Thermal Plasma Synthesis of Nano Composite Particles

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Nanocomposite particles were synthesized from a bulk ZrVFe alloy ingot by transferred DC thermal plasma. Effects of plasma gas flow rate on the characteristics of produced nanocomposite particles were investigated. The characteristics of the synthesized powder were analyzed by field scanning electron microscopy (FE-SEM), light scattering particle size analyzer (PSA), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), and Brunauer-Emmett-Teller (BET) surface area analyzer. As the flow rate of plasma gas increased from 20 L/min to 40 L/min, the average particle size decreased from 91 nm to 55 nm, the particle size distribution became narrower, the surface area increased from 200 m²/g to 255 m²/g, the particle composition was nearly unaffected, and the particle crystallinity was improved.

Keywords: thermal plasma, nano composite particle, particle size distribution, quenching rate

1. Introduction

Plasma Display Panel (PDP) is considered as the most promising candidate for large area display among various Flat Panel Displays (FPD’s) due to its manufacturing process appropriate for a large displaying area, high speed addressing ability and good display quality [1]. The large plasma display panel is divided into tiny cells by barrier ribs, and the electric discharge gas filled in the cell is one of important factors determining the display quality and life time of the PDP. By the nature of plasma, heavy ions in the cell continuously bombard surrounding walls during operation, resulting in the emission of undesired species from the wall, in turns contaminating the plasma gas, and consequently degrading the display quality. To extend the PDP’s life time with good display quality, it is necessary to continuously eliminate contaminants emitted from protective layer, barrier rib and fluorescent substances[2,3]. Installation of getter materials, such as Zr-V-Fe alloy powder, in the PDP cell is a potential solution to overcome such problems.

In fact, Zr-V-Fe getter materials are currently utilized in various applications[4,5] and commercially available (for an example, ST707 from SAES Getters, Inc.). However the particle size of the commercial getters is in the range of micrometers, limiting their applicability to the PDP cell. Hence it is imperative to produce the Zr-V-Fe getter material with the particle size in the range of nanometers.

Nano particles can be produced by various methods, such as sol-gel [6], spray pyrolysis [7], infrared heating [8], freeze-drying [9], laser ablation [10], wire explosion [11], and thermal plasma [12]. While most methods, requiring pre- and post-treatment, are time consuming and expensive, thermal plasma is simple and fast, thus cost effective technique to produce nano particles, specially for materials with extremely high evaporation temperature such as Zr. In this work, nanocomposite particles were prepared from a bulk ZrVFe alloy ingot by transferred DC thermal plasma. Effects of plasma gas flow rate on the characteristics of the produced nanocomposite particles, such as particle size distribution, mean particle size, surface area, particle composition and crystallinity, were investigated.

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2. Experiment

Figure 1 shows a schematic diagram of the thermal plasma system employed to synthesize ZrVFe alloy nano particles. The system consists of five sections: a DC plasma torch generating plasma flame from arc, a reaction chamber providing extremely high temperature environment for the formation of nuclei and clusters as well as for the growth of nano particles, a quenching tube in which the synthesized nano particles are quickly cooled down to prevent further growth, a collection chamber where the particles are deposited on a filter, and a scrubber eliminating pollutants from the effluent.

The constituent elements considered for the generation of nano composite particles were zirconium (Zr), vanadium (V) and iron (Fe). They were melted together by arc in a vacuum chamber, forming a bulk ZrVFe alloy ingot to be used as a raw material. The molar ratio of the constituent elements in the raw material was 57:35.8:7.2 so as to mimic the composition of commercially available getter ST707.

The plasma torch was operated in a transferred mode so that the arc extended from the electrode of the plasma torch to a raw material, ZrVFe alloy ingot. The ingot was placed 3 cm below the plasma nozzle, the input current to generate thermal plasma was fixed at 150 A while the flow rate of the plasma gas, pure argon, was varied from 20 L/min to 40 L/min. The characteristics of the particles produced for 20 min were analyzed by FE-SEM (FEI, Quanta 200), EDS (Horiba), XRD (Rigaku, MAX-2500V), PSA (Malvern, Maserizers & Zetasizers) and BET analyzer (Micrometrics, ASAP2020).

3. Results and Discussion

Figure 2 shows the SEM images and size distributions of the particles synthesized by the transferred thermal plasma with the various flow rates of the plasma gas. From the SEM images, the particles were found to be spherical although severely agglomerated, and the particle size was found to decrease as the flow rate increased. Although the shape of the particle size distribution obtained from the PSA was almost unaffected as the flow rate increased from 20 L/min to 40 L/min, the portion of smaller particles near 50 nm was found to noticeably increase. In the transferred thermal plasma system, the raw material first becomes liquefied by arc and then evaporated in thermal plasma environment, super saturation in vapor phase generates abundant nuclei subsequently forming clusters which grow to particles by condensation and coalescence. As the plasma gas flow rate increase, the current density of the arc more confine in the center, increasing the maximum temperature of the plasma flame. Since the plasma flame radially shrinks,

Figure 2. SEM images and particle size distributions for particles synthesized with plasma flow rates of (a) 20 L/min, (b) 30 L/min, and (c) 40 L/min.

Figure 3. Effects of plasma gas flow rate on mean particle diameter and BET surface area.

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