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Study of electrospun polycarbosilane (PCS) nanofibrous web by needle-less technique

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Abstract

This study reports on the various functional characteristics of silicon carbide (SiC) nanofibrous web. The SiC nanofibrous web was spun by the electrospinning technique using Nano Spider (needle-less) machine. The as-spun nanofibrous web was cured to 180°C and subsequently, pyrolized at 1000°C under inert nitrogen (N_2) atmosphere to convert into silicon carbide nanofibrous web. The various properties of SiC web is characterized by using FESEM, Thermal Analysis, X-ray Diffraction, Energy Dispersive Spectroscopy (EDX), Atomic Force Microscopy and Surface Profilomertry. FESEM microphotographs indicated the interconnected fibres leading to pores of prepared SiC Nanofibrous web. Deep rooted fibre surface porosity was revealed by AFM. The thermal behavior of as-spun, cured and pyrolized PCS webs are influenced by the heat treatment at different temperatures. The surface roughness changes with the heat treatment of PCS nanofibrous webs. The pyrolized web carries higher surface roughness as compared to as-spun and cured webs. The EDX plots indicated the presence of C and Si elements in pyrolized PCS nanofibrous web.

Keywords: DSC; FESEM; Nanofibrous web; PCS; SiC; Topography; XRD

Introduction

A brief introduction to the process of electrospinning in the context of technical grade composite nanofibres is presented in this section. Furthermore, this section elaborates a brief review of current and past research activities that focus on the development of ceramic electrospinning and their applications in various areas.

Electrospinning is an attractive and simple process capable of spinning fibres from melt or polymer solution using an electric field (Salem 2007; Chase et al. 2011). Electrospinning process provides a straightforward electrohydro-dynamical mechanism to produce fibres with diameters even less than 100 nm (Frenot and Chronakis 2003). Under the influence of an electric field, a pendant droplet of the polymer solution at the spinneret is deformed into a conical shape. Such deformation is dependent on solvent physical properties like good miscibility, low boiling point, low surface tension, high conductivity and low dielectric constant (Salem 2007).

The electrospinning of technical grade composite nanofibres is still challenging and attractive for technical applications like biomedical, filtration, conductive and anti-microbial etc. Various synthetic & natural polymers such as; polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), chitosan, polyvinyl acetate (PVAc), polyacrylonitrile (PAN) based carbon fibre, polyvinylidene fluoride, polymethyl methacrylate (PMMA),

polyamic acid are used for preparation of composite nanofibres. These polymer either blended or filled with nanoparticles or carbon nanotubes (CNTs) and have been successfully electrospun into composite nanofibres (Bhardwaj and Kundu 2010; Peresin et al. 2010; Choi et al. 2004, 2008; Ding et al. 2009; Geng et al. 2005; Luhrs et al. 2009; Sundaray et al. 2008; Jirsak et al. 2010; Wang et al. 2009; Ko 2006; Sundaray 2006). Out of these, polyvinyl alcohol (PVA) is one of the most popular polymer, which is employed as a matrix due to its high solubility in water and its good compatibility with many salts, including zinc acetate and copper nitrate (Chuangchote and Supaphol 2006; He et al. 2010).

Now a day, there is much demand and usage of high performance fibre carrying multi-functional characteristics. In this direction, electrospinning has been further explored for the generation of ceramic nanofibre. Various ceramic nanofibres mostly oxide based are fabricated by the combination of two conventional techniques: electrospinning and sol-gel (Yang et al. 2010; Lu et al. 2009). In sol-gel technique, mainly four steps involves; (1) particle to form desired colloidal solution, (2) Colloidal solution either sprayed, coated or electrospun on substrate, (3) sol particles to form gel in a state of a continuous network (4) Finally, pyrolyze the remaining organic or inorganic components to form an amorphous or crystalline ceramic compounds. Various oxide nanofibres and composites can be synthesized by this approach are MgO, ZnO, CuO, NiO, TiO_2, SiO_2, Cr_2O_3, Al_2O_3, SnO_2, Fe_2O_3, $NiFe_2O_4$, $LiFePO_4$ and Pt and in addition to these, hybrid fibres, strontium ferrite composite nanofibres and Mg & Zn mixed nanofibers can also be prepared by using this method (Lu et al. 2009; Wanga et al. 2010; Park et al. 2010; Kim et al. 2009; Hota et al. 2009; Sundarrajan and Ramakrishna 2007; Shukla and Kumar 2008; Yang 2007; Chen et al. 2010; Nakanea et al. 2013.; Dai et al. 2013; Ayku et al. 2013). Preparation and magnetic properties of lanthanum- and cobalt-codoped M-type strontium ferrite nanofibres was recently fabricated by electrospinning of solution, which exhibits significant improvements in the magnetic properties than the nanoparticles obtained by the sol-gel process (Moallemian et al. 2013). Therefore, not much importance has been paid to sol-gel process due to its certain limitation e.g. weak bonding and low degree of functional properties, low wear-resistance and high permeability.

