

FIBER AND METHOD OF FORMING THE SAME

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims the benefit of priority of Singapore application No. 10202201394T filed February 14, 2022, the contents of it being hereby incorporated by reference in its entirety for all purposes.

TECHNICAL FIELD

[0002] Various embodiments of this disclosure may relate to a fiber. Various embodiments of this disclosure may relate to a method of forming a fiber.

BACKGROUND

[0003] Fibers, as the basic building blocks of textiles, have been actively engaged in a wide range of our daily activities, including health management, human-computer interaction, movement monitoring, soft robotics, disease prevention, and more. Research focus is directed on achieving fibers with high mechanical and good electrical performance, with emphasis on conductive materials making up the fibers. These conductive materials range from carbon-based materials, metal-based materials, and conductive polymers-based materials. Amongst all the accessible materials, MXenes, as a new class of two-dimensional (2D) inorganic compounds, are promising materials for the development of fiber-based devices, owing to their combined outstanding mechanical, electrical, and electromagnetic properties. MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets can be prepared into high-performance nanocomposites due to their surface terminated moieties (T_x), such as -OH, -O, and -F. Many studies have been carried out to fabricate MXene fibers based on MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets by various processes, including

wet spinning, coating, electrospinning, and biscrolling method. MXene fibers have therefore been successfully fabricated with the desired electrical conductivity and mechanical properties, such as MXene/reduced graphene oxide (rGO), MXene/cellulose nanofibrils, Kevlar/MXene, and nylon/MXene.

SUMMARY

[0004] Various embodiments may relate to a method of forming a fiber. The method may include forming a wet spun fiber from a plurality of MXene sheets using a wet spinning process. The method may also include forming the fiber by heating the wet spun fiber using a thermal drawing process. The fiber may be a cross-linked MXene-based fiber including a MXene core including the plurality of MXene sheets which are cross-linked. The fiber may further include a protective layer surrounding the MXene core.

[0005] Various embodiments may relate to a fiber. The fiber may include a MXene core. The fiber may include a protective layer surrounding the MXene core. The fiber may be a cross-linked MXene-based fiber such that the MXene core includes a plurality of MXene sheets which are cross-linked.

BRIEF DESCRIPTION OF THE DRAWINGS

[0006] In the drawings, like reference characters generally refer to the same parts throughout the different views. The drawings are not necessarily drawn to scale, emphasis instead generally being placed upon illustrating the principles of various embodiments. In the following description, various embodiments of the invention are described with reference to the following drawings.

FIG. 1 shows a schematic of a method of forming a fiber according to various embodiments.

FIG. 2 shows a schematic of a fiber according to various embodiments.

FIG. 3A is a schematic illustrating the fabrication of ultra-compact MXene-based fibers with the protective layer (MGP-T) via continuous wet spinning and thermal drawing, and the formation of MXene-based textiles according to various embodiments.

FIG. 3B shows (top left) scanning electron microscopy (SEM) images of pure MXene fibers (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the pure MXene fibers according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa) as a function of strain (in percent or %) showing the stress-strain curve of the pure MXene fibers according to various embodiments.

FIG. 3C shows (top left) a scanning electron microscopy (SEM) images of resulting wet spun MXene fibers (MGP) (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the resulting wet spun MXene fibers (MGP) according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa) as a function of strain (in percent or %) showing the stress-strain curve of the resulting wet spun MXene fibers (MGP) according to various embodiments.

FIG. 3D shows (top left) a scanning electron microscopy (SEM) images of resulting MXene-based fibers with protective layers (MGP-T) (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the resulting fibers with protective layers (MGP-T) according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa) as a function of strain (in percent or %) showing the stress-strain curve of the resulting fibers with protective layers (MGP-T) according to various embodiments.

FIG. 4A shows a scanning electron microscopy (SEM) image of titanium aluminum carbide (Ti_3AlC_2) according to various embodiments.

FIG. 4B shows a scanning electron microscopy (SEM) image of accordion-like MXene according to various embodiments.

FIG. 5A shows a scanning electron microscopy (SEM) image of exfoliated MXene nanosheets with a lateral size of about 10 μm according to various embodiments.

FIG. 5B shows a plot of count as a function of lateral size (in micrometers or μm) illustrating the size distribution for the exfoliated MXene nanosheets shown in FIG. 5A according to various embodiments.

FIG. 5C shows an atomic force microscopy (AFM) image of exfoliated MXene nanosheets with a thickness of about 10 μm according to various embodiments.

