# Mechanical and Structural Characterizations of Simultaneously Aligned and Heat Treated PAN Nanofibers

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**ABSTRACT:** A simple and nonconventional electrospinning technique was employed for producing aligned polyacrylonitrile (PAN) nanofibers. A thermal zone was placed between syringe needles and collector in the electrospinning set up to obtain aligned and heat treated nanofibers. Suitable temperatures for heat treat process of PAN nanofibers was determined using differential scanning spectroscopy (DSC) technique. The influence of treatment temperature was investigated on morphology, internal structure and mechanical properties of collected PAN nanofibers. The average fiber diameter measured from SEM images exhibited decreasing trend at higher temperatures. FTIR spectra indicated no considerable difference between chemical structure of untreated and treated PAN nanofibers. Crystallization degree of PAN nanofibers calculated from WAXD patterns showed relatively low change with treatment temperature. Tenacity values of nanofiber bundles increased with increasing temperature while the extension values had an inverse trend. However, the modulus did not show a regular manner, but treated nanofibers had more modulus than untreated ones. The stress and modulus of PAN nanofibers increased to 112.9 MPa and 7.25 GPa at 270°C, respectively. Nanofibers treated at the highest temperature had the largest amount of crystallinity and strength. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 3529–3537, 2012

**Key words:** polyacrylonitrile; aligned nanofibers; heat treatment; electrospinning; mechanical properties; X-ray diffraction

## INTRODUCTION

Polymeric fibers with diameters in the submicron or nanometer range may be optimal candidates for various applications due to their high surface to volume ratio and their potential for mechanical properties. Polymer nanofibers are expected to have mechanical properties different from those of their conventional counterparts. Electrospinning is a quick, straightforward, simple, cost-effective method to produce novel fibers through the use of columbic forces. The electrospinning technique allows for the preparation of reproducible, continuous fibers with diameters in the micron to nanometer size range from polymer solutions and melts at room temperature in a matter of seconds. Because of initial instability of the jet, fibers are often collected as randomly oriented structures in the form of nonwoven mats, where the stationary target is used as a collector. These nanofibers are acceptable only for use in some applications, such as: filtration, wound dressings, and tissue scaffolding. However, to expand the use of electrospun fiber into commercial fiber for textile applications, researchers need to provide a mechanism to obtain a continuous single nanofiber, or uniaxial fiber bundle. Researchers have attempted to obtain aligned electrospun fibers by various approaches, including: spinning onto a rotating drum or onto the sharp edge of a thin rotating wheel, introducing an auxiliary electrode or electrical field, rapidly oscillating a grounded frame within the jet, and using a metal frame as a collector.[1,2] Various degrees of fiber alignment are obtained by these approaches, although only relatively short tows of aligned fiber are obtained. Recent studies have shown that aligned nanofibers have better molecular orientation and as a result improved mechanical properties than randomly oriented nanofibers.[3–5] These nanofibers can be used in applications such as composite reinforcement and device manufacture. Additionally, the aligned nanofibers are better suited for preparing of carbon nanofibers from electrospun PAN nanofibers precursor.[4,5] On the other hand, applying thermal treatment on textile fibers causes fiber stability and as a result better mechanical properties of them.[6–8]

There has been a tremendous growth of research activities dedicated to the generation of new nanostructure materials and their applications, but few researchers have investigated the effect of heat

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treatment to control the mechanical properties of electrospun materials. Zong et al. investigated the use of post-treatment to tailor the mechanical and biodegradation properties of entangled, electrospun, poly (glycolide-*co*-lactide), nonwoven membranes. They studied the control of structure, morphology, and mechanical and biodegradation properties of the membranes by poststretching and annealing treatments. They found that the degree of crystallinity within the fibers could be increased with annealing at elevated temperatures, although annealing did not improve the overall molecular orientation. Rather, it was found that the crystal orientation improved significantly when the membrane was mechanically drawn and then annealed.[8] Choi et al. examined changes of morphology and tensile property of electrospun PEI web collected onto rotating drum with the interfiber bonding. The electrospun nanofiber web was thermally treated at 80–240°C for 1 h in a convection oven. Morphologies of the fibers treated at 80–220°C are not changed, but the fibers treated at 240°C show clear interfiber bonding. The electrospun PEI fibers treated at 240°C do not have the solvent and show better thermal stability than the raw PEI. For the untreated web, the stress increases smoothly to the maximum point and then also decreases smoothly. For the heat-treated web, however, the stress increases steeply to the maximum point and then drops rapidly. The steeper tensile behaviors of the web are due to the interfiber bonding. In summary, they stated that the interfiber bonding in the electrospun PEI fiber web makes its physical properties improve and is formed by thermal treatment above its glass transition temperature.[9] Jalili et al. collected uniaxially PAN nanofibers using parallel stripes as a collector. Posttreatments of nanofibers were carried out in boiling water (about 94°C) for 10 min with the bundle under tension and then were dried at 110°C. Post treated bundle had CI (crystallization index) of 17.4% much higher than CI of 8.8% for untreated bundle. The bundle became much stronger but relatively with lower elongation after the post-treatment, which could be attributed to the increase in the degree of crystallinity for post treated samples.[10] Thus, it seems that the production of nanofibers at aligned form and heat treating them can improve mechanical properties of collected nanofibers.