Hence, now attention has been focused to electrospinning the ceramic nanofibre from base ceramic polymers. Generally, ceramic nanofibres are made by the electrospinning of ceramic precursors followed by calcination at higher temperatures (Teo and Ramakrishna 2009; Goldstein 2004; Huanga et al. 2003). In fact, Silicon carbide (SiC) is one of the well-known non-oxide ceramics used as a high-temperature structural material and it's also demonstrate numerous potential applications in the field of high-tech application areas like aerospace, composite materials, semiconducting devices. These high technical applications are mainly due to their excellent properties like high thermal conductivity, high thermal stability, high strength & hardness and good resistance to oxidation and corrosion (Wallenberger et al. 1999; Shin et al. 2008). In this segment, Polycarbosilane (PCS) is the promising material for fabricating Silicon carbide (SiC) nanofibrous ceramic webs. SiC fibres are derived from PCS with the process, which is more over similar to that of carbon fibre preparation process. The first invention of SiC fibres are oxygen containing fibre having poor thermal stability and second generation SiC fibres are defined by oxygen free carbon with Silicon in the

form of SiC microstructure is stable at higher temperature (Wallenberger et al. 1999). Additionally, due to high surface area to mass ratio, an enhanced efficiency of the ceramic nanoweb could be exploited. Various previous researchers reported about pure SiC web formation and its composites. Moreover, fabrication of pure SiC nanowebs was done by syringe and needle type electrospinning set up and also by solution blowing method (Shin et al. 2008). But the conventional nozzle based electrospinning process has significantly lower production rate (0.1–0.2 g/hour) as compared to the needle-less electrospinning process (1–2 g/min).

Looking at the governance of SiC fibres in various applications, an attempt was made herewith for development of pure SiC nanofibrous web through needle-less technique. This research work is carried out on a latest electrospinning technique having very high throughput rate. The present work has additional aim to fabricate SiC nanofibrous web having well connected fibres leading to pores network, which may be exploited for filtration purpose. This developed nanofibrous SiC web was subjected for evaluation of various characteristics using FESEM, Thermal Analysis, X-ray Diffraction, Energy Dispersive Spectroscopy (EDX), Atomic Force Microscopy (AFM) and Surface Profilomertry. A better understanding of the process and techniques, opens the future path for further researching on nanofibrous coating of various densities on high performance fabric of different morphological structures. In summary, an optimized electrospinning process was established to achieve desired properties of SiC nanofibrous (high performance fibre) web at a very high throughput rate (g/m^2). Such high performance SiC web is obtained by precise control of various process parameters and that could be noted as a high point of this study.

Methods

Solution preparation

Spinnable and amorphous grade of Polycarbosilane (PCS) with average molecular weight of 800 was used as basic polymeric material. The solvent mixture of DMF and toluene in ratio of 20% (v/v) was prepared by gentle stirring for 12 hours at ambient temperature. Subsequently, PCS was added to the mixed solvent at concentration of 1.2 g/ml and stirred for 24 hour at room temperature to obtain uniform solution, which is described elsewhere (Shin et al. 2008).

Electrospinning, curing and pyrolization

The electrospinning work is carried out through the commercially available Nanospider machine (Model NS Lab 200S) supplied by ELMARCO, Inc., Czech Republic. The electrospinning system comprises of three parts: spinning electrode, collecting electrode and high voltage supply (80 kV). The prepared PCS solution was loaded into a spinning electrode and spinning electrode was rotated at 9 rpm. The distance between the spinning electrode and collecting electrode was positioned at 160 mm. The thin layer of PCS solution was carried on the drum surface and exposed to the high voltage electric field and PCS nanofibrous web was obtained (Figure 1). The PCS web was collected on aluminum foil and was cured at 180°C for 1 hour in a furnace. These cured PCS webs are pyrolised under nitrogen gas condition (temperature =1000°C and time = 60 minutes) to convert into SiC nanofibrous web.

Figure 1 Electro-spinning set up and conversion of polymeric material into nanofibrous web.

Surface morphology and diameter characterization

The morphology of electrospun fibres were examined by using the Field Emission Scanning Electron Microscopy (Zeiss EV 050). The diameters of nanofibres were estimated by Image J software using the captured FESEM images. The average fibre diameter was determined from 30 measurements of the random fibres taken from different areas of the scanned images. The 3D structure of nanofibrous web is investigated by Atomic Force Microscope (Agilent 5500 SPM AFM). The FESEM integrated with a Phoenix Energy Dispersive X-ray (EDX) detector was used to analyze the surface structure of pyrolized web.