FIG. 5D shows a plot of height (in nanometers or nm) as a function of lateral size (in micrometers or μm) illustrating the thickness of the exfoliated MXene nanosheets shown in FIG. 5C according to various embodiments.

FIG. 6A shows a transmission electron microscopy (TEM) image of exfoliated MXene nanosheets according to various embodiments.

FIG. 6B shows the corresponding high-resolution transmission electron microscopy (HR-TEM) image of the exfoliated MXene nanosheets; and (inset) selected area electron diffraction (SAED) pattern of the exfoliated MXene nanosheets according to various embodiments.

FIG. 7 is a plot of intensity (in arbitrary units or a.u.) as a function of angle (2θ) showing the X-ray diffraction (XRD) patterns of primitive titanium aluminum carbide (Ti_3AlC_2) and exfoliated MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets according to various embodiments.

FIG. 8 show polarizing optical microscope (POM) images of MXene-glutaraldehyde (GA) spinning dispersion with concentrations from $\sim 5 \text{ mg mL}^{-1}$ to $\sim 30 \text{ mg mL}^{-1}$ according to various embodiments.

FIG. 9A shows a plot of viscosity (in Pascals seconds or Pa.s) as a function of shear rate (in per second or $1/\text{s}$) illustrating the variation of viscosity with shear rate of different concentrations of the spinning dispersion according to various embodiments.

FIG. 9B shows a plot of shear stress (in Pascals or Pa) as a function of shear rate (in per second or 1/s) illustrating the variation of shear stress with shear rate of different concentrations of the spinning dispersion according to various embodiments.

FIG. 9C shows a plot of modulus (in Pascals or Pa) as a function of frequency (in per second or 1/s) illustrating the variation of modulus with frequency of different concentrations of the spinning dispersion according to various embodiments.

FIG. 10A shows a line drawing of several meter long wet spun (MGP) fibers according to various embodiments; and (inset) a magnified line drawing showing the axial SEM morphology of a wet spun (MGP) fiber according to various embodiments.

FIG. 10B shows a plot of intensity (in arbitrary units or a.u.) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the obtained fibers according to various embodiments, glutaraldehyde (GA), polyvinyl alcohol (PVA) and pure MXene.

FIG. 10C shows a plot of reflectance (in percent or %) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the obtained fibers according to various embodiments, glutaraldehyde (GA), polyvinyl alcohol (PVA) and pure MXene.

FIG. 10D shows a plot of reflectance (in percent or %) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the wet spun (MGP) fibers with different weight ratios of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 11 shows a plot of intensity (in arbitrary units or a.u.) as a function of wavenumbers (per centimeter or cm^{-1}) showing the X-ray photoelectron spectroscopy (XPS) spectra of the fabricated fibers according to various embodiments, pure MXene and MAX (Ti_3AlC_2).

FIG. 12A shows a plot of intensity (in arbitrary units or a.u.) as a function of binding energy (in electron volts or eV) showing the titanium (Ti) 2p spectra of the fabricated fibers according to various embodiments and pure MXene fibers.

FIG. 12B shows a plot of intensity (in arbitrary units or a.u.) as a function of binding energy (in electron volts or eV) showing the oxygen (O) 1s spectra of the fabricated fibers according to various embodiments and pure MXene fibers.

FIG. 12C shows a table illustrating the atomic percentage of O-Ti-O, C-Ti-OX, O-C, and C-Ti-OH according to the oxygen (O) 1s peak in the obtained X-ray photoelectron spectroscopy (XPS) spectra of the obtained fibers according to various embodiments and pure MXene fibers.

FIG. 13A illustrates one possible mechanism for the formation of Ti-O-C covalent bond between MXene nanosheets and glutaraldehyde molecules according to various embodiments.

FIG. 13B shows a structure graph of the covalent bond between MXene nanosheets and glutaraldehyde (GA) and the hydrogen bond between MXene nanosheets and polyvinyl alcohol (PVA) according to various embodiments.

FIG. 14A shows (a) wide-angle X-ray scattering (WAXS) patterns of the fabricated intermediate (MG) fibers with different weight ratios of glutaraldehyde molecules from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments; and (b) small-angle X-ray scattering (SAXS) patterns of the fabricated intermediate (MG) fibers with different weight ratios of glutaraldehyde molecules from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments.

