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Enhancing the mechanical features of poly(vinyl alcohol) nanofibers with the addition of boron nitride

Hazal Gergeroğlu[1*], Neslihan Sakar[2], Hamed Ghorbanpoor[3], Çağlar Özer[4]

[1]Dokuz Eylul University, Department of Nanoscience and Nanoengineering, 35340, Izmir, Turkey,
ORCID ID orcid.org/0000-0001-8369-9221
[2]Dokuz Eylul University, Department of Nanoscience and Nanoengineering, 35340, Izmir, Turkey,
ORCID ID orcid.org/0000-0002-9541-194X
[3]Ankara Yildirim Beyazit University, Department of Biomedical Engineering, 06010, Ankara, Turkey,
ORCID ID orcid.org/ 0000-0002-2665-8172
[4]Dokuz Eylul University, Department of Nanoscience and Nanoengineering, 35340, Izmir, Turkey,
ORCID ID orcid.org/ 0000-0002-8127-741X

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ABSTRACT

Improvement of nano-sized products with boron additives with remarkable properties has become a popular trend for many different application areas. Here, we have reported an efficient poly(vinyl alcohol) (PVA) based composite nanofiber which was functionalized with the hexagonal boron nitride (h-BN) via electrospinning technique, for the first time as far as known, for body-armor and protective clothing applications. PVA-based composite nanofibers containing h-BN with a diameter of approximately 326 nm have shown significant mechanical features compared to pure PVA nanofibers with a diameter of about 223 nm. Mechanical features were investigated by nanoindentation and the results of nanoindentation demonstrated that the elastic modulus of PVA nanofibers increased by 77%, and hardness values of polymeric nanofibers reached ten times more, by the addition of h-BN. FTIR and XRD characterizations approved that there is no chemical change of h-BN when it was integrated into the PVA based composite nanofibers. We exceptionally believe that the achieved results offer a potential lightness and cost-effective strategy for body armor and protective clothing applications in contrast to boron carbide-based body armor products, which have main problems such as weightiness and high-priced.

1. Introduction

Recently, the focus has been on the development of boron-based high-tech products, and the expansion of boron-based materials and additives [1-4]. In this sense, when advanced boron compounds are examined, nowadays boron nitride (BN), which offers a wide range of application potential and has outstanding properties, is remarkable [4,5]. Structurally like the carbon system, BNs are synthetic materials that, although discovered in the19th century, did not develop into a commercial material until the 20th century. It is mostly found in hexagonal (h-BN), cubic (c-BN), and amorphous structures [5,6]. h-BN has many different applications in engineering and refractory fields such as catalysis, contaminant removal in very harsh environments, and hydrogen storage due to its extraordinary features such as high resistance to acidic chemical corrosion, non-toxic, refractory features in vacuum up to 2000°C, lightweight, workability, high electrical resistance, and good thermal conductivity [4,6,7].

On the other hand, the nanostructure chemical composition refers to the order of atoms (atomic structure) and the dimension of a solid in one, two, or three. Thus, nanostructures have chemical and physical features that are characteristic of neither atomic nor mass equivalents [8]. Therefore, different studies have been realized for the production and characterization of h-BN in its forms such as nanotubes [9-11], fullerenes [12], nano-cones [13], nano-capsules [14,15], and nanoparticles [16,17] and the superior features of h-BN and the advantages of nanotechnology have been tried to be combined. Recently, limited studies have been published on the BN using PVA as a matrix. For example, Wang et al (2019) investigated the mechanical features and microstructures of PVA nanocomposites reinforced with boron nitride nanotubes (BNNTs). The authors claimed that BNNTs have excellent potential to design composite nanofillers in many applications [18]. Li et al. (2018) reported that, compared with pure PVA hydrogel, the thermal stability, mechanical features, and swelling behavior of BN

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*Corresponding author: hazal.gergeroglu@ogr.deu.edu.tr nanosphere/PVA composite hydrogel are effectively enhanced [19]. Zhang et al. (2017) reported that BN-modified PVA aerogels can be successfully produced via the cheap frozen-drying method to use in environmental remediation applications [20]. Additionally, Zhang et al. (2018) showed that cellulose nanocrystals and boron nitride nanosheets enhanced the thermal conductivity, and the mechanical features of the PVA [21].