This work focuses on the preparation of aligned and heat treated PAN nanofibers at the same time using an unconventional approach. Various treatment temperatures based on differential scanning calorimetry (DSC) results were examined to study structural and mechanical properties of collected nanofibers. Characterizing techniques such as, scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), wide angle X-ray diffraction (WAXD) and tensile test were utilized to analyze morphology, chemical structure, crystallinity and mechanical behavior of nanofibers with treatment temperature.

## EXPERIMENTAL

### Materials

Industrial polyacrylonitrile (PAN) and highly pure dimethylformamide (DMF, 99%) were respectively supplied by Iran Polyacryle and Merck companies. The weight average molecular weight $\overline{M_w}$ and the number average molecular weight $\overline{M_n}$ of the received PAN were $\overline{M_w}$ = 100,000 g mol$^{-1}$ and $\overline{M_n}$ = 70,000 g mol$^{-1}$. The concentration of 14 wt % PAN/DMF solution was prepared using constant power magnetic stirrer at room temperature for 1 h.

### Electrospinning setup

Electrospinning apparatus consists of a high voltage power supply, two syringe pumps, two stainless steel needles (0.7 mm OD) and a rotating collector equipped to an inverter for controlling linear speed. In this setup unlike the conventional technique, two needles were installed in opposite direction and polymer solutions were pumped to needles by two syringe infusion pumps with same injection rate.[11] The flow rate of solutions to the needle tip is maintained constant so that a pendant drop remains during electrospinning. When a high voltage is applied to the needles with opposite voltage and the electric field exceeds a critical value, the electrostatic force will overcome the surface tension of the polymer solution, causing a thin jet ejection from the each needle tip simultaneously. Then the ejected jets under a chaotic motion with opposite charges attract each other, stick together and form a cluster of fibers in the middle part of two syringe needles. This cluster of fibers is neutral as a whole and it will not be attracted by either needle. For collecting nanofibers, the cluster of fibers was towed manually to the rotating collector by a nonconductor crotchety rod. After towing, the fibers are drawn by the mechanical force caused by rotating drum and aligned nanofibers begin to form. The practical conditions used to prepare PAN nanofibers were as follow: The applied voltage was 11 kV. Take-up speed and feeding rate were 1 m s$^{-1}$ and 0.293 mL h$^{-1}$, respectively. The distance between two needles was 13 cm and the distance between needles and collector was 20 cm. The concentration of applied solution was 14% (wt %). These are optimized conditions based on high productivity and molecular orientation that were obtained at previous work.[12]

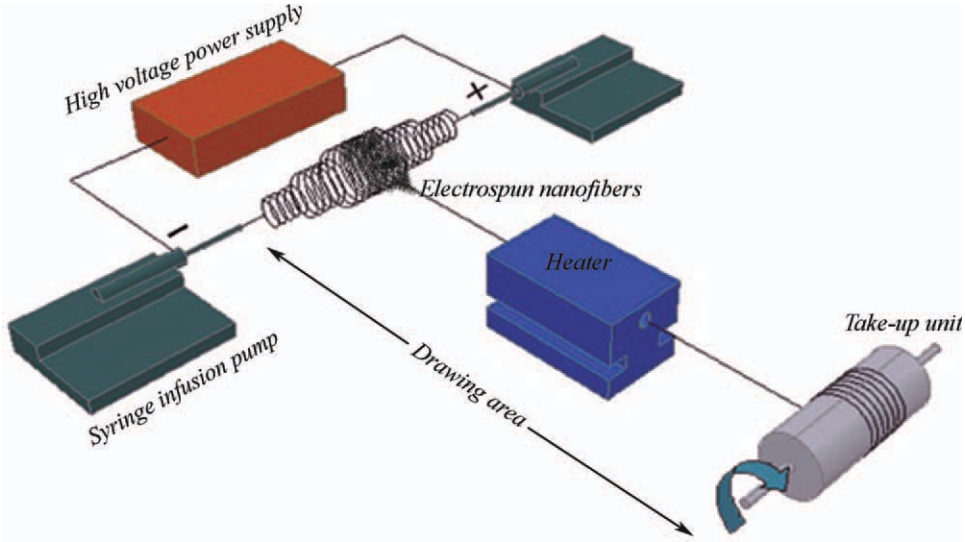


**Figure 1** Schematic of electrospinning setup for collecting aligned heat treated nanofibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

### Dry-heat treatment

Nanofibers were dry-heated and simultaneously collected on a rotating drum to prepare aligned thermal treated nanofibers. A tubular electric heater with a temperature controller, equipped with thermal sensor and entrance power changer and located between the syringe needles and the collector, was used for heating nanofibers passing through thermal zone (Fig. 1). For collecting treated nanofibers, after the electric heater was set at desirable temperature, the cluster of fibers formed between two needles caused by applying high voltage, was pulled manually through the heater and connected to the rotating collector. The rotation of drum lead to aligning heat treated nanofibers continuously. The distance between heater and syringe needles was 8 cm. After heat treated in dry air and aligned on the collector, nanofibers were cooled at room temperature. The PAN nanofibers were spun at 11 kV onto a rotating drum with linear speed of $\sim 1$ m $s^{-1}$.

