Middle-East Journal of Scientific Research 15 (2): 287-290, 2013
ISSN 1990-9233

DOI: 10.5829/idosi.mejsr.2013.15.2.204

Carbon Coating on Graphite Substrates Using Centrifugal Deposition Process: Effect of Centrifugal Rotation Speed and Heat Treatment on Coating Quality

[1]Reza Ahmadi, [2]Naser Ehsani and [2]Ali Khalife Soltani

[1]Research Centre for Sciences and Technology in Medicine,
Tehran University of Medical Sciences, Tehran, Iran
[2]MAU University, Department of Materials Science and Engineering, Tehran, Iran

Abstract: In the present study, electrospinning synthesized Graphite samples with high porosity surface layer have been centrifugally coated using Poly methyl methacrylate as binder and cyclohexane as suspension base liquid phase. After a suitable heat treatment and resin removal under a compact force, a relatively stick adhesive layer formed. Effect of centrifugal rotation speed and heat treatment procedure were investigated on coating thickness and quality using Scanning Electron Microscopy and Thermal Gravimetric Analysis techniques. According to achieved results, the best morphology and highest coating density was obtained at centrifugal rotation speed = 5000 rpm, resin to particles weight ratio of 0.05 and a two-stage heat treatment at 100 and 300°C. At these conditions a 44 µm relatively high density coating layer of carbon nanoparticles was formed on the initial surface of samples.

Key words:

INTRODUCTION

Carbon coating is a conventional procedure in various fabrication techniques such as synthesizing high temperature resistant layers and functional graded materials (FGMs). These materials are suitable for high temperature applications; however carbon oxidizing is a common problem in these applications. So, some supplementary procedures are necessary such as SiC or B_4C layer formation via suitable heat treatment of successive coated Si (or B) and C layers to reduce surface oxidation of carbon layers. Among various conventional Carbon coating procedures such as electrophoretic deposition [1-4], centrifugal coating [5-8], settling [9], screen printing [10, 11] and spraying [12], electrophoretic coating is used frequently due to its advantages. However, coating density is relatively low in this method. Besides, a very smooth and high density initial surface is required for this method. As many industrial samples have a relatively rough surface, this technique cannot be used directly and some pre-coating methods shall be used such as centrifugal coating.

In this work, electrospinning synthesized Graphite samples (ESGSs), with high porosity rough and low density surface layer, have been coated using centrifugal forces. This method has been employed for phosphor deposition before [5], but has not been used for carbon coating yet. In this method, a stable suspension containing carbon nanoparticles or nanotubes and a suitable polymeric resin, Poly methyl methacrylate (PMMA), in cyclohexane solution was centrifuged in a tube; meanwhile the graphite sample was located and fixed in one side of centrifuge tube. So, a smooth controllable high density carbon layer formed on the external surface of Graphite sample. After a suitable heat treatment and resin removal under a compact force, a relatively stick adhesive layer formed. Effect of centrifugal rotation speed (CRS) and heat treatment procedure were investigated on coating thickness and quality.

Experimental Procedure

Sample Preparation: ESGSs with 1 cm^2 surface were inserted and fixed in centrifuge tubes containing 3 cc suspensions of carbon nanoparticles and PMMA in

Corresponding Author: Reza Ahmadi, Research Centre for Sciences and Technology in Medicine, Tehran University of Medical Sciences, Tehran, Iran

Table 1: Process conditions of centrifugal coating method and coating layer properties[1]

Sample	CRS[2] (rpm)	PMMA to Carbon weight ratio	Heat Treatment	Coating Weight (mg) [3]	Coating Thickness (μm) [4]	PMMA Weight%	Water%	Density (gr/cm^3)	%Relative density [5]
A1	2000	0.05	Two-stage [6]	80.1	468	1.6	3.2	1.63	71.9
A2	3000	0.05	Two-stage	49.5	240	1.8	3.3	1.96	86.5
A3	4000	0.05	Two-stage	11.7	54	1.53	3.5	2.05	90.9
A4	5000	0.05	Two-stage	9.8	44	1.51	3.3	2.12	93.7
A5	5000	0.05	One-stage [7]	-	-	-	-	-	-
A6	5000	0.05	One-stage [8]	-	-	-	-	-	-

1- Because of undesired SEM results and non-homogeneous coating layer thickness, thickness measurement and density calculations did not perform for samples A5 and A6.
2- Centrifugal rotation speed
3- Coating weight before heat treatment
4- From SEM images
5- Respect to pure Carbon bulk
6- 30 min at 100°C and 30 min at 300°C
7- 60 min at 300°C
8- 60 min at 100°C

cyclohexane. Suspension concentration was equal to 50 mgC/cc and PMMA /carbon weigh ratio was equal to 0.05. These suspensions were centrifuged 3 min at 2000 rpm and then the graphite samples were died in 300°C oven for 60 minutes for drying and resin removal (PMMA boiling point equals to 200°C). To investigate effect of process conditions on coating thickness and quality the above-mentioned procedure was repeated in centrifugal rotation speeds 3000, 4000 and 5000 rpm and a two-stage heat treatment procedure (30 min at 100°C and 30 min at 300°C). The detailed process conditions are listed in Table 1.