Among the reported h-BN nanostructures, the one-dimensional shape (1D) of h-BN (ie nanotubes, nanofibers) offers high performance in potential electronic and mechanical devices due to its axial ratio. Since the axial ratio plays a crucial role in the physical and chemical features of the 1D structures, it is desirable to being long in the nanostructures. Compared to other nanostructures, the longest axial ratio is found in nanofibers [7]. Therefore, many researchers have tried to produce h-BN nanofibers using complex procedures. The reported chemical synthesis methods are complex, it is very difficult to control the final product to be in the 1D form [7,8]. However, the electrospinning technique is renowned for its easy control of the size of the final product [22-24]. In this method, an electric field is used to produce fibers from polymers, composites, and ceramics to ultra-fine micro and nano scales, and nanofibers are produced efficiently [25]. Therefore, especially in the last decade, there are many studies investigating the production of h-BN nanofiber structures via the electrospinning technique, characterization of these structures, and indicating their potential in various application areas. For instance, multifunctional h-BN nanosheet/ poly(vinyl alcohol) (PVA) nanofiber membranes for use in electronic packaging and water treatment applications [26]; h-BN/poly (p-caprolactone) (PCL) composite nanofibers [27], and h-BN nanofibers [28] for aerospace and high-temperature filtration applications; h-BN/polyacrylonitrile (PAN) composite nanofibers [29] and h-BN/carbon composite nanofibers [30] as high-security low-cost electrolyte and electrode material for lithium-ion batteries applications; PCL, to use in bone-tissue engineering applications tricalcium phosphate (TCP) and h-BN composite nanofibers [31]; high-activity h-BN/titanium dioxide (TiO_2) composite nanofibers [32] as photocatalyst for use in photocatalytic applications have been significant investigation topics. Additionally, it has been stated that the h-BN/polymer composite nanofibers have a significant potential for body armor and protective clothing applications [33].

Body armor and protective clothing are very effective in defense and military forces, especially in reducing soldiers and police injuries and deaths. In today's technology, the armors and protective clothing have designed via considering the futuristic material options. However, academic and industrial studies have focused on reducing the weight of body armor systems and improve the comfort level. Additionally, considering the pricing of standard body armor systems up to several thousand dollars per set, cost reduction is an important factor in the studies. It has been reported that new materials, innovative designs, and lightweight systems that can meet these requirements need to be developed [34]. Although boron carbide is generally one of the strongest candidates for body armor applications, research continues to address basic problems such as cost and lightness [35-37]. Moreover, it is stated that protective clothing and body armors produced via electrospun nanofibers which have multifunctional features such as extremely lightness, cheap producibility, high breathable, and high elasticity can overcome these problems [38].

In this study, h-BN, known to have superior properties mentioned before, has played a role in improving mechanical properties. h-BN was combined then with PVA, which is widely used in body armor applications and the protective clothing industry, via the electrospinning method. Thus, coming together of the superior properties of h-BN with the advantages of nano-size was carried out. Moreover, the effect of adding h-BN on the mechanical features of PVA nanofibers is reported for the first time in the literature. The mechanical analysis was performed via the nano-indentation. Furthermore, resulted composite functionalized nanofibers were investigated for morphology, chemical interactions, and functional groups by performing SEM, XRD, and FTIR characterization.

2. Materials and methods

Poly(vinyl alcohol) (PVA, purity 95,40%) with molecular weight (MW) of (44.0526)n g/mol and 87.16% of hydroxyl groups was obtained from Zag Chemistry. Boron Nitride (BN, purity>99%, particle size 1 µm) was received from Sigma Aldrich. BN and PVA precursors were employed without any further treatment. The solvent was distilled water.

To prepare electrospinning solutions, firstly, an aqueous solution 8% by weight of (wt) of PVA was stirred at room temperature for 24 h. Then, h-BN (8% wt) was dissolved in distilled water at 95°C for 6 h. Aqueous h-BN solution added into pure PVA solution at 60°C via dropping technique and stirred for 2 h. Thus, PVA (pure), and PVA containing h-BN (PVA/BN) electrospinning solutions were prepared. The electrospinning process was performed using a commonly used simple electrospinning apparatus to obtain nanofibers. Nanofibers were obtained from the prepared solutions with the same electrospinning process parameters are applied as flow rate 1 mL/h, collector-syringe distance 20 cm, applied voltage 25 kV.

