JOURNAL OF MULTIFUNCTIONAL MATERIALS & PHOTOSCIENCE
9(1), June 2018, pp. 33-38

Synthesis of Fe-Ti Composite Nano Particles by a Transferred DC Thermal Plasma

Seung Kyu Park and Heon Chang Kim*

Department of Chemical Engineering, Hoseo University, Asan, Chungnam 336-795, Korea

Abstract: Fe-Ti composite nano particles were synthesized by a transferred DC thermal plasma, and the effects of plasma input current on the characteristics of the synthesized composite particles synthesized were investigated. The characteristics of the particles were analyzed by HR-TEM, FE-SEM, EDX and PSA. As the plasma input current increased from 240 A to 280 A, the average particle size decreased from 52 nm to 38 nm. In all cases, the generated composite particles were spherical, and exhibited very narrow particle size distributions.

Introduction

Over the several decades, 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, especially for materials with extremely high evaporation temperature such as zirconium. A potential application of the zirconium containing nano materials is the decontamination of the electric discharge gas used in plasma display panel (PDP).

PDP 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 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 life time with good display quality, it is necessary to install getter materials in

* Corresponding author*: heonchan@hoseo.edu*

the PDP 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 PDP cell is a potential solution to overcome such problems[12-13]. However the particle size of the currently available commercial getters is in the range of micrometers, limiting their applicability to the PDP cell. Hence it is imperative to produce the getter material with the particle size in the range of nanometers. In this regard, we previously utilized a transferred direct current (DC) thermal plasma to prepare nano composite particles from a bulk ZrVFe alloy ingot[14] and investigate the effects of the plasma input current on the characteristics of the produced nano particles[15]. In this study, we extend our work to synthesis of Fe-Ti composite nano particles as a potential gettering material.

Experimental

The thermal plasma system employed in this study is same as one that utilized in our previous work[14, 15], and its schematic diagram is shown in Fig. 1. The system consists of five sections: a DC plasma torch generating plasma jet from an arc, a reaction chamber providing a extremely high temperature environment 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 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 iron (Fe) and titanium (Ti). They were melted together by arc in a vacuum chamber, forming a bulk FeTi alloy ingot to be used as a raw material. The molar ratio of the constituent elements, Fe:Ti, in the raw material was 20:80.

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, FeTi alloy ingot. The ingot was placed 2 cm below the plasma nozzle, the flow rate of the plasma gas (pure argon) was fixed at 10 L/min while the input current to generate thermal plasma was varied 240 A from to 280 A by 20 A. The characteristics of the nano particles were analyzed by HR-TEM(Hitachi, H-9500), FE-SEM (FEI, Quenta 200), EDS (Horiba) and PSA (Malvern, Masrersizers & Zetasizers).

Results and Discussion

The TEM image of the typical particles produced by thermal plasma is shown in Fig. 2 with component mapping data. The particles are mostly spherical shape, and form chain like agglomerates because of their magnetic properties. From the component mapping data, it is proved that our thermal plasma system can synthesize Fe-Ti composite particles not mixture of single component particles. Furthermore, the scanning TEM analysis across the largest particle, around 80 nm, reveals that the most element is Fe although the raw material was comprised of 20% Fe and 80% Ti, mainly due to large difference in their evaporation temperatures. Furthermore a thin oxide layer around the particle was also observed, and corresponding oxygen signals continued to appear across the parcel.

The SEM images and corresponding particle size distributions of the Fe-Ti composite particles synthesized by the transferred plasma with the various plasma input currents are in Fig 3. From the SEM images, the particles were found to be spherical although severely agglomerated. As conjectured in our previous work[14, 15], the maximum plasma