### Microscopy

The morphology of electrospun nanofibers was investigated using a SERON, AIS-2100 scanning electron microscope (SEM). Nanofiber samples were mounted onto SEM plates; sputter coated with gold and inserted in SEM vacuum vessel for image capturing. The diameters of collected nanofibers in various temperatures were quantitatively determined from their SEM images with high magnification (10,000×). The average fiber diameters were acquired by considering 100 random spots on fibers for each sample.

### Differential scanning calorimetry (DSC)

DSC curves of electrospun fibers were obtained using a DSC 2010V4/4E by heating from 30 to 300°C at a heating rate of 10°C $min^{-1}$. The desired values of temperature for characterizing thermal treated nanofibers were acquired by DSC curve of prepared PAN nanofibers at room temperature.

### FTIR

Dried, electrospun fiber bundles were studied using a Bomen MB-Series100 infrared spectrometer (FTIR) to examine chemical structure of untreated and treated PAN nanofibers. FTIR spectra were recorded over the range of 400–4000 $cm^{-1}$ with 21 scans $min^{-1}$.

### X-ray diffraction (WAXD)

Wide angle X-ray diffraction (WAXD) patterns were obtained for bundles of electrospun nanofibers on a Philips system using Ni-filtered Cu Kα (wave length, $\lambda = 1.5405$ Å) radiation. The diffraction scans were collected at $2\theta = 10°$–$40°$ and a background were subtracted. Calculation of crystallinity index (CI) was done according to the extrapolation of crystalline and amorphous part of the diffraction pattern.[13]

### Mechanical testing

The mechanical behavior of bundles of aligned PAN nanofibers was examined using the Zwick 1446-60 with a crosshead speed of 60 mm $min^{-1}$ and gauge length of 25 mm under standard conditions. Then initial modulus, stress and strain of samples were

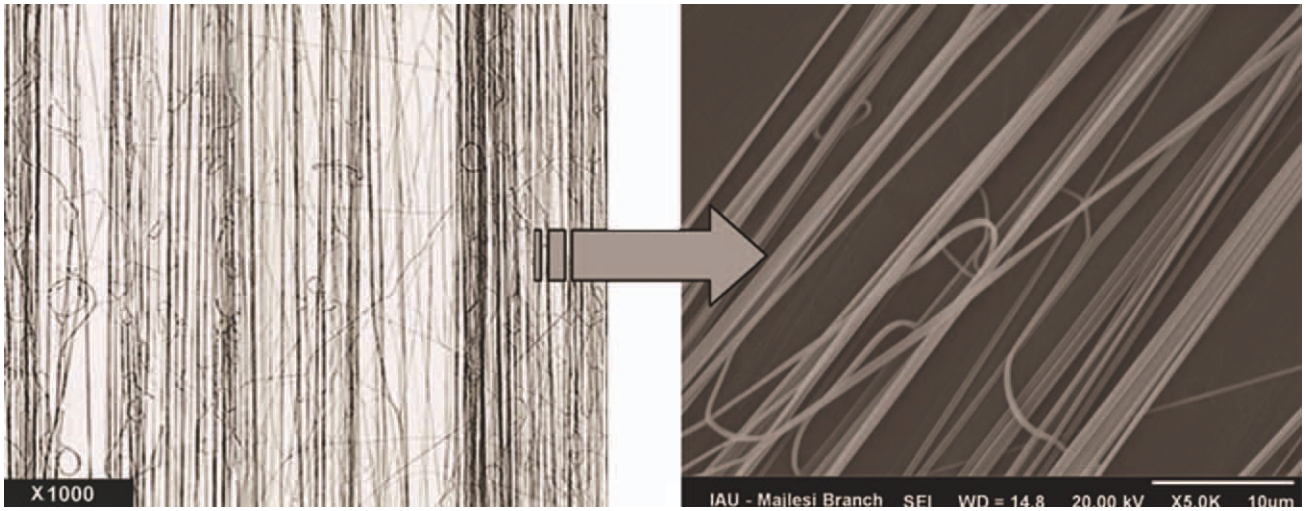


**Figure 2** Unidirectional bundles of aligned PAN nanofibers collected on rotating drum. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

determined. All samples were included in standard container during 24 h under temperature of 25°C and relative humidity of 65% before tested. The numbers of 20 specimens were considered for testing and analyzing of mechanical properties of nanofiber bundles at various temperatures.