The morphological characterization of PVA (pure) and PVA/BN nanofibers was carried out by Philips XL 30S FEG scanning electron microscope (SEM) operat-

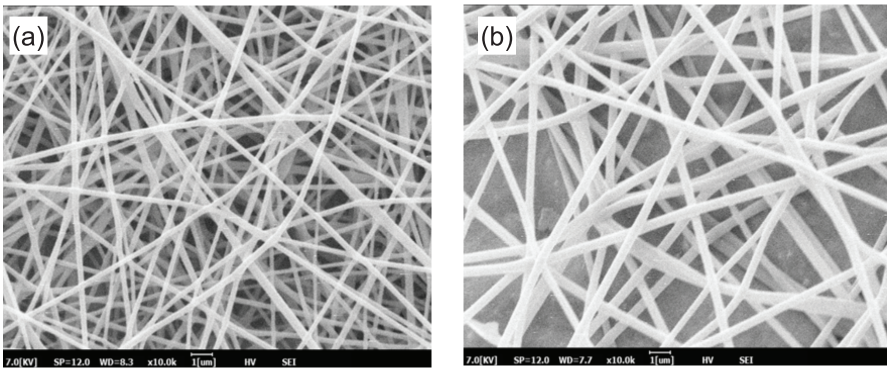

Figure 1. SEM photographs of (a) PVA (pure), (b) PVA/BN nanofibers.

ing at 7 kV voltage. From the SEM images obtained, the average fiber diameter was measured and calculated from 50 different locations using the Image J program. The phase identification and crystal structures of the obtained nanofibers were performed by Thermo Scientific ARL X-ray diffractometer (XRD) with Cu-Kα model (1.5405 Å) operating at 45 kV voltage and 44 mA current values. XRD data were recorded in the range of $10° \leq 2\theta \leq 80°$ in 0.02° steps at a rate of 2°/min. Fourier transform infrared spectroscopy (FTIR) studies were performed using (Thermoscientific Nicolet I10) device, with the wavenumber range of 4,000-500 cm^{-1}, to evaluate whether or not h-BN is integrated into the PVA structure without any chemical changes. Mechanical analysis of nanofibers was performed with IBIS DME Tools DS 95-50. The diamond conical surface with a half-tip angle of 70.3° was used to model the commonly used Berkovich indenter. Also, the thicknesses of PVA (pure) and PVA/BN nanofibers were several micrometers. 1 millinewton (mN) force was applied to five different locations for both samples and the penetration depth (μm) was measured. Nanoindentation load-penetration depth curves were obtained, and elastic modulus (E) and hardness values were calculated.

3. Results and discussion

The morphological structure and average fiber diameter were investigated by SEM. Figure 1 indicates that the average nanofiber diameter increases with the addition of BN. The average fiber diameter, which is measured at 50 different locations using the Image J program, is 223±50 nm for PVA (Figure 1a) and 326±72 nm for PVA/BN (Figure 1b). Although no change was made in the electrospinning process parameters, the diameter and standard deviation of the composite nanofibers increased by the addition of BN. The most important cause is probably integration of the BN into the fibers, and it is very significant evidence for the composite fibers which contain the h-BN.

BN is a chemically inactive ceramic material. Therefore, a chemical reaction between h-BN micron-size particles and PVA aqueous solution cannot be expected [16]. However, XRD analysis was performed to ensure that the h-BN micron particles used in this study were integrated into the PVA structure without any chemical reactions. Additionally, the effect of the electrospinning process was investigated by XRD characterization of initial PVA and BN used in nanofiber production. It has understood that the XRD pattern of the initial PVA indicates the amorphous structure whereas the PVA pure (pink graph) nanofibers produced via electrospinning shows the semi-crystalline structure (Figure 2) with two main peaks at around $2\theta \approx 14°$ and $2\theta \approx 17°$, respectively. Moreover, the initial h-BN pattern in Figure 2, indicates the typical peaks of h-BN with defined, intense peaks at $2\theta = 26.76°$ and $2\theta = 41.46°$ related to the (002) and (100) reflection (ICDD-PDF Card: 00-034-0421), respectively. The main peaks of the BN ceramics are visible in the XRD pattern of PVA/BN nanofibers, but not in the XRD pattern of PVA (pure) nanofibers. However, the fact that a specific peak in the diffraction pattern of PVA is also visible in the PVA/BN nanofiber pattern means that each component shows its own diffraction region. If there were any interactions, these peaks could be seen nested [39]. Therefore, it has been shown that BN is integrated into the PVA matrix without any chemical reaction.