FIG. 14B shows a plot of orientation order (f) as a function of percentage of glutaraldehyde (in weight percent or wt%) showing the variation of the orientation of the intermediate (MG) fibers with different amounts of glutaraldehyde based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments.

FIG. 14C shows a plot of density (in grams per cubic centimeter) / porosity (in percent or %) as a function of percentage of glutaraldehyde (in weight percent or wt%) showing the variation of density and porosity of the intermediate (MG) fibers with different amounts of glutaraldehyde according to various embodiments.

FIG. 15 shows the wide-angle X-ray scattering (WAXS) and (inset) small-angle X-ray scattering (SAXS) patterns of the fabricated wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 16A shows a plot of orientation order (f) as a function of percentage of polyvinyl alcohol (PVA) (in weight percent or wt%) showing the variation of the orientation of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments.

FIG. 16B shows a plot of density (in grams per cubic centimeter) / porosity (in percent or %) as a function of percentage of polyvinyl alcohol (in weight percent or wt%) showing the variation of density and porosity of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 16C shows a plot of tensile strength (in mega-Pascals or MPa) / toughness (in mega-Joules or MJ m⁻³) as a function of percentage of polyvinyl alcohol (PVA) (in weight percent or wt%) showing the variation of strength and toughness of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 17A shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or Å⁻¹) illustrating the variation of intensity of the intermediate (MG) fibers with different amounts of glutaraldehyde from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments.

FIG. 17B shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or Å⁻¹) illustrating the variation of intensity of the wet spun (MGP) fibers with

different amounts of polyvinyl alcohol (PVA) from 1 weight percent (wt%) to 15 weight percent (wt%) according to various embodiments.

FIG. 17C shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or $\text{\AA}^{-1}$) illustrating the variation of intensity of the various fabricated fibers according to various embodiments.

FIG. 18 shows a plot of weight loss (in percent or %) as a function of temperature (in degrees Celsius or $^{\circ}\text{C}$) illustrating the thermogravimetric analysis (TGA) results of polyvinyl alcohol (PVA), glutaraldehyde (GA) and various fabricated fibers according to various embodiments.

FIG. 19 shows a table illustrating the orientation order (f) and porosity (in percent or %) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments.

FIG. 20 shows a table illustrating the orientation order (f) and porosity (in percent or %) of the fabricated wet spun (MGP) fibers with different weight percentage of polyvinyl alcohol (PVA) molecules according to various embodiments.

FIG. 21 shows a table illustrating the tensile strengths (in mega-Pascals or MPa), strain (in percent or %) and toughness (in mega-Joules per cubic meter or MJ m^{-3}) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments.

FIG. 22A is a plot comparing the tensile strengths and toughness of fabricated intermediate (MG) fibers with different weight percentages of glutaraldehyde molecules from 0 wt% to 20 wt% according to various embodiments.

FIG. 22B is a plot comparing the electrical conductivity of fabricated intermediate (MG) fibers with different weight percentages of glutaraldehyde molecules from 0 wt% to 20 wt% according to various embodiments.

FIG. 23A shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of pure Mxene fibers according to various embodiments.

FIG. 23B shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 2 weight percent (2 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 23C shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 5 weight percent (5 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 23D shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 10 weight percent (10 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 23E shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 15 weight percent (15 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 23F shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 20 weight percent (20 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 24 shows a table illustrating the electrical conductivity (in siemens per centimeter or $S\ cm^{-1}$) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments.

FIG. 25A shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 5 weight percent (5 wt%) glutaraldehyde molecules according to various embodiments.

FIG. 25B shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 1 weight percent (1 wt%) polyvinyl alcohol (PVA) according to various embodiments.

FIG. 25C shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 2 weight percent (2 wt%) polyvinyl alcohol (PVA) according to various embodiments.

FIG. 25D shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 5 weight percent (5 wt%) polyvinyl alcohol (PVA) according to various embodiments.

FIG. 25E shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 10 weight percent (10 wt%) polyvinyl alcohol (PVA) according to various embodiments.

FIG. 25F shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 15 weight percent (15 wt%) polyvinyl alcohol (PVA) according to various embodiments.

FIG. 25G shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of pure polyvinyl alcohol (PVA) fibers.

FIG. 26 shows a table illustrating the tensile strengths (in mega-Pascals or MPa), strain (in percent or %) and toughness (in mega-Joules per cubic meter or MJ m⁻³) of various fibers with different weight percentage of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 27 is a plot comparing the electrical conductivity of MXene fibers, intermediate (MG) fibers, and wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 28 shows a table illustrating the electrical conductivity (in siemens per centimeter or $S\text{ cm}^{-1}$) of intermediate fibers and wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments.