## RESULTS AND DISCUSSION

Aligned PAN nanofibers with considerable lengths were electrospun using a simple and unconventional method. Typical images of nanofibers prepared at 140° are shown in Figure 2. In this set up, it can be produce aligned and simultaneously heat treated nanofibers by placing a thermal zone in drawing area. This process has advantages such as coinciding heat treatment and collection of nanofibers, while removing additional process for treatment and preparing mechanically improved nanofibers.[10]

### Heat treatment temperatures

DSC curve of PAN nanofiber collected at room temperature was illustrated in Figure 3. The first peak appeared at low temperature (~ 99°C) and high exothermic peak at ~ 298°C are in relation to glass transition in amorphous regions and occurrence of dehydrogenation and cyclization reactions, respectively.[5,8,14] Thus, the temperatures of 140, 180, 200, 240, and 270°C were selected for heat treatment of nanofibers at above glass transition temperature ($T_g$).

### Morphology

Figure 4 shows SEM images of PAN nanofibers electrospun at different temperatures. The generated nanofibers have uniform structure without any bead. As SEM images exhibit, the fibers were tightly clustered together. This phenomenon was often observed in the treated specimens at higher temperatures. It is probably due to rapid evaporation of solvent with increasing temperature that facilitates to form sticky together nanofibers under drawing force. Based on SEM images, the average diameter of treated nanofibers at higher temperatures (>200°C) shows decreasing trend (Fig. 5). The least average diameter (295 ± 24 nm) of PAN nanofibers was acquired at temperature of 270°C which is about 19% less than the average diameter of untreated nanofibers. The treated nanofibers at 270°C also display more uniform diameter.

These results may be due to the interactive effects of increasing evaporation rate of solvent and improving polymer molecules mobility with temperature. The rapid evaporation of solvent as a facilitative agent for stretching viscose flow has unfavorable effect on

