

Nanostructuring effect of multi-walled carbon nanotubes on electrochemical properties of carbon foam as constructive electrode for lead acid battery

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Abstract In the present study, nanostructuring effect of multi-walled carbon nanotubes (MWCNTs) on electrochemical properties of coal tar pitch (CTP) based carbon foam (CFoam) was investigated. The different weight fractions of MWCNTs were mixed with CTP and foam was developed from the mixture of CTP and MWCNTs by sacrificial template technique and heat treated at 1,400 and 2,500 °C in inert atmosphere. These foams were characterized by scanning electron microscopy, X-ray diffraction, and potentiostat PARSTAT for cyclic voltammetry. It was observed that, bulk density of CFoam increases with increasing MWCNTs content and decreases after certain amount. The MWCNTs influence the morphology of CFoam and increase the width of ligaments as well as surface area. During the heat treatment, stresses exerting at MWCNTs/carbon interface accelerate ordering of the graphene layer which have positive effect on the electrochemical properties of CFoam. The current density increases from 475 to 675 mA/cm² of 1,400 °C heat treated and 95 to 210 mA/cm² of 2,500 °C heat-treated CFoam with 1 wt% MWCNTs. The specific capacitance was decreases with increasing the scan rate from 100 to 1,000 mV/s. In case of 1 % MWCNTs content CFoam the specific capacitance at the scan rate 100 mV/s was increased from 850 to 1,250 μF/cm² and 48 to 340 μF/cm² of CFoam heat treated at 1,400 °C and 2,500 °C respectively. Thus, the higher value surface area and current density of MWCNTs-incorporated CFoam heat treated to 1,400 °C can be suitable for lead acid battery electrode with improved charging capability.

Keywords Carbon foam · Multi-walled carbon nanotubes · Surface area · Electrical conductivity · Electrochemical properties

Introduction

Secondary batteries are next generation energy storage alternatives for wide variety of portables personal computers, cellular phones, digital cameras, items of military electronic equipment, etc. Battery technology has emerged over the past few years as one of the most advanced power sources meeting the requirements of portable, high energy density, specific capacity, high working potential, good cycling behavior, power environment friendly, etc. (Broussely 1999; Tamura and Horiba 1999; Tanake et al. 2001; Marsh et al. 2001). The most efficient hybrid vehicles lack power and models that accelerate quickly do so with the assistance of large internal combustion engines. This significantly degrades their fuel efficiency, barely making the extra cost worthwhile in terms of fuel efficiency. While batteries in these vehicles are capable of storing large quantities of energy, they cannot be charged or discharged quickly. This lack of power density requires the battery packs to be oversized, resulting in increased vehicle weight and reduced efficiency. As with poor discharge rate, battery charging is limited by the same kinetics, thus reducing efficiency gains through full regenerative braking. Moreover, the biggest drawback of lead acid batteries is the heavy weight due to the use of lead as a current collector. Lead grid constitutes 30–40 % of the battery weight. To reduce the weight of lead acid batteries, light weight electrically conducting materials are to be used as a potential substitute for it (Czerwinski and Zelazowska 1996; Das and Mondal 2000; Gyenge et al.

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2002). Carbon materials have been playing a significant role in the development of alternative clean and sustainable energy technologies. Various carbon materials such as natural graphite, hard carbon from various polymer precursor, petroleum coke, and mesocarbon microbeads (MCMB) and light weight carbon foam (CFoam) have been utilized for improving battery performance (Flandrois and Simon 1999; Wu et al. 1998; Chang et al. 1998; Imanishi et al. 2008; Yang et al. 2005).

The carbon foam (CFoam), a lightweight material, three-dimensional network with open cell structure, large specific surface area, thermally and electrically conductive along with good corrosion resistance, is a promising candidate for current collector for lead acid batteries (Jang et al. 2006; Chen et al. 2008). The electrical conductivity of CFoam derived from different organic and inorganic precursors can be tailored by controlling processing parameters. Initially, CFoam was prepared from thermosetting polymeric material by heat treatment under controlled atmosphere (Cowland and Lewis 1967). Later on, coal tar and petroleum pitches were used for the development of CFoam (Chen et al. 2006). To make highly crystalline CFoam of high electrical and thermal conductivity, generally mesophase pitch is used as the starting material (Klett et al. 2000, 2004) and it is prepared by high temperature and pressure foaming process. It is an expensive process, therefore in the present study, using the simple sacrificial template (Chen et al. 2007) technique CFoam is developed from the coal tar pitch (CTP) (Yadav et al. 2011). But CTP-based CFoam does not have high electrical conductivity and hence might have poor electrochemical properties.