FIG. 29 shows scanning electron microcopy images of the cross-sections of intermediate (MG) fibers with different weight percentages (wt%) of polyvinyl alcohol (PVA) from 0 wt% to 20 wt% according to various embodiments.

FIG. 30 shows scanning electron microcopy images of the cross-sections of wet spun (MGP) fibers with different weight percentages (wt%) of polyvinyl alcohol (PVA) from 1 wt% to 15 wt% according to various embodiments.

FIG. 31A shows a plot of temperature (in degree Celsius or $^{\circ}\text{C}$) as a function of scattering vector (per Angstrom or $\text{\AA}^{-1}$) illustrating the in-situ X-ray diffraction (XRD) patterns of a wet spun (MGP) fiber according to various embodiments.

FIG. 31B shows a plot of d-spacing (in Angstroms or $\text{\AA}$) as a function of temperature (in degree Celsius or $^{\circ}\text{C}$) illustrating the d-spacing curve according to the in-situ X-ray diffraction (XRD) pattern according to various embodiments.

FIG. 31C shows a plot of intensity (in arbitrary units or a.u.) as a function of scattering vector q (per Angstrom or $\text{\AA}^{-1}$) showing the in-situ X-ray diffraction (XRD) patterns of fabricated wet spun (MGP) fibers being heated under various temperatures from 25 $^{\circ}\text{C}$ to 350 $^{\circ}\text{C}$ according to various embodiments.

FIG. 31D shows a plot of porosity (in percent or %) as a function of temperature (in degree Celsius or $^{\circ}\text{C}$) illustrated the porosity of wet spun (MGP) fibers being heated according to various embodiments.

FIG. 32A shows a schematic diagram of wet spun (MGP) fibers being heated according to various embodiments.

FIG. 32B shows the in-situ small-angle X-ray scattering (SAXS) patterns of fabricated wet spun (MGP) fibers (with 5% polyvinyl alcohol (PVA)) being heated under variable temperatures from 25°C to 350 °C according to various embodiments.

FIG. 32C shows a plot of intensity as a function of scattering vector q (per Angstrom or $\text{\AA}^{-1}$) illustrating the intensity curves of wet spun fibers under different temperatures based on the small-angle X-ray scattering (SAXS) patterns according to various embodiments.

FIG. 33A shows a schematic diagram of forming the cross-linked MXene-based (MGP-T) fibers with protective layers from wet spun (MGP) fibers with axial stress and perpendicular stress during thermal drawing according to various embodiments.

FIG. 33B shows a line drawing of several meters long cross-linked MXene-based fibers (MGP-T) fibers with protective layers according to various embodiments.

FIG. 33C shows another line drawing of cross-linked MXene-based (MGP-T) fibers with protective layers according to various embodiments.

FIG. 34A is a plot comparing the orientation orders (f) of three cross-linked MXene-based (MGP-T) fibers with protective layers fabricated by various draw-down ratios according to various embodiments, with the inset showing the wide-angle X-ray scattering (WAXS) pattern of the various fibers according to various embodiments.

FIG. 34B shows a plot of intensity (in arbitrary units or a.u.) as a function of azimuthal angle (in degrees or $^\circ$) showing the variation across fibers formed by different draw-down ratios based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments.

FIG. 34C shows a table comparing the orientation order (f) and porosity (in percent or %) of the fabricated MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios according to various embodiments.

FIG. 35A shows a plot of porosity (in percent or %)/conductivity (in $\times 10^3$ siemens per centimeter or S cm^{-1}) comparing the porosity and electrical conductivity of the various

fabricated MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios according to various embodiments.

FIG. 35B shows the scanning electron microscopy (SEM) cross-sectional images of the MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios (a: MGP-T₁, b: MGP-T₂, c: MGP-T₃) with energy-dispersive X-ray spectroscopy (EDX) according to various embodiments.

FIG. 36A shows a high resolution transmission electron microscopy (HR-TEM) image of a Mxene-based fiber with protective layer (MGP-T) according to various embodiments.

FIG. 36B shows a zoomed in view of the area indicated in FIG. 36A according to various embodiments.

FIG. 36C shows the EDX spectrum of the selected area shown in FIG. 36A according to various embodiments.