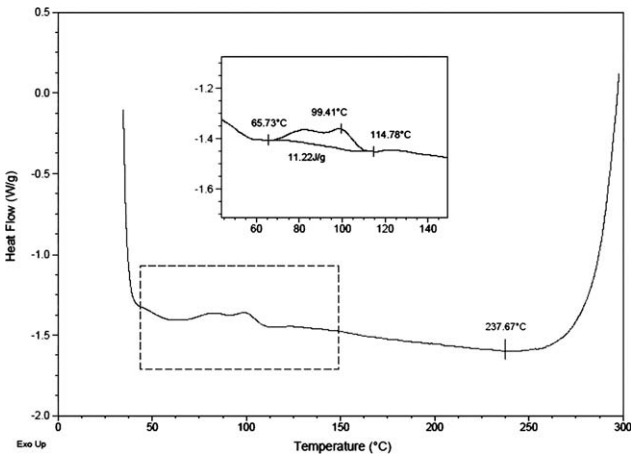


**Figure 3** DSC curve of PAN nanofibers collected at room temperature in practical conditions.

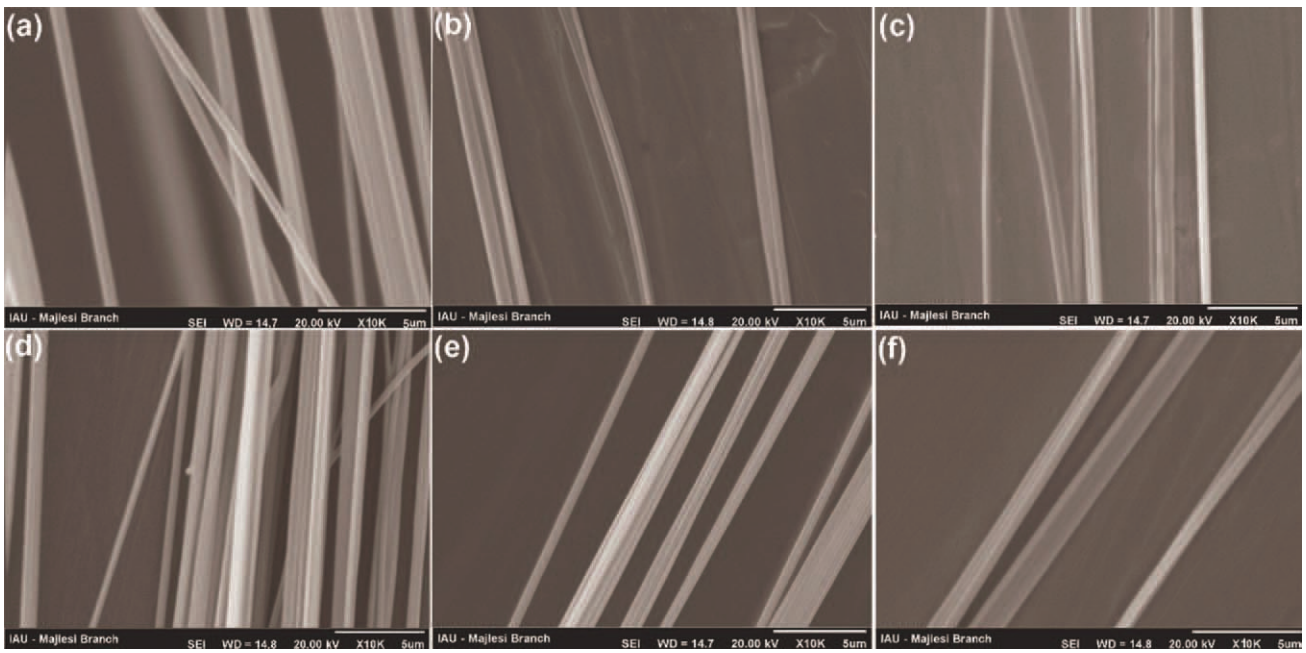


**Figure 4** SEM images of collected PAN nanofibers at (a) room temperature, (b) 140°C, (c) 180°C, (d) 200°C, (e) 240°C, and (f) 270°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

elongating fibers and as a result decreasing diameter of nanofibers by raising temperature. On the other hand, increasing segmental mobility of polymer chains at over $T_g$ temperature allows the tensile force to stretch fiber flow further. According to Figure 4, the average diameters of PAN nanofibers have no change up to the temperature of 200°C because of counteracting above two mentioned parameters which repulse their effects. But, samples prepared at temperatures of 240 and 270°C, really higher than boiling point of DMF (~ 153°C), presented decreased diameters that are probably due to the dominant role of the latter parameter on fiber diameter and more softening of nanofibers at higher temperatures.

## FTIR characterization

FTIR spectra of PAN nanofibers collected in practical conditions at different temperature were presented in Figure 6. Spectra for both untreated and heat treated nanofibers show prominent peaks at 2940 $cm^{-1}$ (CH stretch), 2240 $cm^{-1}$ (CN stretch), and 1451 $cm^{-1}$ ($CH_2$ bend). The absorption peaks at 1730 and 1667 $cm^{-1}$ are due to the C=O stretch vibrations of the aliphatic ketone and the conjugated ketone, respectively.[8,15]

A major decrease in the nitrile stretching at 2240 $cm^{-1}$ and methylene band at 2940 $cm^{-1}$, and a growth of new bands at 2200 $cm^{-1}$ (NC—C= group), 1595 $cm^{-1}$ (C=C, C=N stretch or a combination of two), and 810 $cm^{-1}$ (C=C—H group in the aromatic ring) referring to dehydrogenation and cyclization reactions, are evidences for changed chemical structure of PAN fibers during heat treatment.[8,14,15] In the examination of FTIR spectra of collected PAN nanofibers at various temperatures did not observe the appearance of new peaks (2200, 1595, and 810 $cm^{-1}$) and significant decrease at nitrile stretching and methylene band. Therefore, as mentioned above, heat treatment has no considerable effect on chemical structure of treated PAN nanofibers. This may be attributed to low required time for varying chemical structure of PAN nanofibers in consequence of heating them.