To overcome the electrochemical properties of pitch-based CFoam, new approach of nanostructuring is adopted to improve the electrochemical properties of CFoam by taking the advantage of outstanding properties micro- and nano-forms of carbon, e.g., CTP and carbon nanotubes (CNTs). Nanostructured materials including metal nanoparticles, nanowires and carbon nanotubes have been demonstrated for high energy/power density with significantly improved durability (Arico et al. 2005; Kumar et al. 2013). Since their first observation by Iijima (1991) CNTs attracted considerable attention because of their excellent electrical conductivity (10^3 – 10^6 S/cm), ultra-high strength, high surface area and large aspect ratio that made them an ideal reinforcing additive for development of high-performance advanced materials (Moniruzzaman and Winey 2006; Spitalsky et al. 2010; Ajayan et al. 1994). A combination of extraordinary electrical, thermal and mechanical properties makes CNTs not only attractive materials in nanoelectronic devices, but also excellent building blocks for assembling new hybrid materials for widespread applications, particularly energy storage devices (Snow et al. 2005).

In the present investigation, an effort has been made to develop MWCNTs-incorporated CFoam with improved electrochemical properties, so that it can be a constructive electrode material in lead acid battery with enhanced charging capacity, reduced weight of battery and increased life time. The effect of MWCNTs is ascertained by characterizing CFoam by scanning electron microscopy, surface area by Autosorb 3B, cyclic voltammetry (CV), X-ray diffraction.

Experimental

Development of CFoam

The CFoam was developed by sacrificial template technique from modified CTP. The modified pitch was synthesized from CTP by heat treating at 400 °C for 20 h. MWCNTs were procured from Nanocyl, Belgium. They were dispersed in an organic solvent ethanol by magnetic stirring for 10–15 h and mixed with the modified CTP in different weight fractions (0, 0.5, 1.0 and 2.0 wt%). The water slurry of MWCNTs mixed modified pitch with 3 % polyvinyl alcohol (PVA) was impregnated into a polyurethane foam (density 0.030 g/cc and average pore size 0.45 mm) template under vacuum. MWCNTs mixed modified CTP impregnated polyurethane foam was converted into CFoam by several heat treatments in air as well as in an inert atmosphere up to 2,500 °C (Kumar et al. 2013).

Characterization

The morphology of the CFoam, MWCNTs-incorporated CFoam was observed by scanning electron microscope (SEM model LEO 440). Electrical conductivity of CFoam was measured using the four-probe technique. Keithley 224 programmable current source was used for providing constant current (I). The voltage drop (V) in between two pinpoints with a span of 1.2 cm was measured by Keithley 197A auto ranging microvolt digital multimeter.

The CFoam was also characterized by X-ray diffractometer (XRD, RIGAKU Tokyo) to understand the structural changes that take place due to the incorporation of MWCNTs.

The (110) reflection is used to calculate average crystallite width (L_a) by Scherrer equation (Braun and Huttlinger 1996). $L_a = k\lambda/\beta\cos\theta$, where k is Scherrer constant = 1.84

The (002) reflection is used to obtain average crystallite height (L_c), λ wave length of CuK_α radiation is 1.5418 Å, $L_c = k\lambda/\beta\cos\theta$, where $k = 0.9$, is the Scherrer constant, β is the corrected full width at half maxima (FWHM) value in radians.

The electrochemical properties of CFoam were studied with the help of a potentiostat PARSTAT 2263 (Princeton Applied Research). The CV was used to examine the electrochemical behavior of the CFoam with different contents of MWCNTs (0–2 %) using 1 M H₂SO₄ solution as an electrolyte. The CFoam was used as working electrode. Ag/AgCl electrode and platinum plate were used as the reference electrode and counter electrode, respectively.

Result and discussion

Physical and mechanical properties of CFoam

Figure 1 shows the density and porosity of CFoam with increasing content of MWCNTs. Initially, bulk density of CFoam heat treated at 2,500 °C is 0.50 g/cm³ and on addition of MWCNTs, bulk density of CFoam increases slightly. The increase is related to ordering of graphene layer parallel to MWCNTs axis. It was indicated that suitable addition of MWNTs promoted graphitization degree CFoam due to carbon atoms can orderly grow along MWNTs. During pyrolysis the mechanical stresses exert at MWCNT/carbon interface and accelerate ordering of the graphene layer (Li et al. 2011). The increase in the bulk density of CFoam results in the decrease in porosity as shown in Fig. 1.

The mechanical property in the CFoam, i.e., compressive strength is measured by Universal Instron testing machine. The compressive strength of CFoam depends mainly on two factors namely microstructure and bulk density. The microstructure mainly includes width of the ligaments and quantity of micro cracks. The compressive strength of the 1,400 and 2,500 °C heat-treated CFoam is

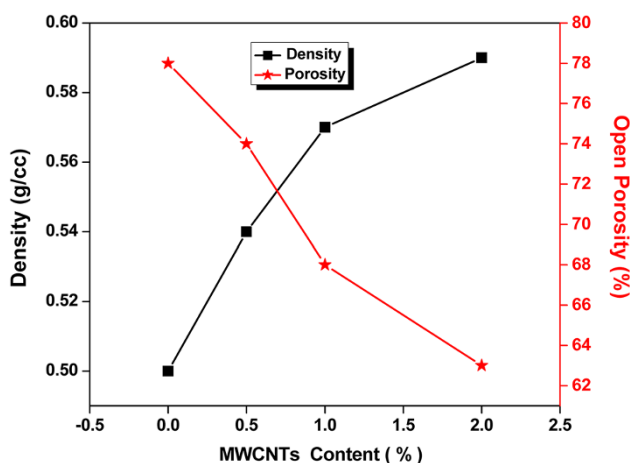


Fig. 1 Variation of bulk density and open porosity of CFoam with increasing MWCNTs content in starting material heat treated at 2,500 °C increasing MWCNTs content