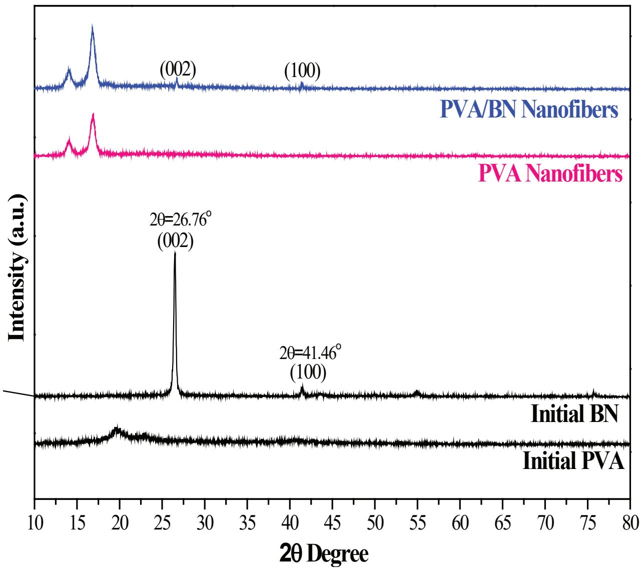

Figure 2. XRD patterns of the initial PVA, BN (Initial), PVA (pure), and PVA/BN nanofibers

As a result, it was considered in the XRD analysis that BN was not chemically affected by the electrospinning process. Although XRD is a highly reliable method of analysis for crystalline materials, the same may not apply to amorphous, semi-crystalline materials [16]. Due to the polymer used (PVA), FTIR characterizations were performed in addition to XRD studies. In the FTIR spectra (Figure 3a), the B-N bond represents two peaks of h-BN located at 767 and 1317 cm^{-1}, respectively, while the B-H bond corresponds to the two bands at 2331 and 2535 cm^{-1} [20]. Figure 3b shows that the main peaks of PVA (pure) nanofibers were observed at 845, 1093, 1260, 1434, 1734, 2930, and 3290 cm^{-1}. These peaks are appointed to the C-C stretching vibration, C-O stretching of acetyl groups, C-H deformation vibration, C-H bending vibration of CH_2, C=O carbonyl stretch, CH_2 asymmetric stretching vibration, and the O-H stretching vibration of the hydroxy group, accordingly [40]. For the PVA/BN nanofibers, similar peaks were observed (Figure 3c). The addition of BN is assigned to small shifts in the main peaks. Moreover, the FTIR spectra of PVA/BN confirms that the h-BN structure. In Figure 3 (blue curve), due to the B-N and BN-O bonds, which are attributed to the h-BN and B-OH bonds, respectively, peaks are observed at 804, 1088, 1374, and 3306 cm^{-1} [32,41]. Consequently, it has been demonstrated that BN has been successfully integrated without any chemical change in the structure of PVA.

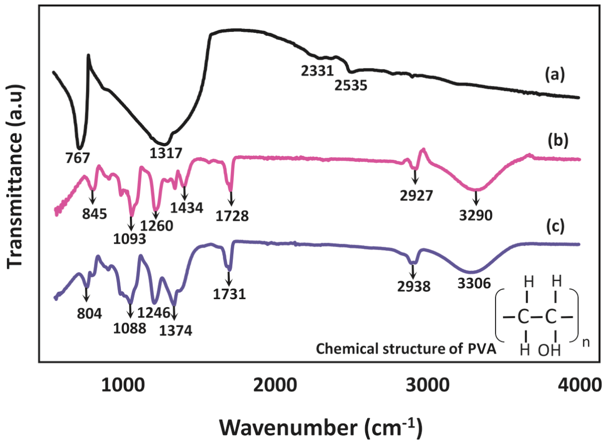

Figure 3. The FTIR spectra of (a) BN (Initial), (b) PVA (pure), (b) PVA/BN nanofibers.