Characterization: Scanning Electron Microscopy (SEM) was performed using Philips XL 30 instrument to investigate coating thickness and morphology. Thermal Gravimetric Analysis (TGA) procedure was employed using a controlled-atmosphere oven and a digital weigher (accuracy 0.0001g) to track samples' weight changes.

RESULTS AND DISCUSSION

SEM images of sample A0, the initial uncoated electrospinning synthesized sample, are presented in Figures 1-a to 1-c. Some 5 μm width carbon fibers are seen in these images beside 50-150 nm carbon particles. This unpacked low density layer structure is not suitable for conventional electrophoretic coating procedures.

(a) (b) (c)

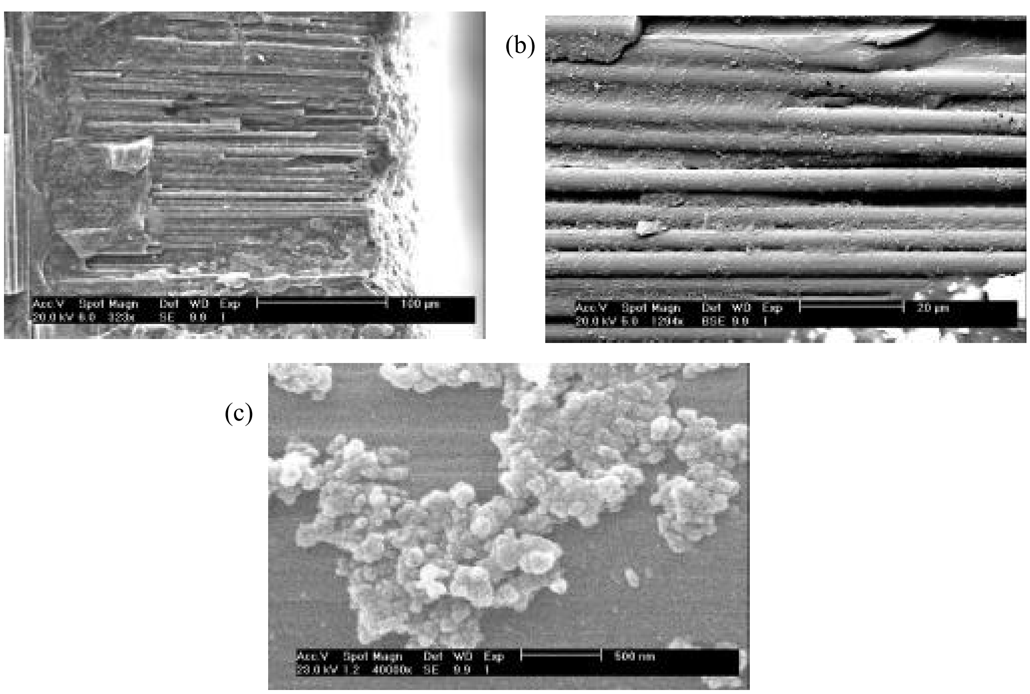

Fig. 1: SEM image of sample A0, initial electrospinning synthesized sample, a) Scale bar = 100 μm, b) Scale bar = 20 μm and c) Scale bar = 500 nm.