found to be 6.0 and 5.3 MPa. On the other hand, MWCNTs-incorporated CFoam heat treated at 1,400 °C, compressive strength is not enhanced significantly. However, after heat treatment at 2,500 °C, MWCNTs-incorporated CFoam strength increases from 5.3 to 6.4 and 8.0 MPa for 0.5 and 1.0 wt% of MWCNTs contents. However, at higher MWCNTs content, even though the bulk density of the CFoam increases but the simultaneous aggregation of MWCNTs may restrain the enhancement of the compressive strength of CFoam (Kumar et al. 2013).

Figure 2 shows the electrical conductivity with increasing MWCNTs content in 1,400 and 2,500 °C heat-treated CFoam. The electrical conductivity of 1,400 °C heat-treated CFoam is 58 S/cm and with increasing MWCNTs content it increases and maximum 84 S/cm at 1 wt% of MWCNTs. While in case of 2,500 °C heat-treated CFoam, without MWCNTs electrical conductivity is 80 S/cm and with increasing nanotube content conductivity increases. On incorporation of MWCNTs (0–2 wt%), electrical conductivity increases with increasing content of MWCNTs up to 1 wt%, i.e., from 80 to 135 S/cm. The increase in conductivity is due to the increase in conduction path of electron which is directly related to the structure of reinforcing material. The higher content of MWCNTs incorporation has the negative effect on the electrical conductivity due to agglomeration of nanotubes and formation MWCNTs–MWCNTs interfaces.

The surface area data of CFoam are measured by the sorption of nitrogen; it is observed that with increasing MWCNTs content, the surface area increases continually from 2.439 m²/g to 5.25 for 1 wt% of MWCNTs and 7.60 m²/g with 2 wt% of MWCNTs content in CFoam. This clearly shows the surface area increase by two to three

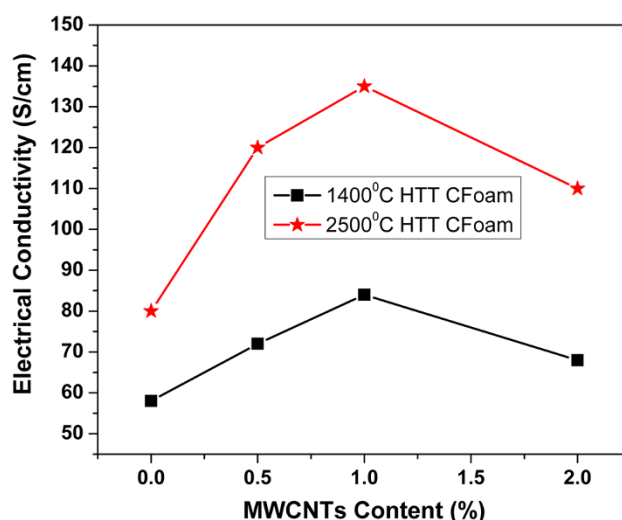


Fig. 2 Electrical conductivity with increasing the MWCNTs content in CFoam of 1,400 and 2,500 °C

times of CFoam on nanostructuring. This may have positive effect on the electrochemical properties of CFoam. Increase in the surface area can enhance utilization level of lead acid chemistry, i.e., liquid diffusion can be increased. Such structure results in much higher power, greater energy delivery and faster recharge process.

