Preparation of Ultrafine $\gamma$-Al$_2$O$_3$ Particles in Non-ionic Water in Oil Microemulsions

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Abstract  Ultrafine $\gamma$-Al$_2$O$_3$ particles are synthesized in Triton X-100/n-hexanol/cyclohexane/water microemulsion by mixing two separately prepared microemulsions containing Al(NO$_3$)$_3$ and (NH$_4$)$_2$CO$_3$ respectively. The ultrafine Al$_2$O$_3$ particles are characterized by transmission electron microscopy (TEM) and X-ray diffraction (XRD) and their size and distribution are measured. The effects of water, surfactant and reactant concentrations on the particle size and distribution are studied. The results show that the particle size and distribution can be changed by varying the preparation conditions, and the size of the microemulsion droplets has a controlling effect on the size of the particles. A possible mechanism of ultrafine particles (UFPs) prepared by microemulsions is proposed.

Key words: ultrafine particles, aluminum oxide, w/o microemulsion

1 Introduction

The synthesis of ultrafine particles (UFPs) is of increasing scientific and technical interest. The small size of these particles endows them with unusual structural, electronic, optical, magnetic and chemical properties, leading to many important applications such as catalysts, electro-optical devices and fine ceramics.

Aluminum oxide (Al$_2$O$_3$) has been widely used to make fine ceramics, because of its special properties such as high strength, high hardness, heat resistance and further resistance. With the development of science and technology and continued research on fine ceramics, more and more amount of ultrafine Al$_2$O$_3$ particles are urgently demanded of high quality with specified particle size, good size uniformity and purity. Therefore, it is very important to study and develop synthesis process of ultrafine Al$_2$O$_3$ particles. There are several methods to prepare ultrafine Al$_2$O$_3$ particles, such as electron beam evaporation and liquid precipitation.

There is still important challenge in obtaining ultrafine Al$_2$O$_3$ particles by simple and powerful methods nowadays.

Recently, many approaches to the synthesis of UFPs have focused on constraining the reaction environment by using amphiphilic association structures such as Langmuir-Blodgett (LB) films, vesicles and water-in-oil (w/o) micro-emulsions or reverse micelles. Specifically, w/o microemulsions have attracted considerable attention in view of their aqueous cores, which are ideal media for the preparation of UFPs.

A w/o microemulsion is a thermodynamically stable, optically isotropic dispersion of surfactant-stabilized microdrops of water in an external oil phase. The highly dispersed nanosize droplets are suitable for particles synthesis and have the potential for controlling the microenvironment where chemical reactions may occur. For example, the surfactant-stabilized microcavities provide a cage-like effect that limits particle nucleation, growth and agglomeration.

In this paper, ultrafine Al$_2$O$_3$ particles are synthesized in Triton X-100/n-hexanol/cyclohexane/water w/o microemulsions by mixing two separately operated microemulsions containing Al(NO$_3$)$_3$ and (NH$_4$)$_2$CO$_3$, respectively.
The ultrafine Al₂O₃ particles are characterized by transmission electron microscopy (TEM) and X-ray diffraction (XRD). The effects of water, surfactant and reactant concentrations on the particle size and distribution are studied. A possible mechanism of UFPs prepared by microemulsions is discussed.

2 Experiments

2.1 Materials

The non-ionic surfactant Triton X-100 (polyoxyethylene tert-octylphenyl ether with an average number of ethylene oxide (EO) units of 9.5, hereafter referred to as TX-100) was obtained from Rohm & Haas. Al(NO₃)₃ (A.R. grade), (NH₄)₂CO₃ (A.R. grade), n-hexanol (A.R. grade), and cyclohexane (A.R. grade) were purchased from Shanghai Chemical Agent Co. The water used in this work was deionized and distilled.

2.2 Preparation of microemulsions

We selected a microemulsion system with TX-100 as the surfactant, n-hexanol as the cosurfactant, cyclohexane as the continuous oil phase, and a salt solution as the dispersed aqueous phase. This system solubilizes considerable aqueous phase to form stable w/o microemulsions[11]. For the preparation of w/o microemulsions, TX-100 and n-hexanol were first mixed at weight ratio of 3:2 to form a surfactant mixture and a blend was formed. A solution of the blend in cyclohexane was then prepared with a certain volume content (e.g. 10% (v/v)). Water-in-oil microemulsions were obtained at room temperature by adding different amount of aqueous phase to this blend/cyclohexane solution. The volume content of the aqueous phase in microemulsions was varied in the range between 2.0 % and 3.0 % (v/v). When aqueous salt solution Al(NO₃)₃ or (NH₄)₂CO₃ was used as the aqueous phases, the concentration of the salt solution was 0.1m, water content was 2.0 % (v/v), blend content was 16 % (v/v). Fig.3 presents the XRD pattern of Al₂O₃ UFPs after thermal treatment. One can see from the pattern that the particles are γ-Al₂O₃.

3 Results and Discussions

3.1 Physical properties of Al₂O₃ UFPs

Fig.1 shows the photograph of TEM of Al₂O₃ UFPs and it is observed that the particles are spherical with narrow size distribution. Fig.2 shows the particle size and distribution of the same sample, where Dᵥ=21nm, GDS=1.5. The conditions of preparation were as follows: concentrations of the salt solution Al(NO₃)₃ and (NH₄)₂CO₃ were 0.1m, water content was 2.0 % (v/v), blend content was 16 % (v/v). Fig.3 presents the XRD pattern of Al₂O₃ UFPs after thermal treatment. One can see from the pattern that the particles are γ-Al₂O₃.

![TEM photograph of Al₂O₃](image1)

![The log normal distribution of particle size](image2)