Thermogravimetric analysis

Thermogravimetric analysis (Universal V47A TA) is employed to monitor the changes, which occurred during heating of web under nitrogen (N_2) atmosphere with a heating rate of 10°C/min with a temperature range between 30–1000°C. The obtained TGA curves are interpreted for the thermal behavior of the samples by matching the endothermic peaks. Moreover, TGA experiments were performed under identical conditions of pyrolization.

DSC analysis

Differential Scanning Calorimetry (DSC Q 200 V24.8) was used to measure the physical properties of samples under N_2 atmosphere with a heating rate of 10°C/min in a temperature range between 30–300°C.

Crystalline structure

To understand the structural properties, the Wide Angle X-ray (WAXD) diffraction traces of webs are recorded with a Phillips diffractometer (Model like Expert Pro) using copper K alpha radiation in reflectance mode (40 Kv/20 mA) at a scanning speed of 0.02 degrees/second. The samples are scanned for 2θ angle from 10 to 80 degrees.

Optical profiler

The topography (roughness) of the nanofibrous samples were measured by μScan (Nano Focus Inc.) using a confocal sensor for as-spun to pyrolized webs.

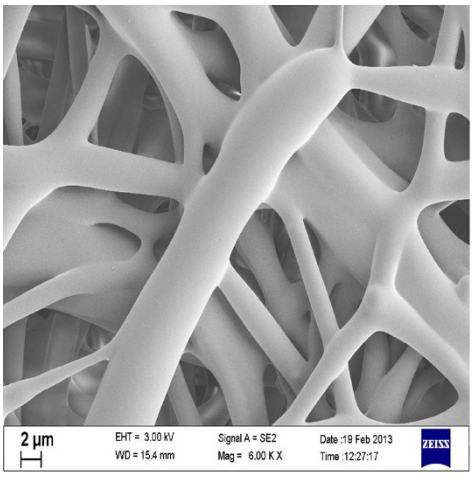
Results and discussion

Morphology of as-spun, cured and pyrolized nanofibrous webs

The FESEM images were used to observe the surface morphology of webs before and after heat treatment. The FESEM images of as-spun, cured and pyrolized nanofibrous webs are shown in Figure 2. It is observed that nanofibres diameter get thinner and more irregular after pyrolization as compared to the as-spun fibres. The as-spun webs are having higher average diameter (2 μm) than the pyrolized web (1.5 μm). The FESEM images also revealed that on evaporation of solvent (after curing), micro pores are created on fibre surface. The heat treatment has caused two physical changes in the nanofibrous web; solvent get evaporated, which leads to formation of pore and shrinkage in the web, which has resulted in thinner and more irregular fibre web. The similar kind of finding of morphological transformation due to heat treatment of PCS has been also reported by researchers (Lu et al. 2010; Ulrich 2006). The FESEM images also confirmed that the fibers have on fine circular cross-section. It can be also noticed from the FESEM image of as-spun web comprises of interconnected fibers creating pore between fibres. However, pore which has been seen on the surface of fibre, after curing needs to further investigated in terms of whether pore is superficially on fibre surfaces or it is deeply rooted. Hence, AFM was used to capture the image in 3D format. The 3D AFM image (Figure 3) of the pyrolized web reveals the surface pores is deeply rooted (depth). Therefore, it may be concluded that two types of pore is observed; (1) surface pore which is deeply rooted in fibre (revealed by AFM) and (2) interconnected fibres causing pores network (revealed by FESEM). Therefore, this finding indicated that it is excellent for filtration applications. This kind of phenomenon is in good agreement with those of previously reported results (Lu et al. 2010; Ulrich 2006).

Thermal behaviour of webs

In order to monitor thermal performace of webs TGA experiment was performed under identical condtions of pyrolization. The TGA plots of as-spun, cured and pyrolized webs are presented in Figure 4. It can be observed from the TGA plots of the as-spun and cured webs that the weight is decreasing with the increase in temperature. The cured web showed a sharp decomposition above 330°C and the ceramic yield up to 64%, which was substantially lower than that of the as-spun web. This is closely related to the H_2 or O_2 evolution, which might have contributed to sharp weight loss around 330°C lowering of SiC yield of cured web, which might be caused by the evaporation of the solvent during curing process and this is possibly hindering the conversion of PCS into SiC fibres. This result suggests that the curing process significantly changed the chemistry and bonding characteristics of the electrospun PCS fibres. The pyrolized web reflects a very peculiar kind of behaviour, indcating two stage weight loss and one stage weight gain. At temprature ranging from 120–350°C weight loss is about 1.4%, being attibuted to the evapertation of volatile & slovents componets in PCS. At temeperture ranging 480–610°C, a minor weight gain of 0.9% was observed and this may be