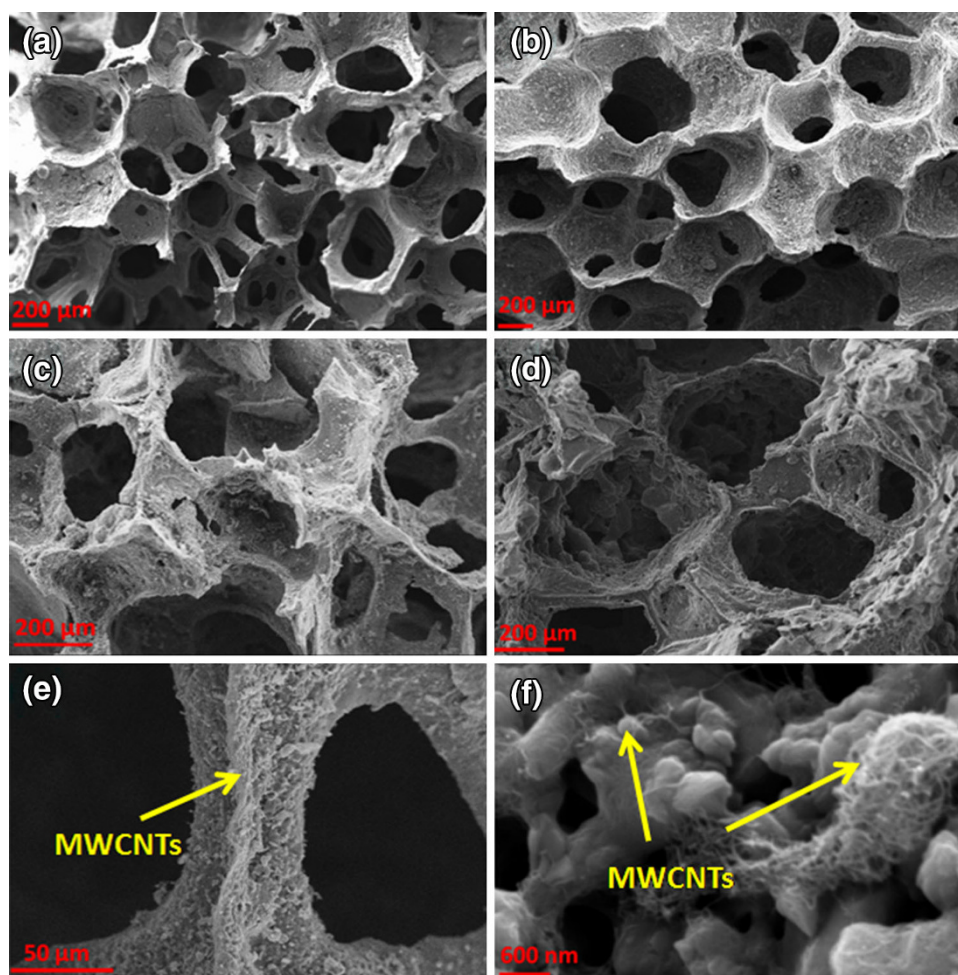
Microstructure of CFoam

Figure 3 shows the SEM micrographs of the CFoam with MWCNTs. Figure 2a, show the CFoam without MWCNTs heat treated at 2,500 °C, the pores are uniformly distributed and some pore walls (i.e., ligaments) are broken during machining of samples for SEM characterization because of the brittle nature of material. Figure 3b–d shows the SEM image of CFoam with 0.5, 1.0 and 2.0 wt% of MWCNTs content, respectively. The CFoam encapsulation of MWCNTs in the ligament leads to wider and thicker cell walls as compared to CFoam. Further, the number of cracks got reduced in the Fig. 3b, c due to the presence of MWCNTs that can act as barrier for propagating the cracks. The MWCNTs also act as nucleation site in

modified CTP-derived carbon for the alignment of carbon atoms or graphene layers along the MWCNTs axis (Fig. 3c, MWCNTs-incorporated CFoam with 1 wt%). Figure 3e shows the SEM image of CFoam ligaments which reveal the deposition of MWCNTs over ligaments which influence the ligament thickness. Further, homogeneous and dense distribution of MWCNTs over entire surface of CFoam can be visible at higher magnification in Fig. 3f. It shows that addition of MWCNTs obviously influence the pore structure in terms of cell wall thickness, width of ligament, and open porosity.

Figure 4 shows the XRD spectra of 2,500 °C heat-treated CFoam. The diffraction peaks are observed at $2\theta \sim 26.3^\circ, 43^\circ, 45^\circ, 54^\circ$ and 77° correspond to different diffraction planes 002, 100, 101, 004 and 110, respectively. The incorporation of MWCNTs influences the structure of CFoam, and as a consequence peaks registered at diffraction angle and intensity of each peak also change. The interlayer spacing of 0 % MWCNTs CFoam is 0.3387 nm and that of MWCNTs-incorporated foam is 0.3381, 0.3374, 0.3394 nm for MWCNTs loading 0.5, 1.0 and 2.0 wt%, respectively.

Fig. 3 SEM of CFoam heat treated at 2,500 °C **a** 0 % MWCNTs, **b** 0.5 % MWCNTs, **c** 1.0 % MWCNTs, **d** 2 % MWCNTs, **e** MWCNTs deposited in ligament of CFoam and **f** showing agglomeration of MWCNTs



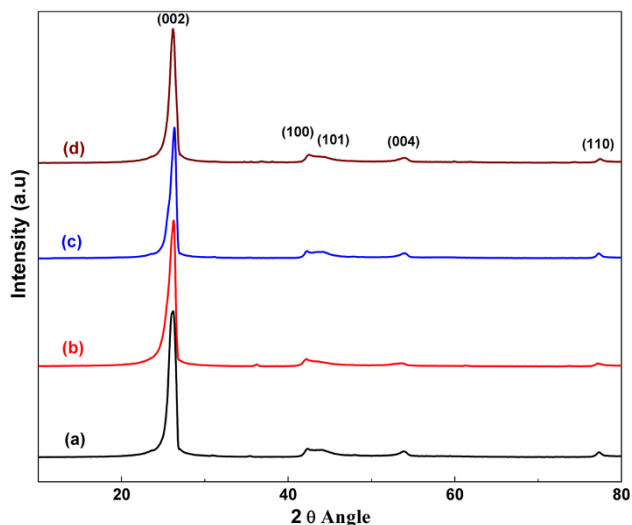


Fig. 4 XRD spectra of CFoam with increasing MWCNTs content, curve (a) 0 %, curve (b) 0.5 %, curve (c) 1.0 % and curve (d) 2 % MWCNTs incorporated in starting material and heat treated at 2,500 °C