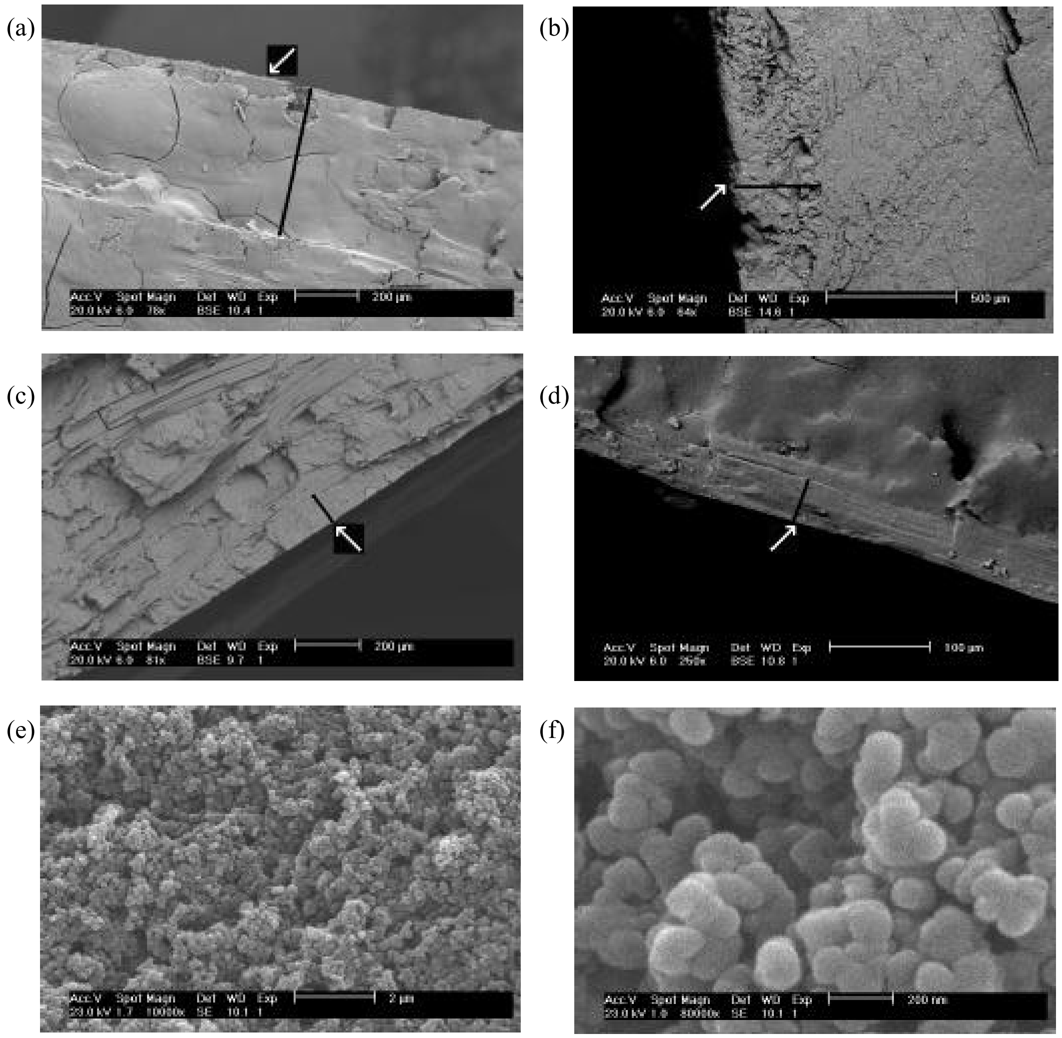

Fig. 2: SEM image of sample a) A1: centrifugal coated at 2000 rpm, b) A2: centrifugal coated at 3000 rpm, c) A3: centrifugal coated at 4000 rpm, d) A4: centrifugal coated at 5000 rpm, Scale bar = 100 μm, e) A4: centrifugal coated at 5000 rpm, Scale bar = 2 μm and f) A4: centrifugal coated at 5000 rpm, Scale bar = 200 nm (Table 1).

SEM images of samples A1 to A4 centrifugally coated at CRSs 2000 to 5000 rpm are presented in figures 2-a to 2-f. As seen in these images, coating thickness is decreased by increasing CRS. On the other hand, coating layer density calculated by measurement of samples' weight increase after centrifugal carbon coating is increased by increasing CRS (Table 1). This can be explained by this fact that by increasing CRS from 2000 to 5000 rpm, centrifugal force increased and thus a rather high density coating will form. A rather porous low density coating is formed in low CRSs (Samples A1 and A2). On the other hand, in high amounts of CRS, a large fraction of carbon particles will tend to scatter due to the suspension severe agitation and settle on the centrifuge machine tube wall instead of samples' surface and thus, the coating layer weight will be less than low CRSs (Table 1).

Figure 3 presents effect of heat treatment procedure on coating quality. Using one-stage heat treatment at 300°C instead of two-stage heat treatment led to formation of surface cracks and porosities due to quick removal of water and PMMA simultaneously from samples' surface (Figure 3-a, sample A5). These cracks are not seen in samples A1 to A4 with two-stage heat treatment. Some residual PMMA agglomerates are seen in regions A and B of Fig. 3-a. PMMA agglomerates' structure is shown in SEM images 3-b and 3-c, including some nanometric PMMA particles. On the other hand, heating sample up to 100°C remains more residual PMMA agglomerates in coating layer without any surface cracks (Figure 3-d, sample A6).