FIG. 37 shows a table illustrating the electrical conductivity of the fabricated MXene-based fibers with protective layers (MGP-T) formed using various draw-down ratios according to various embodiments.

FIG. 38A shows a plot of tensile strength (in mega-Pascals or MPa)/toughness (in mega-Joules per cubic meter or MJ m⁻³) comparing the tensile strength and toughness of the various fabricated cross-linked MXene-based (MGP-T) fibers formed using different draw-down ratios according to various embodiments.

FIG. 38B shows a table illustrating the tensile strength (in mega-Pascals or MPa) and toughness (in mega-Joules per cubic meter or MJ m⁻³) of the fabricated MXene-based fibers with protective layers (MGP-T) formed using various draw-down ratios according to various embodiments and polycarbonate (PC).

FIG. 39A shows a plot of tensile strength (in mega-Pascals or MPa) as a function of electrical conductivity (in $\times 10^3$ siemens per centimeter or S cm⁻¹) comparing the tensile strength and

electrical conductivity of the obtained compact MXene-based fibers according to various embodiments with other reported MXene-based fibers.

FIG. 39B shows a table comparing the tensile strength (in mega-Pascals or MPa), toughness (in mega-Joules per cubic meter or MJ m⁻³) and electrical conductivity (in siemens per centimeter or S cm⁻¹) of the obtained compact MXene-based fibers according to various embodiments with that of other reported MXene-based fibers

FIG. 40A shows a plot of toughness (in mega-Joules per cubic meter or MJ m⁻³) as a function of electrical conductivity ((in siemens per centimeter or S cm⁻¹) comparing the toughness and electrical conductivity of the ultra-compact MXene fibers according to various embodiments with that for other reported MXene-based fibers, graphene fibers, and carbon nanotube (CNT) fibers.

FIG. 40B shows a table comparing the tensile strength (in mega-Pascals or MPa), toughness (in mega-Joules per cubic meter or MJ m⁻³) and electrical conductivity (in siemens per centimeter or S cm⁻¹) of the obtained compact MXene-based fibers according to various embodiments with that of other reported carbon -based fibers.

FIG. 41 shows (a) a schematic diagram of the apparatus used for the thermal drawing according to various embodiments; and (b) a polycarbonate (PC) hollow tube model with an inner diameter of 0.06 mm, an outer-diameter of 0.08 mm, and a length of 0.50 mm long constructed to study the mechanical behavior of the PC hollow tube via finite element analysis according to various embodiments.

FIG. 42A shows a simulation of the stress distribution of the polycarbonate (PC) hollow tube model according to various embodiments.

FIG. 42B shows plots of stress (in mega-Pascals or MPa) as a function of draw-down ratio illustrating the Y and Z stress curves with the increment of the draw-down ratio according to finite element analysis according to various embodiments.

FIG. 43A shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) as a function of frequency (in gigahertz or GHz) illustrating the EMI reflection shielding efficiency (SE_R), absorption shielding efficiency (SE_A) and total efficiency (SE_T) of the textiles according to various embodiments at the frequencies of 8.2 GHz – 12.4 GHz.

FIG. 43B is a schematic illustrating plain-weaved textiles based on MXene-based fibers according to various embodiments for electromagnetic interference (EMI) measurements.

FIG. 44A shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) comparing the reflection shielding efficiency (SE_R), absorption shielding efficiency (SE_A) and total efficiency (SE_T) of textiles fabricated from the MXene-based fibers with protective layers (MGP-T) obtained using various draw-down ratios according to various embodiments at the frequency of 8.2 GHz.

FIG. 44B shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) as a function of frequency (in gigahertz or GHz) illustrating the electromagnetic interference (EMI) properties of 3-layered textiles fabricated using various MXene-based fibers with protective layers (MGP-T) according to various embodiments at frequencies from 8.2 GHz to 12.4 GHz.

FIG. 44C shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) comparing the electromagnetic interference (EMI) properties of 3-layered textiles fabricated using various fabricated and pure MXene fibers according to various embodiments at the frequency of 8.2 GHz.

FIG. 45A is a schematic illustrating the electromagnetic interference (EMI) mechanism of textiles fabricated using the MXene-based fibers with protective layers (MGP-T) according to various embodiments.

FIG. 45B shows a plot of electromagnetic interference shielding efficiency (EMI SE) retention (in percent or %) as a function of number of bending cycles illustrating the electromagnetic

interference shielding efficiency retention of the textile according to various embodiments after 5×10^4 bending cycles.

