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Original Research Paper

The nanostructure evolution of Ag powder synthesized by high energy ball milling

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ABSTRACT

In this paper, nanostructured silver with an average crystallite size of 28 nm and internal strain of 0.44% was synthesized by mechanochemical reduction of Ag_2O using graphite in a high energy planetary ball mill. XRD, SEM and TEM techniques were used to characterize the structural evolution and morphological changes of products. The results showed that the reaction is progressed by a nucleation and growth mechanism process. Although the changes of crystallite size and internal strain in Ag_2O were regular during the milling, there was an irregularity in the aforementioned parameters of Ag particles. This irregularity was probably owing to the progressive generation of silver during the milling.

1. Introduction

Application of milling process to create mechanochemical effects such as phase transformation and solid state reaction is widely available in literature [1–4]. It has been shown that the reaction rate was enhanced and dynamically extended during the milling as a result of mixing processes and microstructural refinement [5]. Silver nanostructure have been prepared by different methods such as electrochemical method, thermal decomposition, laser ablation, microwave irradiation, sonochemical synthesis, chemical reduction, photochemical reduction and radiolysis and silver organosols preparation [6]. However, there has been no research directed to the mechanochemical synthesis of silver nanoparticles yet. This research was carried out to synthesis of Ag nanostructure through solid state reaction between Ag_2O and graphite using mechanochemical reduction. Also, the mechanism of Ag powders refinement was investigated in more detail according the morphological and microstructural observations.

2. Experimental procedure

Starting materials were commercially pure Ag_2O powder (99%, 5–40 μm and graphite (99.9%, 10–50 μm). Mechanochemical reduction of Ag_2O together with 40 mol% of extra graphite as reducing agent (according to Eq. (1)) was performed in a planetary ball mill.

$$2Ag_2O + xC = 4Ag + CO_2 + (x-1)C, x = 1.4 \quad (1)$$

The excess graphite was used as a diluent and supplied maximum interaction between the precursors during the milling. Experiments were carried out in a high-energy planetary ball mill. Details of ball milling process were given in Table 1. The phase identification of the products was determined by Philips PW-1730 X-ray diffraction (XRD) using Cu Kα radiation. The line broadening owing to the instrument was computed from Warren's method [7,8]. The average crystallite size and internal strain was calculated using Williamson–Hall plot [9]. The morphology, size and size distribution of products were evaluated by SEM (JEOL-JSM 5310) equipped with an energy dispersive spectrometer (EDS) (Oxford Instrument) and TEM (CM200Philips), respectively. Samples preparation for TEM studies were done by placing drops of the silver nanoparticles/ethanol solutions on the carbon-coated TEM grids. The histograms of size distribution were calculated from the TEM images by measuring the diameters of at least 85 particles. The Microstructure Measurement Program and Minitab Statistical Software have been used to determine the average size of each particle.

3. Results and discussion

Fig. 1 shows XRD spectra of the powder mixture milled for various times, revealing the structural evolution as a function of milling time in the powder mixture. As milling time increased, the peaks of Ag_2O are gradually broadened and their height decreased. The Ag peaks appear between 3 and 6 h milling. Further milling resulted in an increase in the Ag peaks intensities, and single phase of Ag appeared after 22 h milling time.

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