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Droplet size control in the filter expansion aerosol generator

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Abstract

A new capability for droplet generation through the filter expansion aerosol generator (FEAG) process has been found. The mean sizes and size distributions of droplets generated by the FEAG process have been estimated from the resulting powders. The mean sizes of the droplets could be controlled from 1.7 to 25 µm. Surface tension and the viscosity of spray solutions controlled by adding polyethylene glycol (PEG) affected the mean sizes of the droplets. The mean sizes of droplets generated at a pressure of 160 Torr changed from 4.3 to 12.1 µm when the concentrations of PEG were changed from 1.6×10^{-4} to 1.6×10^{-3} M. The addition of PEG into the spray solution improved the size distribution of the droplets. The mean sizes of droplets generated from the spray solution with a concentration of 4.8×10^{-4} M PEG changed from 1.7 to 25 μ m when the reactor pressures were changed from 60 to 400 Torr. The droplets generated at pressures between 160 and 360 Torr had a narrow size distribution. © 2008 Elsevier Ltd. All rights reserved.

Keywords: Powders-gas phase reaction; Traditional ceramics; Powders-chemical preparation

1. Introduction

Spray aerosol generators can be applied in the formation of thin films and the preparation of fine powders by spray pyrolysis.¹⁻³ Small, uniform-sized droplets are needed for the preparation of ultrafine powders and thin film for integrated circuits with submicron features. For these applications, the droplet size should be around 1 µm and large production rates are required. An ultrasonic spray generator offers a reasonably high production rate and yields droplet diameters on the order of µm. Ultrasonic spray generators have therefore been used for the commercial production of useful ceramic and metal powders.

Kang and Park developed a new liquid aerosol generator named the filter expansion aerosol generator (FEAG).⁴ The FEAG process comprised a liquid aerosol generator which produces fine-sized droplets under low pressure. It has been applied to the preparation of advanced ceramic, metal and glass powders.^{5–9} The resulting powders have similar characteristics to those prepared by ultrasonic spray pyrolysis.

The required mean size for ceramic, metal, and glass powders used in the electronic industries is becoming smaller and smaller. However, in some application fields, powders with mean

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sizes between 3 and 10 µm are required. Therefore, droplet generators must be able to generate droplets with mean sizes of several microns and several tens of microns. Pneumatic nozzles have the capability of producing droplets with mean sizes of several tens of microns.¹⁰ On the other hand, the droplets generated by these pneumatic nozzles have a broad size distribution. Electro-spray generators can be used to generate droplets smaller than 2 µm but the production rate of these size droplets is restrictive.¹¹ Ultrasonic spray generators are also restricted in the generation of droplets smaller than 2 µm or droplets larger than $10 \,\mu m$.

In this study, a new capacity for droplet generation via the FEAG process was investigated. The mean sizes of the droplets were controlled from micron sized to several tens of microns. The droplet generation characteristics were estimated from the characteristics of aluminum oxide powders. Surface tension and viscosity of the spray solution were controlled by changing the concentrations of polyethylene glycol added to the spray solutions. The pressure of the reactor was changed from 60 to 400 Torr by controlling the capacity of the vacuum pump.

2. Experimental

The schematic diagram of the modified FEAG process used in this study is shown in Fig. 1. The apparatus consists of a

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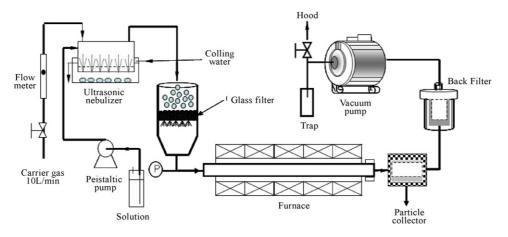


Fig. 1. Schematic diagram of filter expansion aerosol generator (FEAG) for preparing Al₂O₃ powders.

porous glass filter, an ultrasonic spray generator, a vacuum pump and a bag filter. An ultrasonic spray generator was used as a supply system for the spray solution to ensure a continuous, uniform quantity. The detail droplet formation mechanism in the FEAG process has been investigated in previous research.⁴ The spray solution was supplied through an ultrasonic spray generator using carrier gas on to a glass filter surface where it formed a thin liquid film. The liquid flows through pores of the glass filter with carrier gas. Liquid flow rate is controlled so as to maintain two phase flow in the pores of the glass filter. At the bottom of the filter, multiple expansion occurs through multiple channels and aerosol stream is formed.

Aluminum oxide powders were prepared from aqueous and polymeric spray solutions using the FEAG process. The aqueous solution was prepared by dissolving aluminum nitrate in distilled water. The concentration of aluminum nitrate was 0.2 M. Polyethylene glycol (PEG 200) was dissolved in the spray solutions to control surface tension and viscosity. The concentration

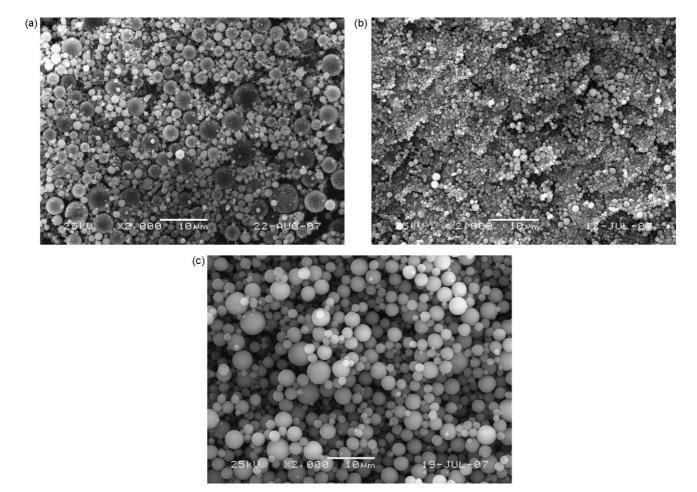


Fig. 2. SEM photographs of alumina powders prepared by FEAG process (0.2 M Al nitrate, 900 $^{\circ}$ C, 160 Torr): (a) no additive, (b) 4.8×10^{-4} M, and (c) 16×10^{-4} M.

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