures of TMAH, PEG and PEI.

2.2. Suspension preparation

The suspensions used for all measurements were prepared by adding the as-prepared TiC powder to deionized water at the selected volume fraction. A density of $4.93\ g/cm^3$ for TiC was used in the calculations. The amounts of added dispersant were expressed as a dry weight percentage of the TiC powder. The procedure for preparing the suspensions is as follows: (i) the dry powder was slowly added to the deionized water with the desired dispersant content; (ii) the suspension was then ultrasonically deflocculated (JY96-IIN, Ningbo Scientz Biotechnology Co. Ltd., Zhejang, China) at an output power of 180 W to break up any agglomerates; and (iii) after ultrasonication, the suspension was stirred for 4 h continuously before analysis. The pH of TiC suspensions was adjusted by adding 0.1 M hydrochloric acid (HCl) and 1 M sodium hydroxide (NaOH) solutions.

2.3. Sedimentation measurements and FT-IR analyze

To investigate the suspension behavior and to determine the optimal processing conditions for the suspensions, several sedimentation trials were carried out. Sedimentation tests were carried out under various conditions to determine the pH_{IEP}, the best dispersant type and the optimum dosage of dispersants. 2 vol.% TiC suspensions with various amounts of dispersants were prepared by ultrasonic treatment and then stirred for 4 h to reach equilibrium. The initial pH of all the suspension was adjusted to the desired value. Sedimentation measurements were carried out by drawing 10 mL samples of the suspensions into 10 mL graduated test tubes and then covering them with a cap to avoid the evaporation of water. The stability of the suspensions was evaluated by time to determine the variation of dispersion height versus the setting time at various intervals, until a severe flocculation was observed.

In this study, the dispersion height is defined as the distance between the dispersion/supernatant interface and the bottom of the tube, including the height of any sediment. Thus, a larger dispersion height indicates better dispersions.

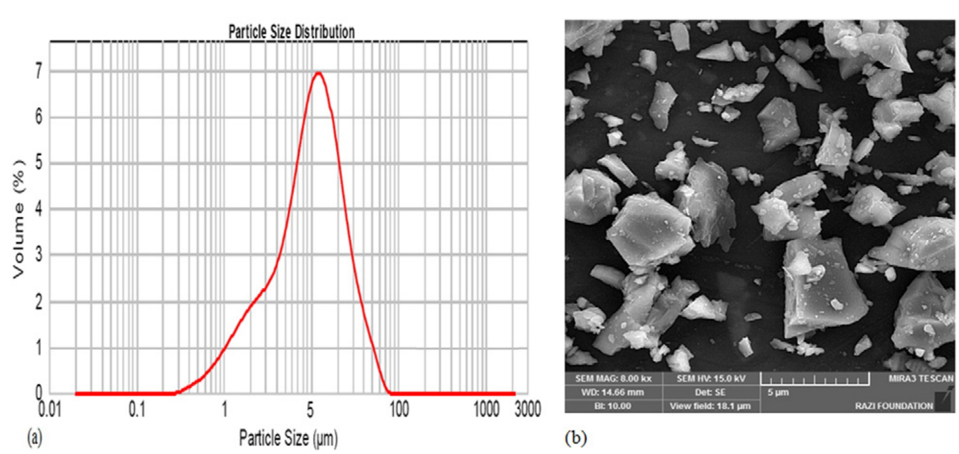

Fig. 1. (a) Particle size distribution of TiC, (b) SEM image of the TiC powders used in the experiments.