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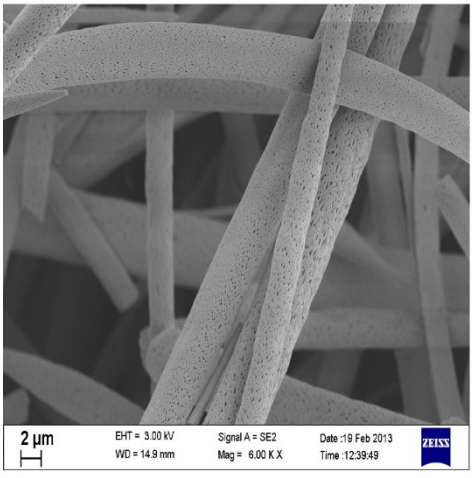

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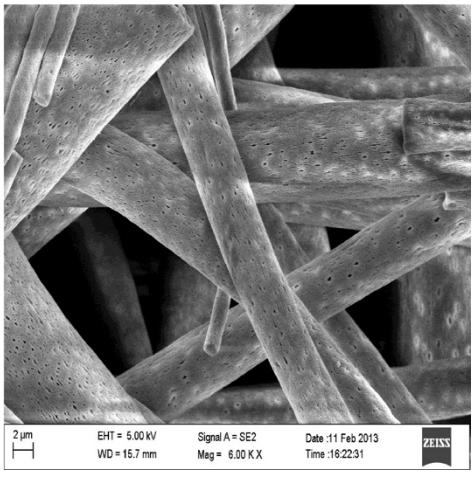


(c)

Figure 2 FESEM images of PCS nano webs: (a) as-spun (b) cured (c) pyrolized.

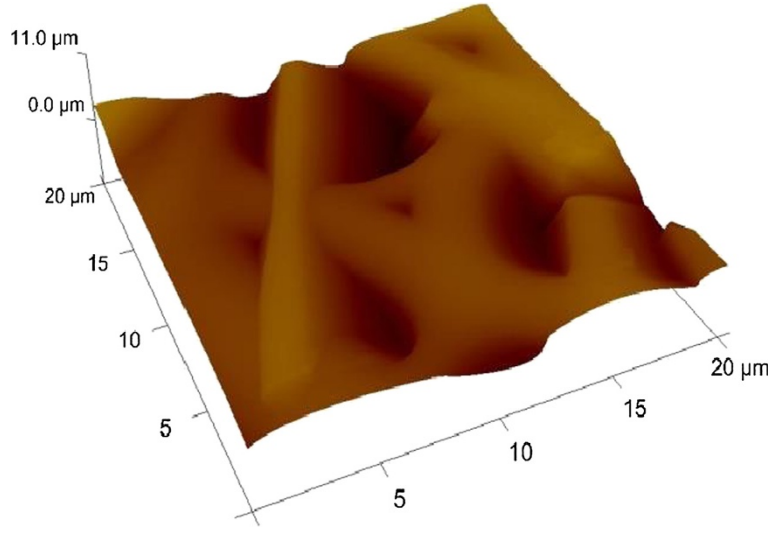


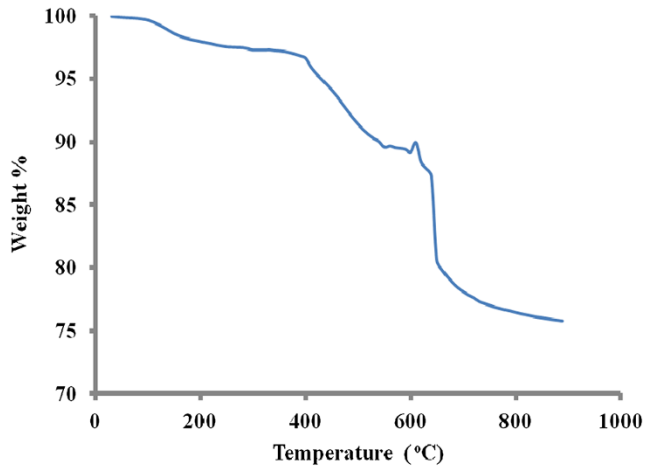
Figure 3 AFM image of SiC nanofibrous web.