The crystalline width (L_a) and height (L_c) is calculated from the diffraction peak of 110 and 002 (Fig. 4), and both increase with increasing content of MWCNTs in CFoam. After certain amount of MWCNTs the L_a L_c value decreased. The L_a value of CFoam is 24.1, 26.0, 34.6 and 18.6 nm, while L_c of CFoam is 7.9, 8.8, 11.3 and 8.8 nm for 0, 0.5, 1.0, and 2 wt% of MWCNTs content. This suggests that up to 1 wt% MWCNTs can help in improving the structure of CF, i.e., increases in L_c value. However, higher content of MWCNTs has negative effect on the crystalline parameters. This attributed to the improvement of staking order of graphene layers which can positively influence the electrical conductivity and electrochemical properties of CFoam.

Electrochemical properties of CFoam

The cyclic voltammetry (CV) is used to examine the electrochemical behavior of the CFoam with different contents of MWCNTs (0–2 %) in the voltage range (–1 to +1 V) using 1 M H_2SO_4 solution as an electrolyte. The CFoam is used as working electrode. Ag/AgCl electrode and platinum plate are used as the reference electrode and counter electrode, respectively. Figures 5 and 6 show the CV of MWCNTs containing CFoam electrode in H_2SO_4 electrolyte at different scan rates (from 100 to 1,000 mV/s) heat treated at 1,400 and 2,500 °C. The CV curve attributes to two peaks, i.e., anodic and cathodic peaks. The electrons transferred in a redox (reduction and oxidation) reaction arise from the change of the valance state of materials.

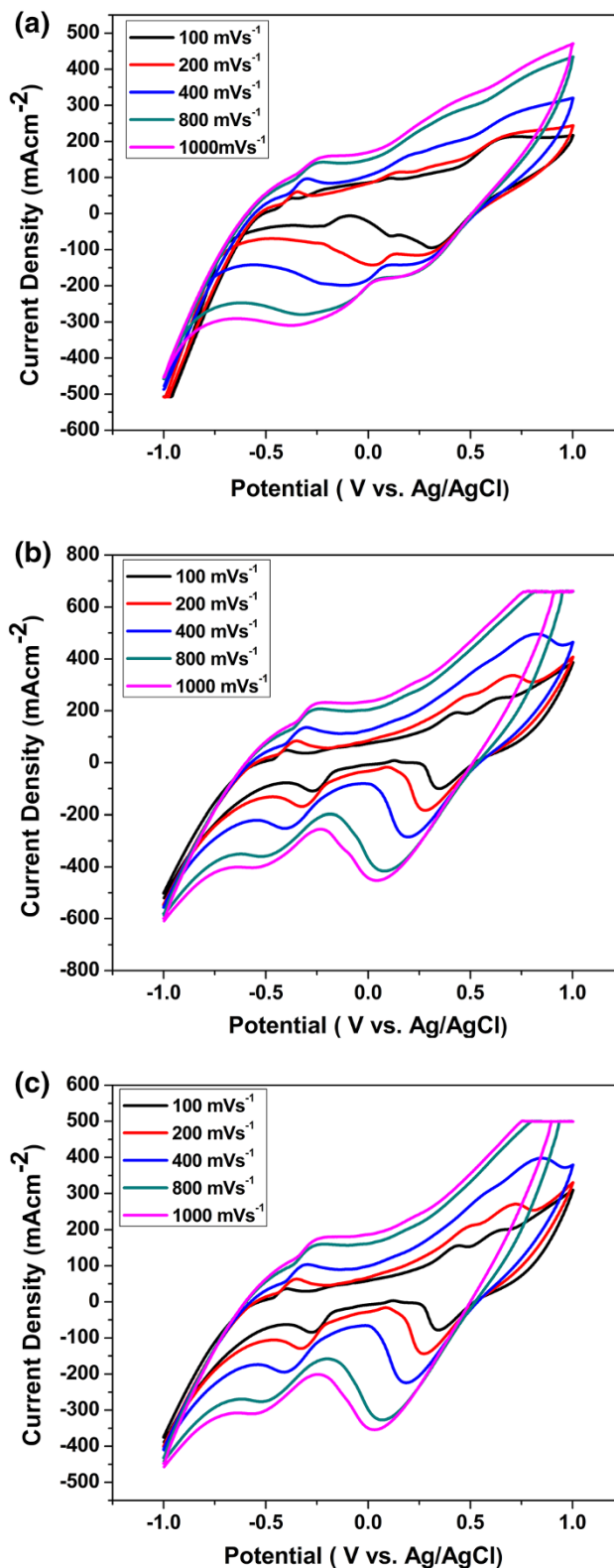


Fig. 5 Cyclic voltammetry behavior in 1 M H_2SO_4 electrolyte solution at different scan rates of 1,400 °C heat-treated CFoams with a 0, b 1, and c 2 % MWCNTs content