To investigate the mechanical effect of BN integration into PVA nanofibers, nano-indentation tests were performed. For both samples (PVA and PVA/BN nanofibers), 1 mN of force was applied to five different locations and the penetration depth was measured. Thus, nanoindentation load- penetration depth curves were obtained (Figure 4). Comparing the load penetration depth curves of PVA (pure) nanofibers (Figure 4a) with the curves of PVA/BN nanofibers (Figure 4b), it is clear that PVA (pure) nanofibers have a deeper penetration depth to the surface under the same load. This means that the strength and hardness of PVA/BN nanofibers are higher than that of PVA (pure) nanofibers.

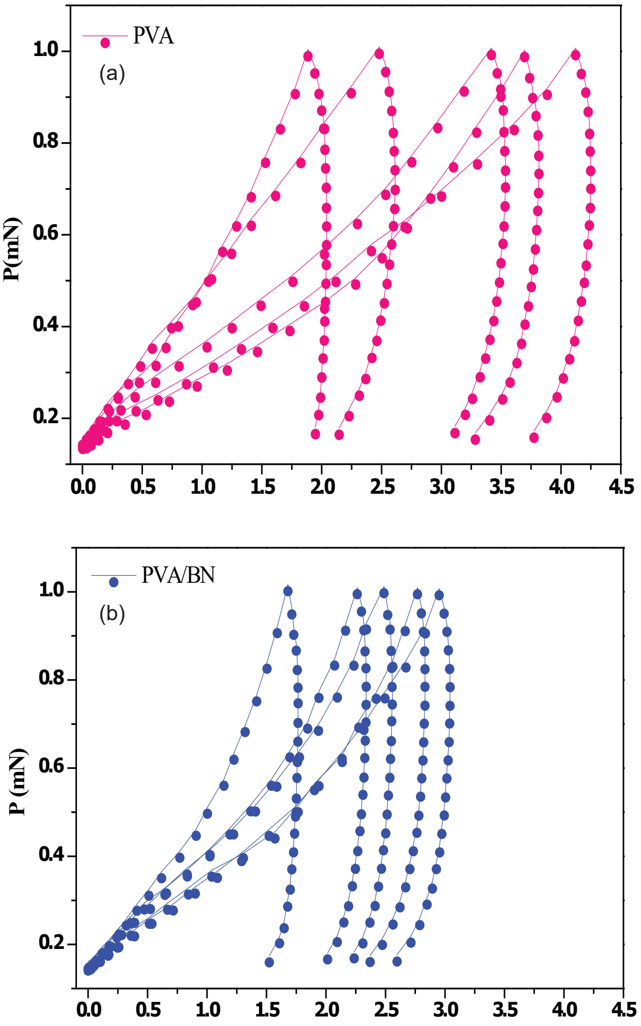

Figure 4. Nanoindentation load-penetration depth curves; (a) PVA (pink), (b) PVA/BN (blue) nanofibers.

To confirm the results obtained from the graphs, the elastic modulus (E) was calculated from the tilt of the first part of the unloading curve (S) (Figure 4) using Eq (1) and (2) [42,43].

$$S = 2\beta\sqrt{\frac{A}{\pi}}E_r \qquad (1)$$

In (1), A is the projected area of the indentation, b is a constant dependent on the geometry of the indenter, and E_r is the reduced elastic modulus given by,

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \qquad (2)$$

where ν and E are the Poisson's ratio and elastic modulus for the sample and ν_i and E_i are equal quantities for the indenter, respectively. On the other hand, in nanoindentation, hardness (H) is determined as the average contact pressure (P_{max}) and is acquired by dividing the peak force by the projected area (A) of the contact between the sample and tip using Eq(3) [44,45]. The values obtained from all calculations are shown in Table 1.

$$H = \frac{P_{max}}{A} \quad (3)$$

Results of calculations (Table 1) were confirmed the obtained nanoindentation load versus penetration depth curves (Figure 4). The existence of h-BN leads to increasing in E from 134 MPa to 238 MPa. Furthermore, the hardness values were calculated as 7.5 and 86.2 MPa for PVA and PVA/BN nanofibers, respectively (Table 1). Since the electrospinning process parameters for both of them were the same, the comparison indicates that the improvements in mechanical features were due to h-BN addition. Thus, it can be claimed that the critical enhancement in the elastic modulus and hardness of PVA nanofibers with the integration of h-BN is presumably due to as follows: (i) homogeneous distribution of h-BN additives in the PVA matrix via the electrospinning process, (ii) considerable increase in crystallinity of the PVA matrix, (iii) effective transfer of the load due to the intermolecular H-bond between the PVA matrix and h-BN, which is a powerful interaction that limits the chain mobility of the PVA.