attibuted to presence of O_2 as an impurity in N_2 gas, which may leads to formation of some sort of Silicon oxy compunds. But the weight gain is not significant enough to affect the thermal behaviour and can be treated as negligibale gain. At temeperture ranging 650–750°C, a minor weight loss of 0.35% was observed and is being attibuted to complete pyrolysis of PCS. This minor weight loss could be interpreted as formed/trapped atmospheric gases available in atmosphere, which has been possibly release on heating. Above 750°C, there is no weight loss and this indicated the completition SiC formation. Hence, it can be concluded that the thermally stable SiC web formation was ocuuured though pyrolisis and found to be stable beyond 750°C. However, other researchers found that no weight loss occurs after 700°C (Shin et al. 2008; Qiao et al. 2007), which is consistent to our reported results. There is a minor decomposition of SiC web above 650°C and overall ceramic yield was up to approximately 97.35%, which was substantially higher than that of the as-spun web (Wallenberger et al. 1999; Shin et al. 2008).

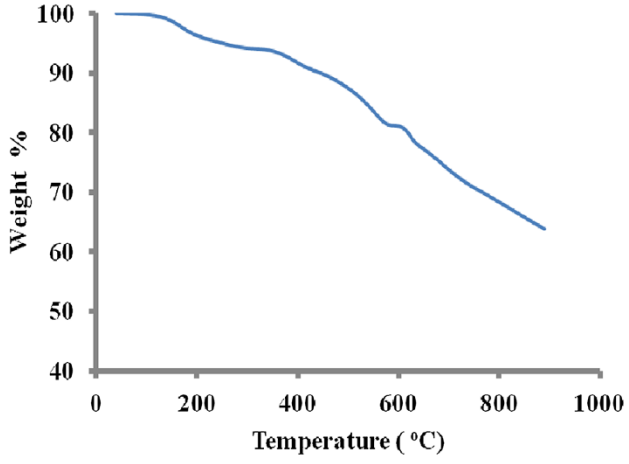
The DSC plots of as-spun, cured, and pyrolized webs are shown in Figure 5. The three webs showed the similar trend and the peak is more pronounced and boarder in case of as-spun web and its peak temperature is shifted from 170°C to 65–75°C respectively. It could be seen that DSC and TGA results supports to each other. The sharp DSC peak appeared in as-spun web revealed a formation of new crystalline morphology possibly due to internal stress and elongation of polymeric fluid jet during electrospinning process (Reneker and Yarin 2008). A very minute exothermic hump is shown in DSC plot of pyrolized web (Figure 5c) and coincidently minor weight gain is also noticed in pyrolized web, as observed by TGA plot (Figure 4c) and this minor weight gain is possibly confirming some sort of Silicon oxy compunds foramtion due to presence of impurties.

Structural identification of PCS and SiC

The XRD plot for the PCS as-spun and SiC (PCS pyrolized) webs are shown in Figure 6. A diffused peak around 10 degree (2θ) was seen in as- spun web, which corresponds to



(a)



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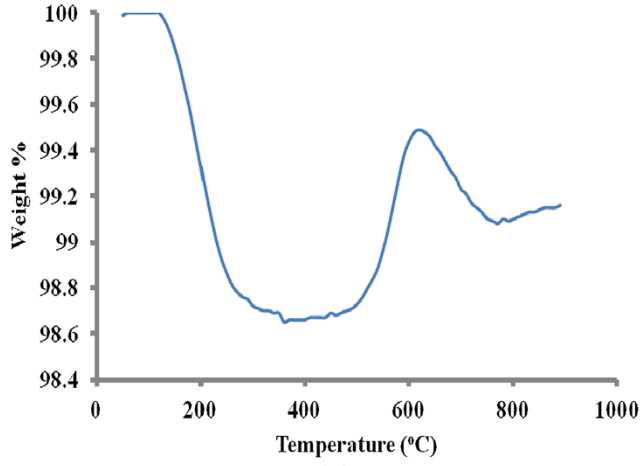


(c)

Figure 4 TGA plots of PCS: (a) as-spun (b) cured (c) pyrolized.