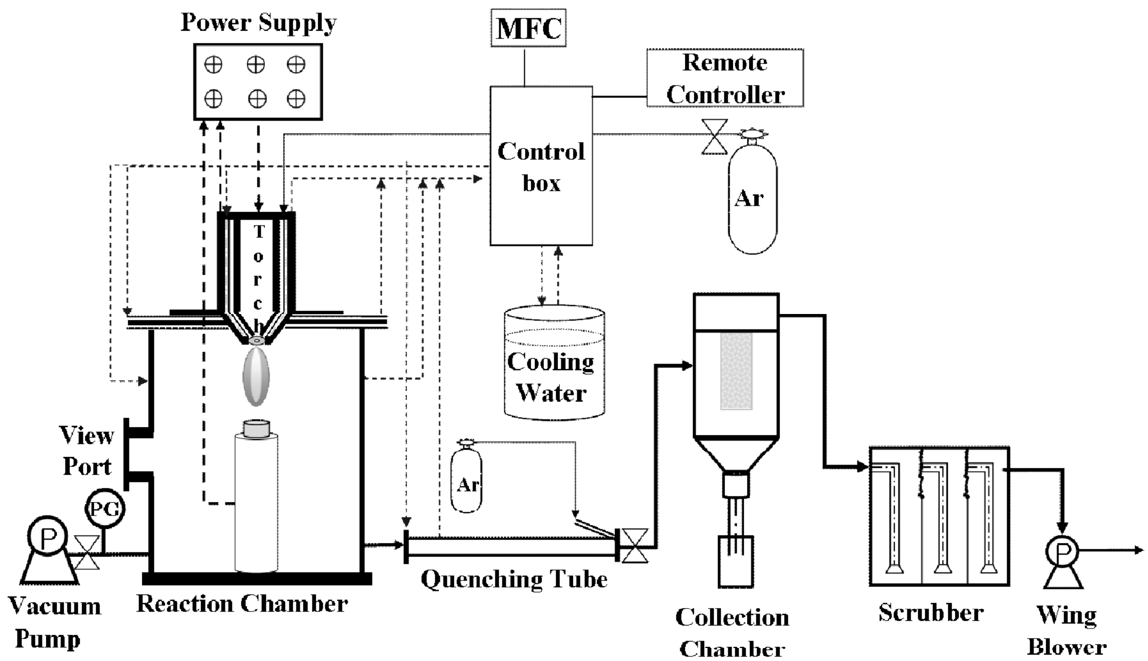

Figure 1: Schematic diagram of the thermal plasma system

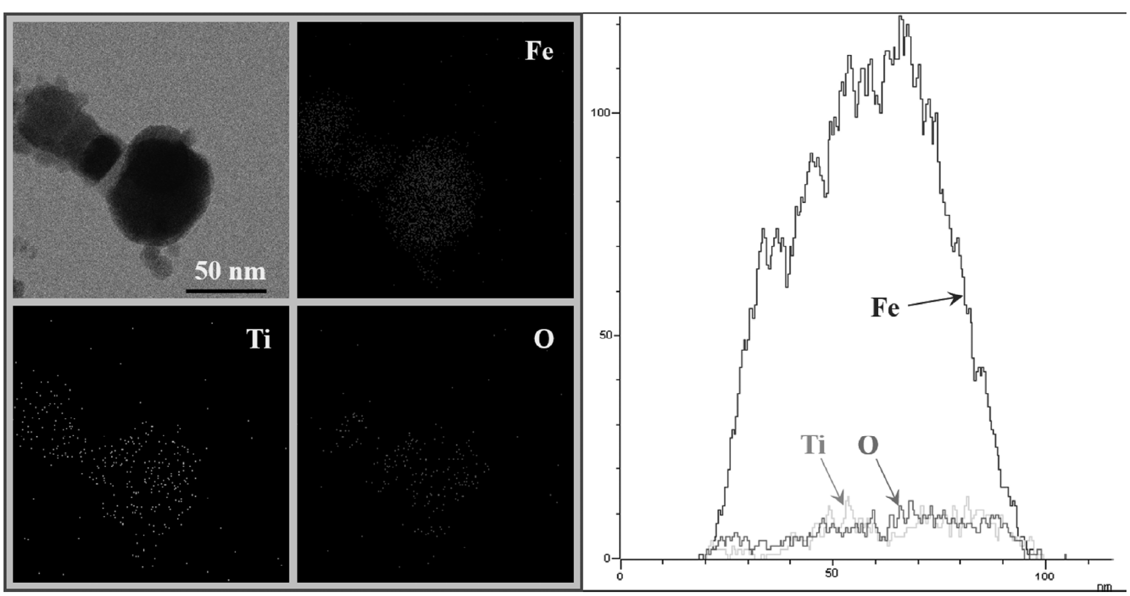

Figure 2: TEM images and component mapping of the typical Fe-Ti particles produced by thermal plasma