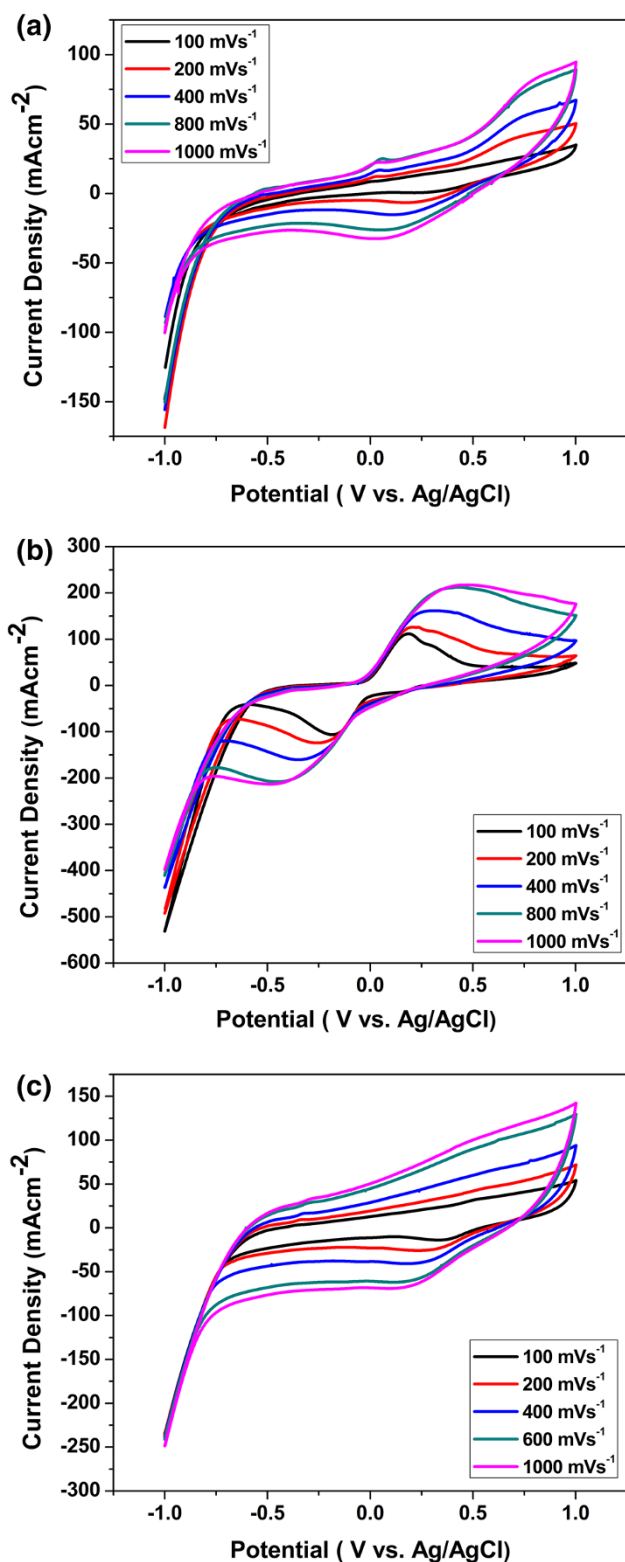


Fig. 6 Cyclic voltammeter behavior in 1 M H_2SO_4 electrolyte solution at different scan rates of 2,500 °C heat-treated CFoams with a 0, b 1, and c 2 % MWCNTs content