Table 1. Results of nano-indentation.

Sample	Force (mN)	Elastic Modulus (MPa)	Indentation Hardness (MPa)
PVA	1	134	7.5
PVA/BN	1	238	86.2

Moreover, the increase in the elastic modulus and hardness values of PVA/BN composite nanofibers because of the existence of h-BN was similarly reported in PVA/BN nanocomposite films performed in previous studies [46-48]. For instance, Sreedhara et al., in 2018, with the addition of 0.2% wt h-BN sheets into PVA films, have reported that the elastic module and the hardness values increased from 4.3 GPa to 7.3 GPa, and from 76.7 MPa to 109.2 MPa, respectively [47]. In another study, it has been shown that the elastic modulus increased from 8.6 GPa to 12.9 GPa and the hardness value increased from 0.25 GPa to 0.6 GPa by the addition of two different sizes (0.5 and 1 μm) of BN nanoplate into PVA/tannic acid (TA) films [46]. However, according to our best knowledge, for the first time, the mechanical features of PVA/BN nanofibers and the mechanical effect of the existence of h-BN into the PVA nanofibers structure were investigated in this work. Furthermore, this study focused on the contribution of h-BN to improving the mechanical features of the PVA nanofibers with a uniform fiber morphology. Therefore, its use at the maximum possible concentration has been investigated. However, the concentration of additives that can be incorporated into the polymer in the electrospinning method is limited because when the optimum amount is exceeded, it makes production conditions difficult and bead structures may be formed in the fibers. In this case, the homogeneous fiber structure can be damaged. Also, the distribution of the particles inside the polymeric nanofibers is a significant parameter. Herein, the significant point is that particles tend to stand apart when a small amount of additive material was added, whereas when high amounts of additives are added, the particles tend to aggregate, in which case agglomeration occurs [8]. In light of this knowledge, the main purpose of this study is not only to obtain fibers with reinforced mechanical features but also to obtain a fiber morphology without bead structures. Therefore, the maximum amount of h-BN that we can use without damaging fiber morphology and obtain maximum improvement in mechanical features has been determined as 8% by weight.

4. Conclusion

h-BN is known to have superior properties such as non-toxic, lightweight, workability, high electrical resistance, and good thermal conductivity. It has been reported in previous studies that the addition of h-BN into the composite film form of PVA, which is widely used in textile and protective clothing applications, improves its mechanical properties [46,47,49,50]. In our study, unlike previous studies, the mechanical features of nanofiber morphology were examined; the elastic modulus and hardness of PVA (pure) nanofibers with the addition of h-BN increased from 134 MPa to 238 MPa and 7.5 MPa to 86.2 MPa, respectively. Moreover, the incorporation of h-BN into PVA nanofibers has been observed to develop a critical improvement in PVA nanofibers, similar to composite PVA/BN films. Thus, a substantial enhancement of the mechanical features can be attributed to the incorporation of h-BN into PVA nanofibers.

After adding the h-BN to the PVA matrix structure, the average diameter of electrospun composite nanofibers increased from approximately 223 nm to about 326 nm, increasing the diameter of the nanofibers probably due to integration of the h-BN into the fibers. Furthermore, this evidence is critically important in addition to FTIR analysis shows that the PVA/BN composite fibers contain the h-BN. Moreover, the h-BN was successfully incorporated into PVA nanofibers without any chemical change based on the FTIR and XRD results.

In this study, it has been shown that PVA/BN composite nanofibers offer high potential in body armor and protective clothing application areas with their lightness, cheapness, and high strength properties. Moreover, this work has suggested a novel approach for the building of cost-effective and light composite nanofiber-based structures that support body armor and protective clothing applications.

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