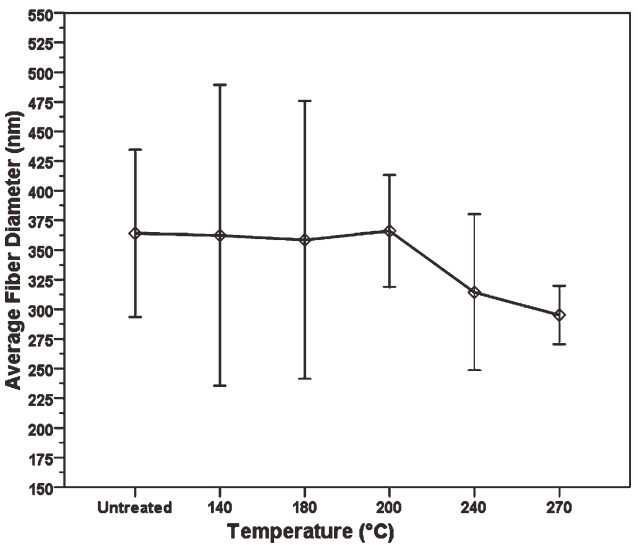


**Figure 5** The average diameter of PAN nanofibers versus treatment temperature.

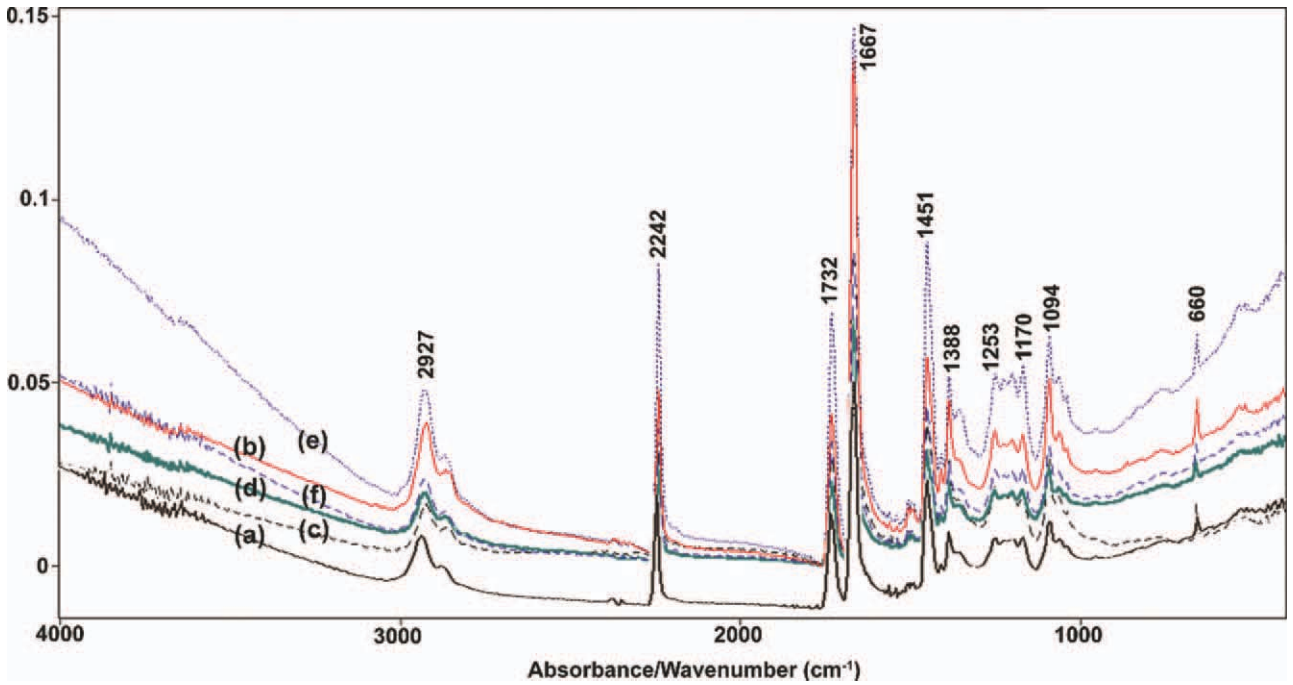


**Figure 6** FTIR spectra of prepared PAN nanofibers at various temperatures (a) room temperature, (b) 140°C, (c) 180°C, (d) 200°C, (e) 240°C, (f) 270°C. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

### X-ray characterizations

Wide angle X-ray diffraction (WAXD) patterns were obtained from bundles of fibers electrospun from a 14 wt % PAN in DMF solution at 11 kV collected onto a target rotating with a surface velocity of $\sim 1$ m $s^{-1}$ at various temperatures. The diffraction patterns of all samples show two main equatorial peaks, a weak peak at $2\theta = 28.5°$ corresponding to a spacing of $d \approx 3.1$ Å from the (110) reflection and a strong peak at $2\theta = 17°$ corresponding to a spacing of $d \approx 5.2$ Å from the (100) reflection. The equatorial peaks at $2\theta = 28.5°$ and $2\theta = 17°$ are common to the fiber diffraction pattern of PAN with hexagonal packing.[3,10,14,15] A diffuse meridional peak at $2\theta = 38.6°$ ($d \approx 2.33$ Å) and a diffuse off-equatorial peak at $2\theta = 25.7°$ ($d \approx 3.46$ Å) were also observed in more collected WAXD patterns, as has been reported previously for PAN fibers.[15,16] Examples of WAXD patterns were illustrated in Figure 7.

Heating PAN fibers at over $T_g$ temperature results in releasing more and more nitrile groups from their bound state due to dipole–dipole interaction of the nitrile groups and increasing segmental mobility of molecular chains.[8,13] By providing sufficient time for polymeric chains, because of the motion of molecular chains in amorphous regains, some smaller crystals might be disturbed while other larger crystals were formed so that the crystallinity of fibers was improved.[6,7,13] The degree of crystallinity measured from WAXD patterns of PAN nanofibers has been slightly enhanced with treatment temperature (Table I). Although, the crystallization index of heat treated PAN nanofibers at 270°C is 18% higher than that of untreated nanofibers, however heat treatment has not considerably affected crystallinity of aligned PAN nanofibers. As explained above, this is probably due to low time of heating nanofibers moving through heater and as a result, lack of enough opportunity for organizing molecular chains in ordered crystalline structure. Moreover, rapid evaporation of DMF solvent as a facilitative agent for arranging molecular chains into crystal structure may be impressed the crystallinity of collected nanofibers at higher temperatures.