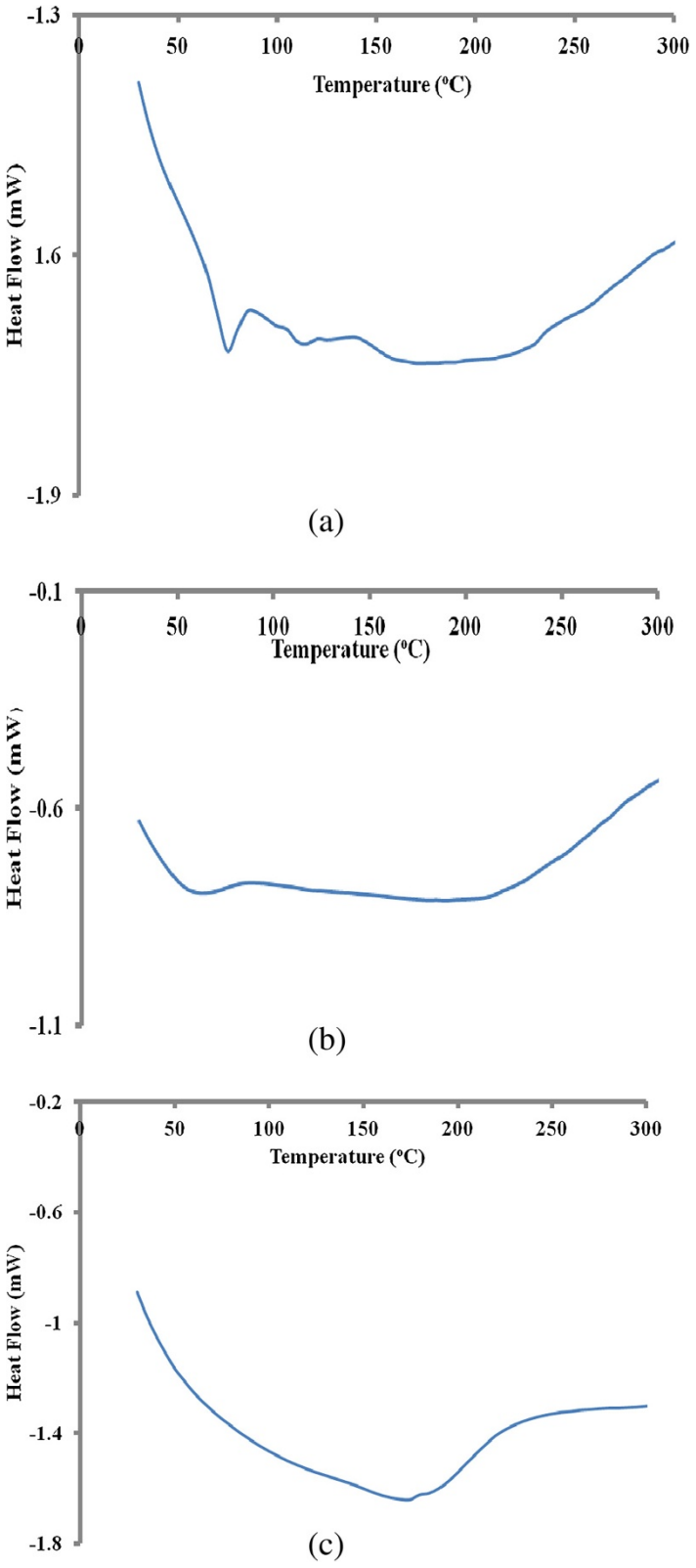


Figure 5 DSC plots of PCS: (a) as-spun (b) cured (c) pyrolized.