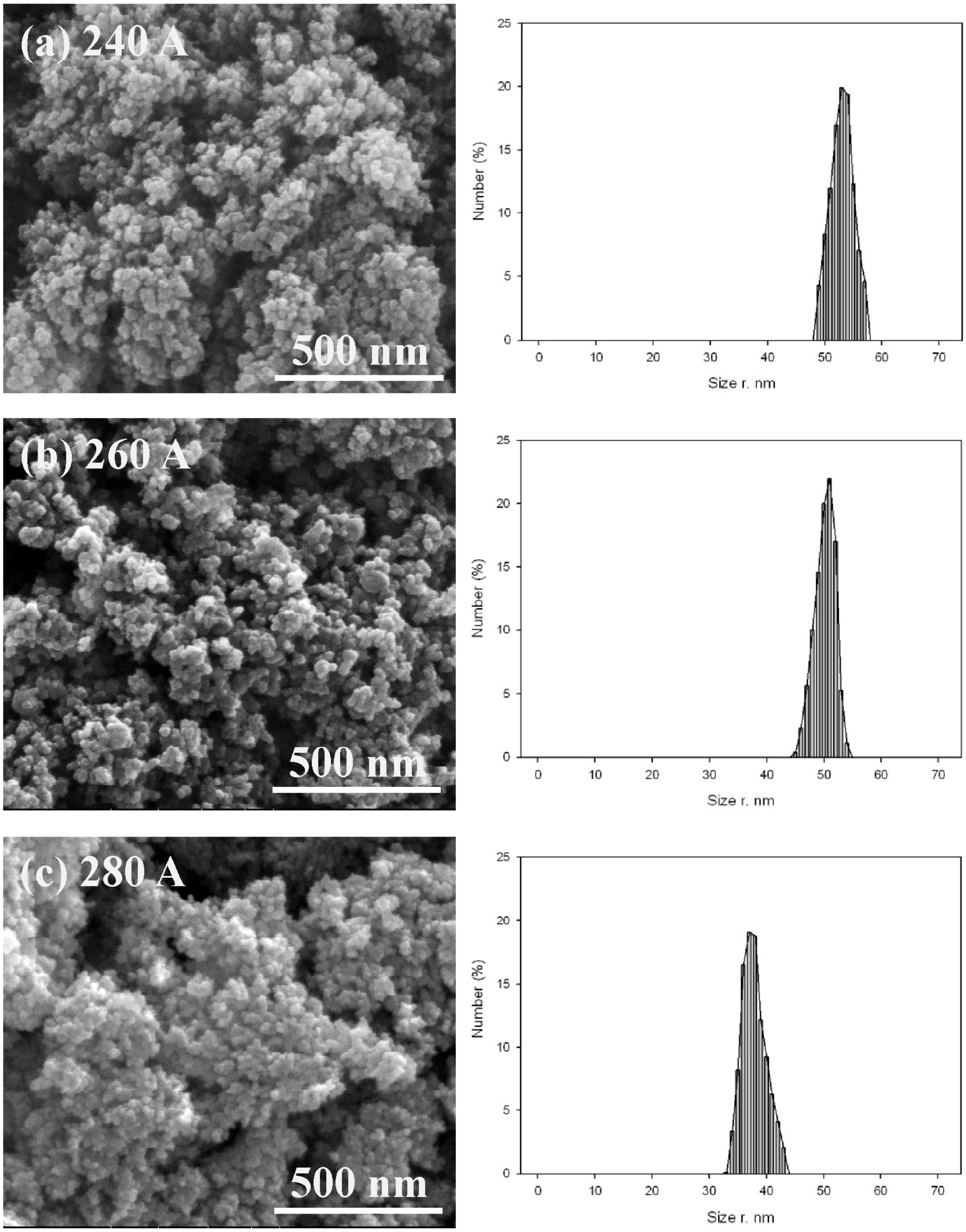

Figure 3: SEM images and size distributions of the Fe-Ti composite particles produced with various plasma input currents

temperature increases as the plasma input current 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, which also confirmed by the PSA measurement. The average particle size decreased from 83.2 nm to 47.8 nm as the plasma input current increased from 240 A to 280 A, as shown in Fig. 4. In the EDS analyses, the oxygen peak for the thin oxide layer found in Fig. 2 was also observed in all cases, implying that the oxide particles were formed. It was believed, however that the surface of the synthesized particles was oxidized during sampling although they were stabilized for a long time before taken out to atmosphere.