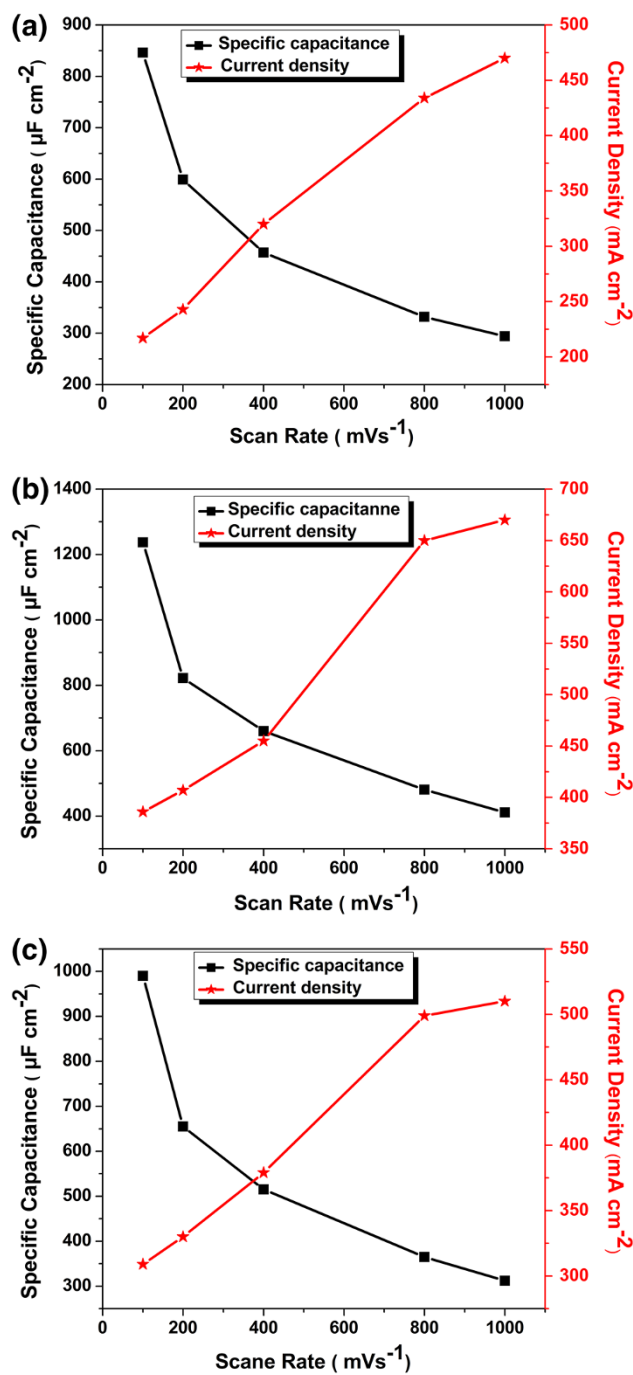


Fig. 7 Specific capacitance and current density of 1,400 °C heat-treated CFoams with a 0, b 1, and c 2 % MWCNTs content at different scan rates

Figure 5, in case of 0 % MWCNTs-incorporated CFoam HTT at 1,400 °C shows the current density 500 mA/cm² at the scan rate 1,000 mV/s; on increasing the MWCNTs content (1 %) in the CFoam the current density increases

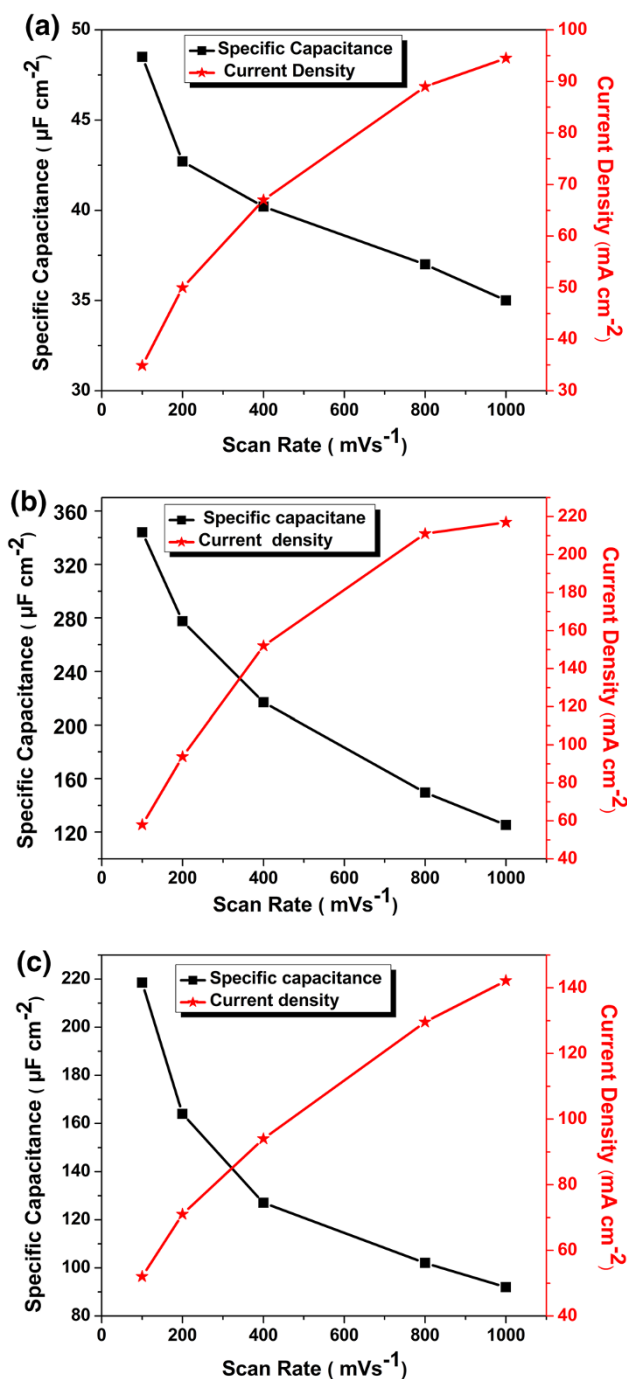


Fig. 8 Specific capacitance and current density of 2,500 °C heat-treated Cfoams with **a** 0, **b** 1, and **c** 2 % MWCNTs content at different scan rates