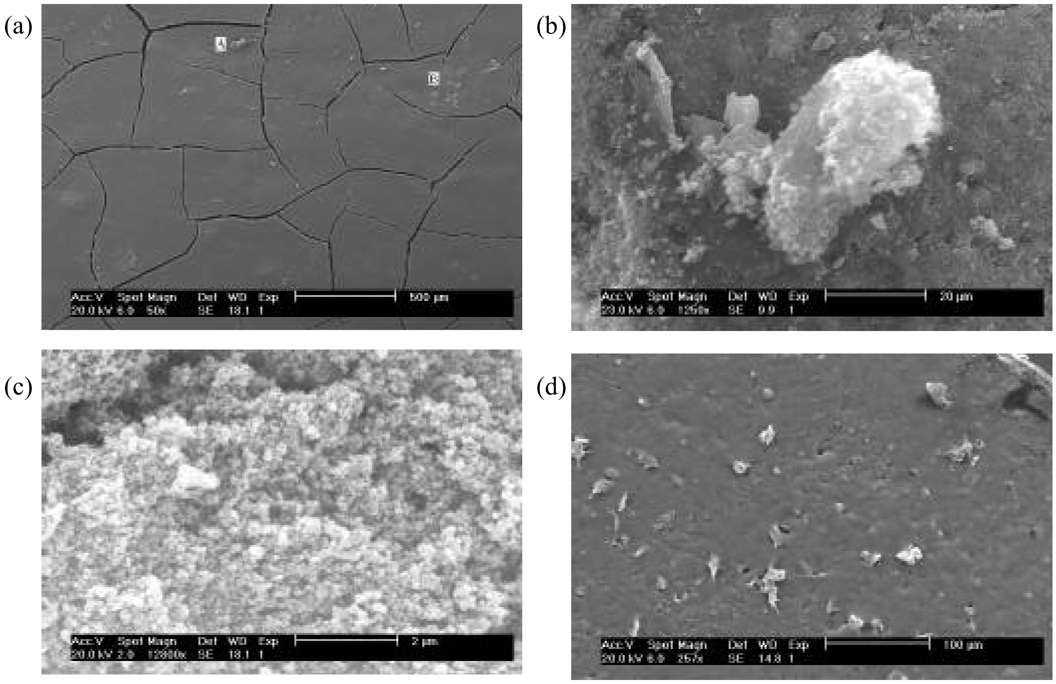

Fig. 3: SEM image of: a-c) sample A5 in three magnifications and d) sample A6 (Table 1).

Samples' coating density was calculated using SEM and TGA results. As the samples' initial surface area equals to 1 cm^2, coating volume can be calculated easily from coating thickness (Table 1). Thus, coating density can be determined by dividing final coating weight (after heat treatment) to this amount. These results are listed in table 1 for samples A1 to A4.

CONCLUSION

Centrifugal coating process is described and employed in this study as an effective technique to produce high density smooth Carbon coating layer on the surface of electrospinning synthesized samples. The optimum conditions led to a 44 µm Carbon coating layer with relative density of %93.7. Increasing centrifugal rotation speed and using two-stage heat treatment led to decreasing coating layer thickness and increasing its density. It seems that this method can be used for similar applications that an initial rough surface shall be coated to produce a smooth coating layer.

REFERENCES

1. Boccaccini, A. and I. Zhitomirsky, 2002. Current Opinion in Solid and Materials Science, 6: 251.
2. Somiya, S., 2003. Handbook of Advanced Ceramics, Elsevier Academic Press.
3. Boccaccini, A., J. Cho, T. Subhani, C. Kaya and F. Kaya, 2010. Journal of the European Ceramic Society, 30: 1115.
4. Maria, J., C. Alberto, C. Francisco, Q. Nancy and R. Aldo, 2010. International Journal of Applied Ceramic Technology, 7: 30.
5. Ozols, A., M. Barreiro, E. Forlerer and H.R. Sirkin, 2006. Surface and Coating Technology, 200: 5884.
6. Tong, H., L.B. Kong and C.M. Wang, 2006. Thin Solid Films 496: 360.
7. Atobe, M., A. Murotani, S. Hitose, Y. Suda, M. Sekido, T. Fuchigami, A. Chowdhury and T. Nonaka, 2004. Electrochimica Acta, 50: 977.
8. Liedekerke, P.V., E. Tijskens, E. Dintwa, F. Rioual, J. Vangeyte and H. Ramon, 2009. Powder Technology, 190: 348.
9. Technical Information Booklet CM-9045, Method of Settling Phosphor Slides, GTE Sylvania, 3/82.
10. Lee, T.M., Y.J. Choi, S.Y. Nam, C.W. You, D.Y. Na and H.C. Choi, 2008. Thin Solid Films 516: 7875.
11. Erath, D., A. Filipoviæ, M. Retzlaff, A.K. Goetz, F. Clement, D. Biro and R. Preu, 2010. Solar Energy Materials and Solar Cells, 94: 57.
12. Wu, J., J. Yang, H. Fang, S. Yoon and C. Lee, 2006. Applied Surface Science 252: 7809.