### Mechanical properties of treated and untreated PAN nanofibers

Unidirectional bundles of PAN nanofibers were prepared in functional situations with a temperature ranging from 25 to 270°C. The average linear density of the bundles was about 140 den. The stress–strain behavior of the bundles was examined and the initial modulus, tenacity and extension were measured as a function of temperature (Fig. 8). Tenacity values increases gradually from 72.5 to 113 MPa at 25–270°C, respectively. The extension values followed an inverse trend; it decreased from 19.2 to 9% with

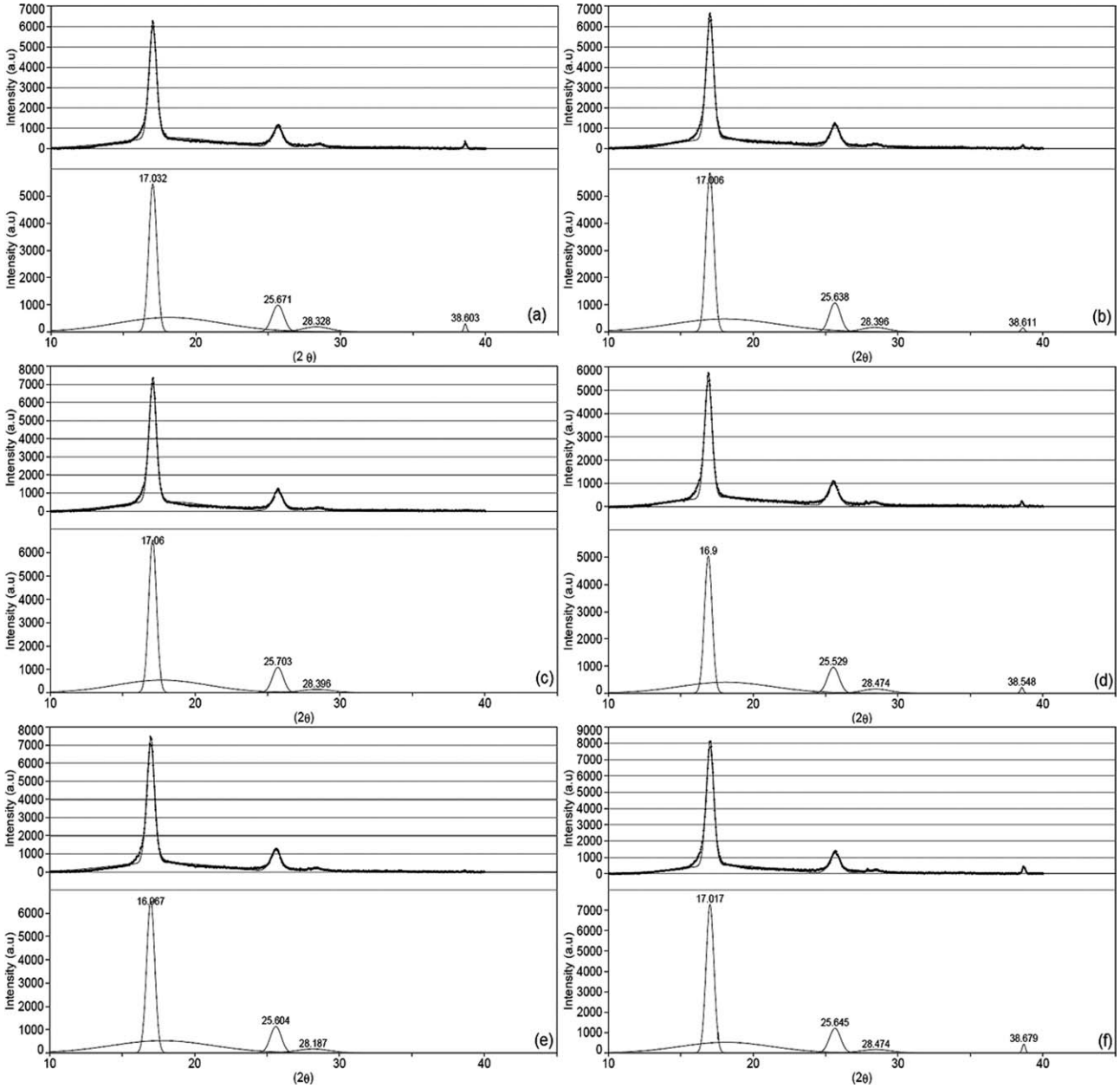


**Figure 7** WAXD patterns of PAN nanofiber bundles at different temperatures (a) untreated, (b) 140°C, (c) 180°C, (d) 200°C, (e) 240°C, (f) 270°C.

increasing temperature from 25 to 270°C, respectively. However, the initial modulus of the bundles does not show regular trend with increasing temperature, but heat treated nanofibers exhibit more modulus than untreated nanofibers. The most value of modulus was obtained 7.2GPa for treated nanofibers at 270°C in comparing with the modulus of 4.1 GPa for untreated nanofibers.