600 mA/cm, due to increase in its electrical conductivity (Kumar et al. 2013). After a certain amount of MWCNTs (2 wt%) in the Cfoam the current density decreases to 447 mA/cm². This may be due to the decrease in its electrical conductivity or higher amount of MWCNTs in the matrix of carbon foam or agglomeration effect which is shown in SEM images. Due to the agglomeration of

nanotubes that formed MWCNTs–MWCNTs interfaces which inhibit conduction path and as result decreases in electrical conductivity. In case of 1 % MWCNTs-incorporated Cfoam HTT at 2,500 °C shows the maximum current density 200 mA/cm² at the scan rate 1,000 mV/s. As we compare the current density of 1 % MWCNTs Cfoam heat treated at 1,400 and 2,500 °C, it displays that the current density of Cfoam heat treated at 1,400 °C is (600 mA/cm), which is higher as the Cfoam heat treated at 2,500 °C (200 mA/cm²). Further, MWCNTs play an important role on the enhanced of electrical properties of Cfoam. This is attributed to higher electrical conductivity of the Cfoam, this type of Cfoam favored for battery electrode with high open circuit. The specific capacitance (SC) of Cfoam has small equivalent series resistance, high rate handling capability and excellent electrochemical performance. Moreover, CV curves exhibit nearly mirror-image current response on voltage reversal, indicating a good reversibility (Wu et al. 2010). In this case, anodic and cathodic peak potentials have maintained the proportionality, i.e., reversible electrochemical behavior.

Figures 7 and 8 show the SC and current density of the Cfoam with increasing scan rate heat treated at 1,400 and 2,500 °C. The SC is calculated from the CV curve by the following equation (Srinivasan and Weidner 2002).

$$SC = \frac{1}{Av(V_f - V_i)} \int_{V_i}^{V_f} I(V)dV$$

where A is the area of the active electrode material, v is the scan rate, V_f and V_i are the integration potential limits of the voltammetric curve, and I(V) is the voltammetric current. The SC curve of 0 to 2 wt% MWCNTs content Cfoam heat treated at 1,400 and 2,500 °C shown in Figs. 7 and 8 respectively. The SC of 1,400 °C HTT Cfoam with 0, 1 and 2 wt% of MWCNTs content is 846, 1,237 and 990 μF/cm² at the scan rate 100 mV/s and current density at the same scan rate is 217, 386 and 300 mA/cm. The SC is still as high as 1,237 μF/cm² even at a high current density 386 mA/cm, in 1 wt% of MWCNTs Cfoam due to higher electrical conductivity. The higher content of MWCNTs (2 wt%) has negative effect on electrical properties and also decreases its specific capacitance due to agglomeration effect of MWCNTs in Cfoam

The SC of graphitized Cfoam with 0, 1 and 2 wt% of MWCNTs content is 48.5, 344 and 218 μF/cm² at the scan rate 100 mV/s and current density at same scan rate is 35, 58 and 52 mA/cm², respectively. The 1 wt% of MWCNTs content Cfoam has maximum SC 344 μF/cm². The SC of Cfoam increases by 600 % with the addition of 1 % MWCNTs in Cfoam as compared to Cfoam without MWCNTs. However, CNTs with large surface area have been extensively studied in supercapacitors, with SC of

4–180 F/g in a solution of H_2SO_4 (Chen et al. 2002). Again after certain amount of MWCNTs, the SC starts decreasing due to the poor dispersion of MWCNTs in CFoam. With the increasing scan rate in all the cases, SC decreases and current density increases. As we compare the SC of 1,400 and 2,500 °C HTT CFoam, it displays that 1,400 HTT CFoam with 1 % MWCNTs has a maximum SC 1,237 $\mu\text{F}/\text{cm}^2$ as compared the 2,500 °C HTT CFoam (344 $\mu\text{F}/\text{cm}^2$). The decrease in SC is associated with intercalation and deintercalation of bisulphate ions and sulfuric acid molecules between graphite layers graphitized CFoam, indicating that non-graphitized CFoam is electrochemically stable in the voltage range of lead acid battery. This demonstrates that the MWCNTs-incorporated CFoam heat treated at 1,400 °C is most suitable with as electrode material in lead acid battery.

Conclusions

In this investigation, cost-effective approach for the development of MWCNTs-incorporated CFoam with improved electrochemical properties by simple sacrificial template technique is reported. It is observed that nanostructuring of CFoam by MWCNTs can enhance the surface area and electrical conductivity. The surface area of CFoam increases from 2.439 m^2/g to 5.25 for 1 wt% of MWCNTs and 7.60 m^2/g with 2 wt% of MWCNTs. During the heat treatment, stresses exert at MWCNTs/carbon interface and accelerate ordering of the graphene layer which have positive effect on the electrical conductivity and electrochemical properties of CFoam. The current density increases from 475 to 675 mA/cm^2 of 1,400 °C heat treated and 95 to 210 mA/cm^2 of 2,500 °C heat-treated CFoam with 1 wt% MWCNTs. The specific capacitance was decreases with increasing the scan rate from 100 to 1,000 mV/s . In case of 1 % MWCNTs content CFoam the specific capacitance at the scan rate 100 mV/s was increased from 850 to 1,250 $\mu\text{F}/\text{cm}^2$ and 48 to 340 $\mu\text{F}/\text{cm}^2$ of CFoam heat treated at 1,400 °C and 2,500 °C respectively. Thus, the higher value surface area and current density of MWCNTs-incorporated CFoam heat treated to 1,400 °C can be suitable for lead acid battery electrode with higher power, greater energy delivery, faster recharge process and significant reduction in the weight of battery. In the next course of investigation, Pb and PbO_2 will be coated on nanostructured CFoam and it will be characterized by cyclic voltammetry evaluation for charge–discharge cycle in cell.

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