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Synthesis of Nano Composite Particles by a Transferred Direct Current Thermal Plasma: Effects of Plasma Input Current†

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Effects of input current on the characteristics of nano composite particles synthesized from a ZrVFe alloy ingot by transferred DC thermal plasma were investigated. The characteristics of the synthesized powder were analyzed by FE-SEM/EDX, XRD, PSA and BET. As the plasma input current increased from 120 to 160 A, the average particle size decreased from 83.2 to 47.8 nm, the particle size distribution became narrower and the surface area increased from 100.9 to 211.2 m^2/g and the particle crystallinity was improved. In all cases, the generated composite particles were spherical.

Key Words: Thermal plasma, ZrVFe alloy, Nano Composite particle, Getter, Plasma display panel.

INTRODUCTION

Over the last decade, there has been an increasing interest in nano particles due to their attractive characteristics such as high surface-to-volume ratios, unusual mechanical and chemical properties[1]. Nano particles can be produced by various methods, such as sol-gel[2], spray pyrolysis[3], infrared heating[4] freeze-drying[5], laser ablation[6], wire explosion[7] and thermal plasma[8]. 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 zirconium. A potential application of the zirconium contained nano materials is the decontamination of the electric discharge gas used in plasma display panel (PDP).

Plasma display panel is considered as the most promising candidate for large area display among various flat panel displays due to its manufacturing process appropriate for a large displaying area, high speed addressing ability and good display quality[9]. 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 plasma display panel. 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 life time with good display quality, it is necessary to install getter materials in the plasma display panel cell for the continuous elimination of contaminants, such as O_2, H_2, CO, *etc*., emitted from protective layer, barrier rib and fluorescent substances[10,11]. Installation of getter materials, such as ZrVFe alloy powder, in the plasma display panel cell is a potential solution to overcome such problems. In fact, ZrVFe getter materials are currently utilized in various applications[12,13] 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 plasma display panel cell. Hence it is imperative to produce the getter material with the particle size in the range of nanometers. In this regard, we previously attempted to prepare nano composite particles from a bulk ZrVFe alloy ingot by a transferred direct current (DC) thermal plasma[14]. In this study, we investigate the effects of the plasma input current on the characteristics of the produced nano particles.

EXPERIMENTAL

A schematic diagram of the thermal plasma system employed to synthesize nano composite particles is shown in Fig. 1. The system consists of five sections: a direct current plasma torch generating plasma jet from an arc, a reaction chamber providing a extremely high temperature environment

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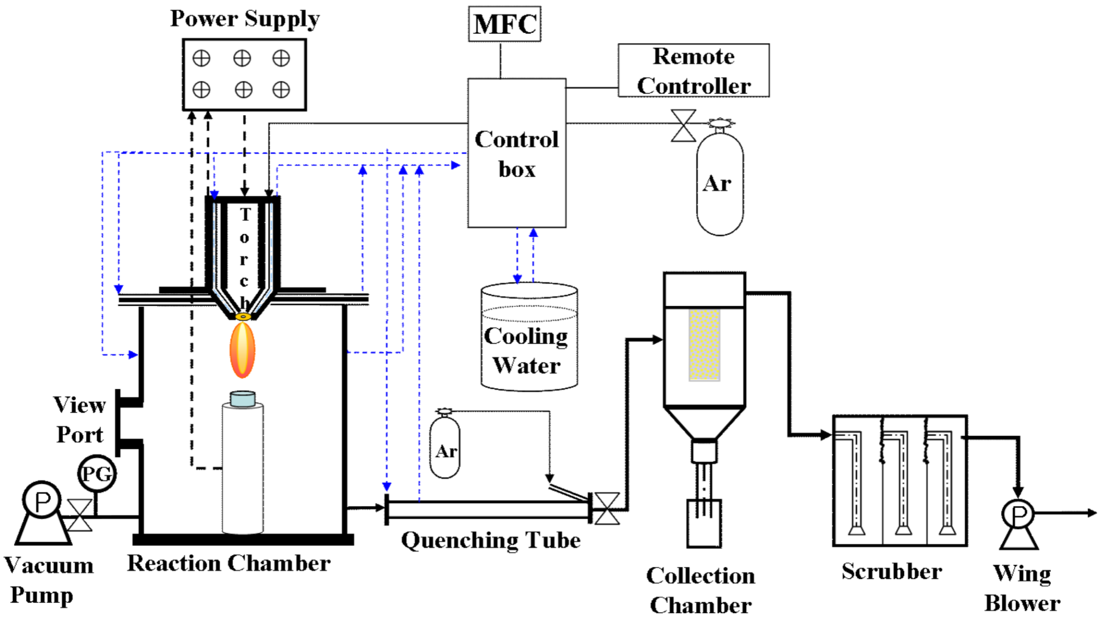