Table II shows the Pearson correlation factors acquired by SPSS statistic software. Statistically, the negative Pearson correlation factor of -0.87 between stress and average fiber diameter demonstrates inverse relation of them which is significant at the 0.05 level. This means that the stress of nanofiber bundles increases with decreasing fiber diameter. As regards SEM images (Fig. 4), it can be offered that

**TABLE I**
**Degree of Crystallinity Measured for Untreated and Treated PAN Nanofibers**

| Treatment temperature | Untreated | 140°C | 180°C | 200°C | 240°C | 270°C |
|---|---|---|---|---|---|---|
| Degree of crystallinity (%) | 52.2 | 55.5 | 57.7 | 59.1 | 57.2 | 61.7 |

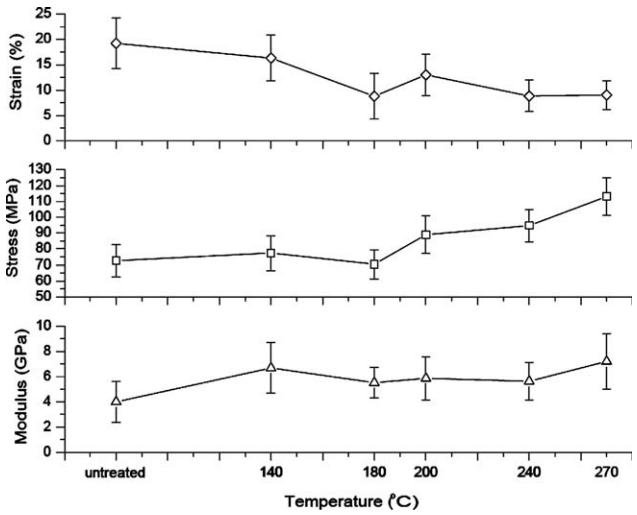


**Figure 8** Stress, strain and initial modulus of prepared PAN nanofiber bundles versus treatment temperature.

decreasing fiber diameter with temperature lead to increasing surface area to volume ratio of fibers and as a result the more clustering of nanofibers at tightly aligned and sticky form under drawing force. In this situation, the high numbers of fiber conjointly resist on an applied tension in the direction of fiber axis.

The measured stress and crystallization index of PAN nanofibers have the positive Pearson correlation of 0.77. This is the evidence for direct relation of them, but low amount of this coefficient does not suggest high linear correlation. Thus, it may be stated that the crystallinity performs some positive effect in response of mechanical properties and particularly stress of PAN nanofibers. Nanofibers heat treated at temperature of 270°C have the highest value of stress and crystallinity and the lowest amount of average fiber diameter.

Fiber shrinkage occurs during thermal treatment of PAN fibers due to provided kinetic conditions for molecules to relax strain acquired by stretching during spinning at over $T_g$ temperature and also, chemical reaction (cyclization and dehydrogenation) at high temperature which can disturb orientation of molecular chains and causes poor mechanical properties of final fiber.[8,13] In this study, the possibility of shrinkage happening is mainly low for treated PAN nanofibers, because of occurring no chemical reaction during thermal treatment of nanofibers (Fig. 6) and collecting nanofibers under tension caused by rotating drum at take up speed of $\sim 1$ m s$^{-1}$. Therefore, absence of shrinkage phenomenon in treated PAN nanofibers had prominent effect on the resulted mechanical properties.

Additionally, in the process of heat treatment with tension, the molecular chain is apt to orient in the direction of applied force, i.e., direction of fiber axis. The higher the temperature and the greater the applied force, the higher the molecular chain orientation, resulting in higher tenacity and modulus for the fibers.[6,7,8,13] Whereas nanofibers were collected under tension and heated at over $T_g$ temperature in present work and by considering the strong and direct relation obtained between stress and molecular orientation parameter at previous study,[12] it seems that the improvement of molecular chain orientation in the direction of nanofiber axis has had positive role in the attained mechanical behavior of treated PAN nanofibers.

**TABLE II**
**The Pearson Correlation Among Stress, Average Fiber Diameter, and Crystallization Index**

| | | Diameter | Crystallization index |
|---|---|---|---|
| Stress | Pearson correlation | −0.872[a] | 0.769 |
| | Sig. (two-tailed) | 0.023 | 0.074 |
| | *N* | 6 | 6 |

[a] Correlation is significant at the 0.05 level (two-tailed).

## CONCLUSION

The process of electrospinning and heat treatment of PAN nanofibers were carried out at the same time using two syringe needles in opposite positions. A rotating drum perpendicular to needle axis and a heater was located between the needles and the collector. PAN nanofibers were electrospun from 14 wt % solutions of PAN in DMF at 11 kV onto a rotating drum with surface speed of $\sim 1$ m s$^{-1}$. DSC curve of collected PAN nanofibers at room temperature was examined to choose appropriate treatment temperatures. Various characterization techniques were employed to find out the influence of temperature on the average fiber diameter, chemical structure, degree of crystallization and mechanical properties of collected PAN nanofibers. Based on micrographs captured by SEM, the average diameter of treated nanofibers at over temperature of 200°C obtained lower than the other samples. Investigation of FTIR spectra shows no evidence with respect to occurring dehydrogenation and cyclization reactions during heat treatment. The best degree of crystallinity was obtained for those of nanofibers collected at temperature of 270°C. In the case of mechanical properties, stress and strain exhibit increasing and decreasing modes, respectively with the rising of treatment temperature, especially at temperatures higher than 200°C. Increased modulus of treated nanofibers at over $T_g$ temperature can be concluded by comparing the modulus of untreated and treated specimens.

The stress and modulus of treated PAN nanofibers at 270°C are 56 and 76% higher than untreated nanofibers, respectively.

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