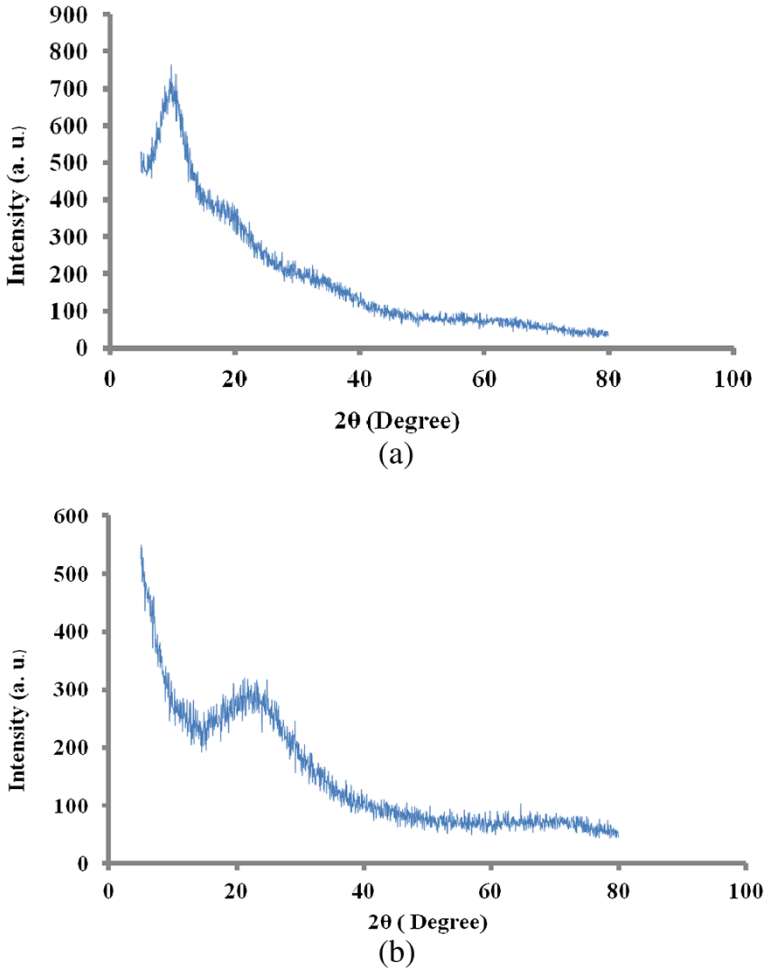


Figure 6 XRD plots of PCS: (a) as-spun (b) pyrolized.

dveopement of new crystalline morophology due to electrospinng process, which is having crystallinity of 10.4%. Thus X-rays confirm the development of new crystalline morphology as a result of electro spinning process in as-spun web. Broad peak at around 24 degree (2θ) is detected in SiC web and this is in close agreement with previous reported work (Correa 2007). During the pyrolysis at 1000°C, the PCS fibre is converted into inorganic phases of SiC nano-crystals and accordingly, peak is obtained at approximate 24 degree and nanofibre showed very broad bands consist of primarily α amorphous silicon carbide having crystal size of 1.4 nm and crystallinity of 22%. It was not easy to deduce the structure of the nanofibre, but the elemental composition shown in Figure 6(b) corresponded to an amorphous silicon carbide.

Chemical identification

The EDX plot of SiC nanofibrous web is s presented in Figure 7. The spectrum revealed the existence of Si and C in the nanofibres. The EDX mappings of C/Si ratio of 0.97 (approximately 1), which clearly reveals the formation of microcrystalline SiC on pyrolysis of PCS (Wallenberger et al. 1999; Lu et al. 2010).

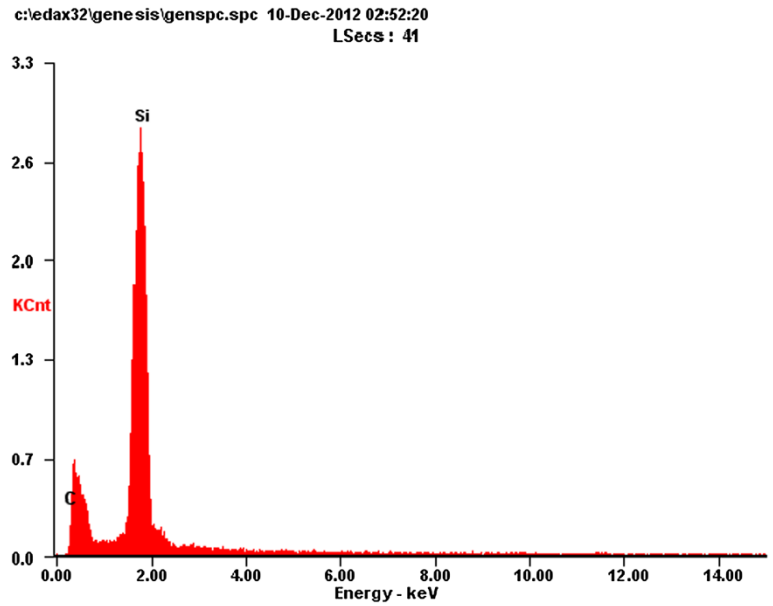


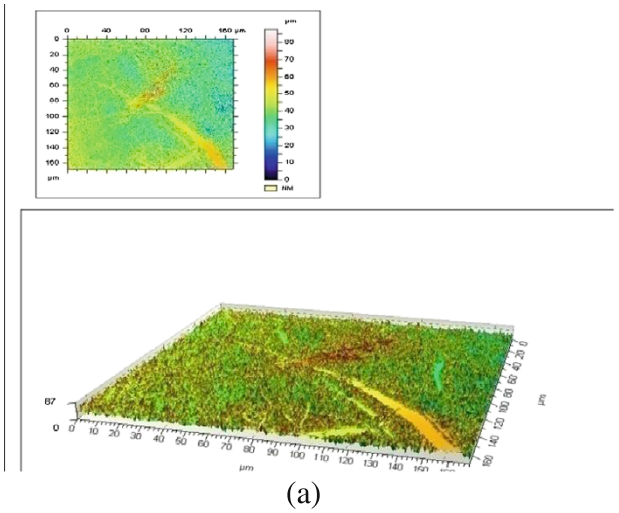
Figure 7 EDX plot of SiC nanofibrous web.