Fig. 1. Schematic diagram of the thermal plasma system

for the formation of nuclei/clusters as well as the growth of nano particles, a quenching tube in which the synthesized nano particles are quickly cooled down to be prevented from 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 the 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 flow rate of the plasma gas (pure argon) was fixed at 20 L/min while the input current to generate thermal plasma was varied 120 to 160 A. The characteristics of the nano particles were analyzed by FE-SEM (FEI, Quenta 200), EDS (Horiba), XRD (Rigaku, MAX-2500V), PSA (Malvern, Masrersizers & Zetasizers) and BET analyzer (Micrometrics, ASAP2020).

RESULTS AND DISCUSSION

The SEM images and corresponding particle size distributions of the nano composite particles synthesized by the transferred plasma with the various plasma input currents are in Fig. 2. From the SEM images, the particles were found to be spherical although severely agglomerated. As the plasma input current increases, the maximum plasma temperature increases, in turn generating much more abundant nuclei. Since the reaction chamber was cooled with the cold water, the temperature gradient become stiffer and the quenching rate increases, limiting the particle growth. As a consequence, the particle size decreased as the plasma input current increased,

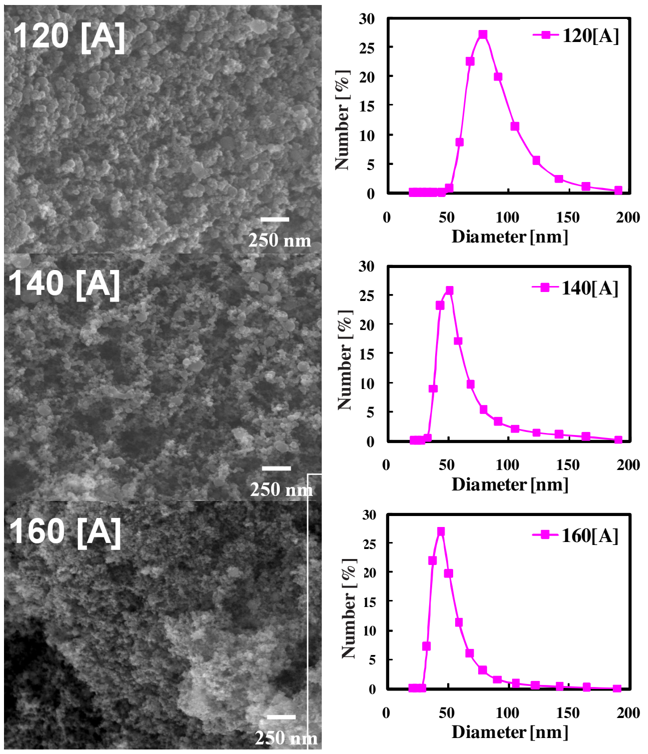


Fig. 2. SEM images and size distributions of the particles produced by thermal plasma

which also confirmed by the PSA measurement. The average particle size decreased from 83.2 to 47.8 nm as the plasma input current increased from 120 to 160 A. Furthermore the plasma input current had a profound effect on the particle size distribution. The particle size distribution became narrower implying that the particle size become more uniform as the plasma input current increased. The surface area measured by the BET correspondingly increased from 100.9 to 211.2 m^2/g as the plasma input current increased from 120 to 160 A, as shown in Fig. 3.

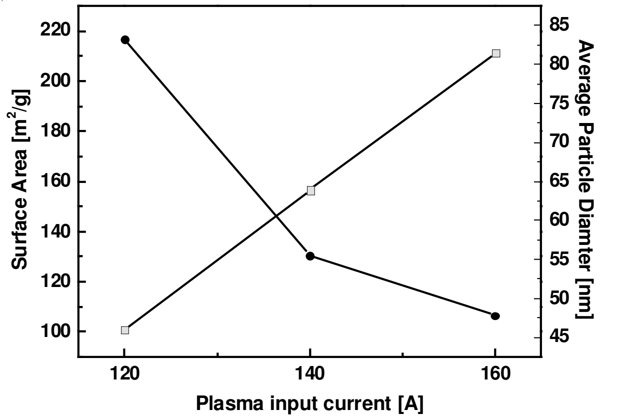


Fig. 3. Effects of the plasma input current on the surface area (measured by BET)