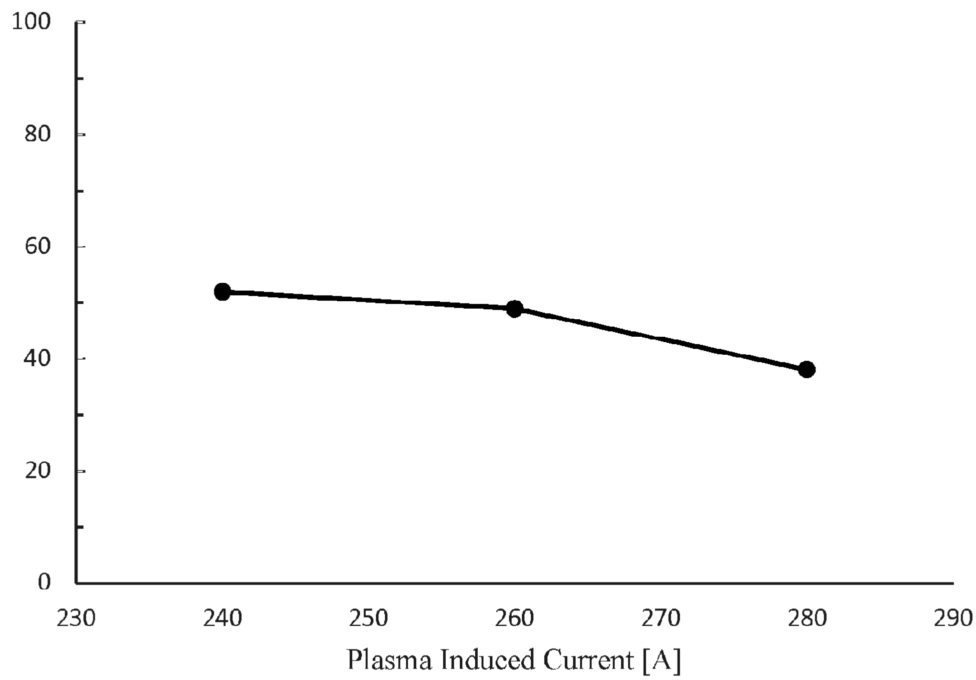

Figure 4: Effects of the plasma input current on the average particle size of the synthesized Fe-Ti composite particles.

Conclusions

In this work, we synthesized Fe-Ti composite nano particles, by a transferred DC thermal plasma, as a potential material applicable to the gettering process, and investigated the effects of plasma input current on the characteristics of the synthesized composite particles. The results showed that our thermal plasma system could produce spherical nano particles comprised of Fe and Ti with very narrow particle size distribution. The average particle size decreased from 52 nm to 38 nm as the plasma input current increased from 240 A to 280 A. In our future investigation, sorption capacities of the Fe-Ti composite nano particles produced in this experiment will be characterized for the application to the gettering process.

References

[1] S. Hoeppener, R. Maoz, S. R. Cohen, L. F. Chi, H. Fuchs and J. Sagiv, *Adv. Mater.*, **14**, 1036 (2002).

[2] W. Liu, D. Wu and J. Yang, *Int. J. Mater. Prod. Tech.*, **37**, 297 (2010).

[3] H. Y. Koo, J. H. Yi and Y. C. Kang, *J. Alloys Compd.*, **489**, 456 (2010).

[4] S. Oh and S. Lee, *J. Nanosci. Nanotechnol.*, **10**, 366 (2010).

[5] X. Xi, X. Xu, Z. Nie, S. He, W. Wang, J. Yi and Z. Tieyong, *Int. J. Refract. Met. Hard Mater.*, **28**, 301 (2010).

[6] C. A. Crouse, E. Shin, P. T. Murray and J. E. Spowart, *Mater. Lett.*, **64**, 271 (2010).

[7] L. H. Bac, Y. S. Kwon, J. S. Kim, Y. I. Lee, D. W. Lee and J. C. Kim, *Mater. Res. Bull.*, **45**, 352 (2010).

[8] A. J. Song, M. Z. Ma, W. G. Zhang, H. T. Zong, S. X. Liang, Q. H. Hao, R. Z. Zhou, Q. Jing and R. P. Liu, *Mater. Lett.*, **64**, 1229 (2010).

[9] W. B. Choi, B. K. Ju, Y. H. Lee, S. J. Jeong, N. Y. LEE, M. Y. Sung and M. H. Oh, *J. Electrochem. Soc.*, **146**, 400 (1999).

[10] G. Chakhovskoi, C. E. Hunt and M. E. Malinowski, *Displays*, **19**, 179 (1999).

[11] R. Chalamala, D. Uebelhoer and K. A. Dean, *J. Vac. Sci. Tech. A*, **18,** 343 (2000).

[12] D. Petti, M. Cantoni, M. Leone, R. Bertacco and E. Rizzi, *Appl. Surf. Sci.*, **256**, 6291 (2010).

[13] P. Roupcová and O. Schneeweiss, *J. Alloys Compd.*, **492**, 160 (2010).

[14] M. H. Jeong and H. C. Kim, *Appl. Chem. Eng.*, **21**, 676 (2010).

[15] S. K. Park, K. W. Park and H. C. Kim, *Asian J. Chem.*, **24**, 4141 (2012).