Surface profilemetry analysis

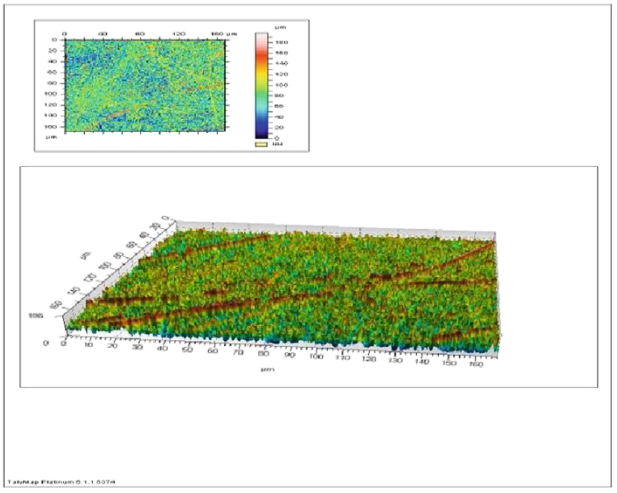
There is intensive use of nanofibre based structures in reinforcement of composites. For this end use, roughness is of prime importance for obtaining high degree of adhesion during composite development. Basically heat is one of the components, which affect the surface roughness. The topography (roughness) plots of the nanofibrous samples (as-spun, cured and pyrolized web) are shown in Figure 8. In the topography plots, light (maximum) and dark (minimum) colours indicate the variation in topography. The measured value of Root Mean Square Deviation of the roughness profile are 9.66, 11.23 & 30.12 μm for as-spun, cured and pyrolized webs respectively. Hence, it is confirmed that thermal treatment makes much impact on the surface roughness of these developed nanofibrous webs and the pyrolized web is having higher roughness, which enhances its suitability for composite preparation.

Conclusion

In the present study, we successfully fabricated and evaluated the functional properties of PCS nanofibre web by using latest Nano Spider machine, having very high throughput rate. A precise degree of process control was carried out to fabricate such SiC web. The fabricated SiC web is having average fibre diameters of approximately 1.5 μm. Heat treatment has significant influence on the morphology of PCS nanofibrous web. The as-spun PCS fibre is straight and becomes thinner & more irregular after pyrolization. SiC nanofibrous web is having interconnected fibre and leading to pores network and it is also confirmed that surface porous structure is deeply rooted, as revealed by 3D AFM image. The formation of pores network by the interconnected fibres could be exploited for filter applications. There are chances of new crystalline morphology formation during electrospinning of as-spun PCS web as indicated by the XRD and DSC. The formation and growth of new crystalline morphological structure is due to stress



(a)



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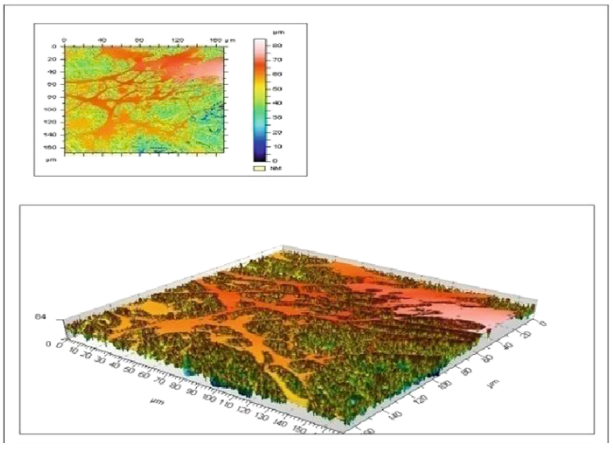

(c)

Figure 8 PCS web Surface profilometry images webs: (a) as-spun (b) cured (c) pyrolized.

on the PCS fluid during the high speed electrospinning process. Surface profilemetry revealed increase in surface roughness with the heat treatment of PCS webs. The SiC web is having highest roughness followed by cured and as-spun PCS webs, as it is confirmed by surface profilemetry in quantitative term and values. A thermally stable SiC web is fabricated, as confirmed by TGA analysis. DSC and TGA results correlate to each other. The ratio of C/Si is approximately 1 in SiC nanofibrous web, confirming high yield of SiC fibres without any impurities or other compounds. In summary, an optimized process was established, which is having scope for bulk production and also explore light weight nano coating on various high performance fabrics.

Competing interests
The authors declare that they have no competing interests.

Authors' contributions
M-KS carried out the electospinning of nano fibrous web and also drafted the manuscript (as a main author). B-RD conducted the characterization of webs. R-M & A-R synthesized and prepared the PCS and its evaluation. A-S and A-KS performed the analysis and interpretation of the test results. All authors read, approved and contributed the final manuscript.

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