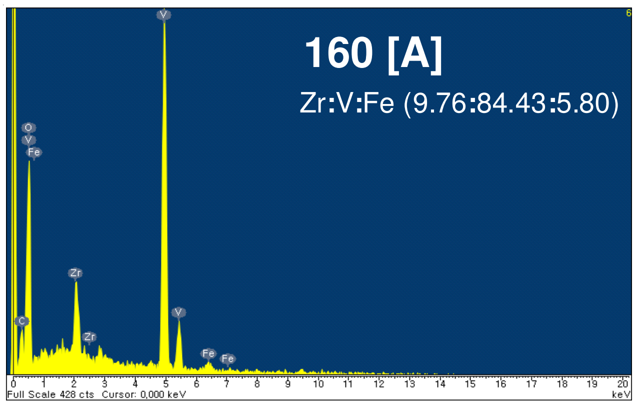


Fig. 4. Effects of the plasma input current on the XRD pattern

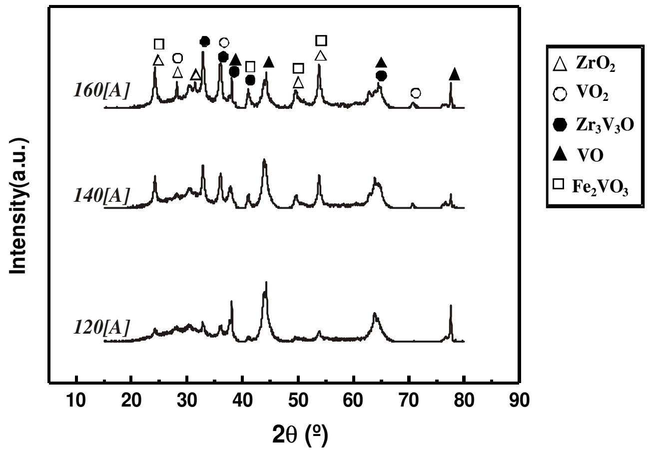
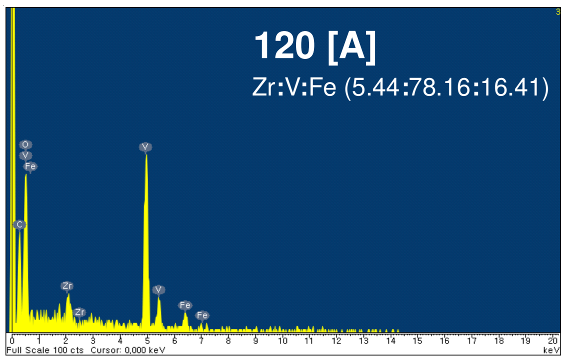
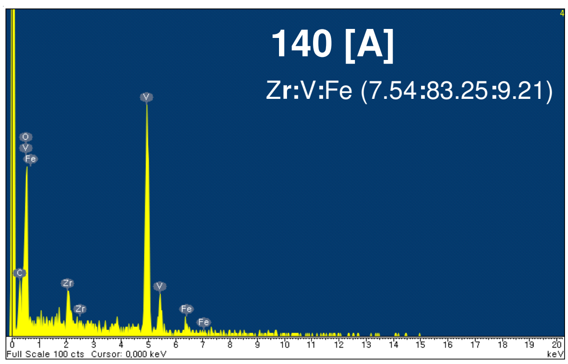


Fig. 5. Effects of the plasma input current on the XRD pattern

The electron diffraction analyses of the particles synthesized with various plasma input currents are shown in Fig. 4. Although constituting 57 wt % of the raw material, Zr was found to be less than 10 wt % of the synthesized nano composite particles because Zr has much higher boiling point than V and Fe. Nonetheless the Zr content in the synthesized nano composite particles increased as the plasma input current increased simply because the maximum temperature of the plasma flame contacting the raw material increased. The oxygen peak was also observed implying that the oxide particles were formed. It was believed that the surface of the synthesized particles was oxidized during sampling although they were stabilized for a long time before taken out to atmosphere for sampling. The XRD patterns of the particles synthesized with various plasma input currents are shown in Fig. 5. As expected from the EDS analyses, various oxide particles were formed. As the plasma input current increased, the intensity of the Zr_3V_3O peak increased significantly and each peak became sharper, implying that the particle crystallinity improved.





Conclusion

Spherical nano composite particles were synthesized by a transferred direct current plasma from a ZrVFe alloy ingot prepared by the arc melting method. The effects of the plasma input current on the characteristics of the synthesized nano composite particles were investigated. When the plasma input current increased, the maximum plasma temperature became higher, generating much more abundant nuclei and the temperature gradient became stiffer, limiting the particle growth. As the plasma input current increased, consequently, the particle size decreased, the particle size distribution became narrower, the content of Zr in the synthesized nano composite particles increased and the particle crystallinity improved.

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