FIG. 46A shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating the temperature-time curves of single MXene-based fibers with protective layers (MGP-T) according to various embodiments when a direct current (DC) voltage of 6 V is applied.

FIG. 46B shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating the temperature-time curves of a MXene-based fiber with protective layer (MGP-T) according to various embodiments when voltages from 2 V to 8 V are applied.

FIG. 46C shows a plot of performance retention (in percent or %) as a function of the number of cycles illustrating the cycling life of a MXene-based fiber with protective layer (MGP-T) according to various embodiments, while the inset is a plot of temperature (in degrees Celsius or °C) as a function of time (in seconds or s) illustrating the temperature variation profile in the first and last few cycles according to various embodiments.

FIG. 46D shows images of various MXene-based fibers with protective layers (MGP-T) arranged in shapes of different letters upon application of various direct current (DC) voltages of 2 V to 8 V according to various embodiments.

FIG. 46E shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating temperature-time curves of a single MXene-based fiber with protective layer (MGP-T) according to various embodiments at the bending angles from 0° to 180°.

FIG. 46F shows a plot of performance retention (in percent or %) as a function of diameter (in millimeters or mm) illustrating the temperature performance retention of the knots of MXene-based fibers with protective layers (MGP-T) according to various embodiments.

FIG. 46G shows a plot of performance retention (in percent or %) as a function of the number of bending cycles illustrating the temperature performance retention of a single MXene-based

fiber with protective layer (MGP-T) according to various embodiments after 1.1×10^5 bending cycles from the bending angles of 0° to 180° .

FIG. 47A shows a line drawing of the piece of cotton cloth with the size of 0.6 m by 2.0 m woven by 18 meter-long ultra-compact MXene-based fibers with protective layers (MGP-T) according to various embodiments.

FIG. 47B shows an enlarged line drawing of a portion of the cloth shown in FIG. 47A according to various embodiments.

FIG. 47C shows a line drawing of designed patterns woven into the cotton cloth according to various embodiments; and (inset) the cloth according to various embodiments being subjected to complex deformation.

FIG. 47D shows a line drawing of a sweater woven with several MXene-based fibers with protective layers (MGP-T) according to various embodiments to generate heat.

FIG. 47E shows a thermal image of the sweater in FIG. 47D according to various embodiments when a potential difference of 2 V is applied.

FIG. 47F shows a thermal image of the sweater in FIG. 47D according to various embodiments when a potential difference of 4 V is applied.

FIG. 47G shows an enlarged line drawing of a portion of the sweater shown in FIG. 47D according to various embodiments.

DESCRIPTION

[0007] The following detailed description refers to the accompanying drawings that show, by way of illustration, specific details and embodiments in which the invention may be practised. These embodiments are described in sufficient detail to enable those skilled in the art to practise the invention. Other embodiments may be utilized and structural, logical, and electrical changes may be made without departing from the scope of the invention. The various

embodiments are not necessarily mutually exclusive, as some embodiments can be combined with one or more other embodiments to form new embodiments. For instance, embodiments described in the context of one group of fibers are analogously valid for other groups of fibers. Similarly, embodiments described in the context of a method of forming a plurality of fibers are analogously valid for a plurality of fibers, and vice versa. Features that are described in the context of an embodiment may correspondingly be applicable to the same or similar features in the other embodiments. Features that are described in the context of an embodiment may correspondingly be applicable to the other embodiments, even if not explicitly described in these other embodiments. Furthermore, additions and/or combinations and/or alternatives as described for a feature in the context of an embodiment may correspondingly be applicable to the same or similar feature in the other embodiments.

[0008] In the context of various embodiments, the term “about” or “approximately” as applied to a numeric value encompasses the exact value and a reasonable variance, e.g. within 10% of the specified value.

[0009] As used herein, the term “and/or” includes any and all combinations of one or more of the associated listed items.

[0010] By “comprising” it is meant including, but not limited to, whatever follows the word “comprising”. Thus, use of the term “comprising” indicates that the listed elements are required or mandatory, but that other elements are optional and may or may not be present.

[0011] By “consisting of” is meant including, and limited to, whatever follows the phrase “consisting of”. Thus, the phrase “consisting of” indicates that the listed elements are required or mandatory, and that no other elements may be present.

[0012] Both the electrical conductivity and mechanical properties of MXene nanosheets have not been fully utilized in the form factor of a fiber currently, mainly due to the inherently loose layers caused by the structure defects (voids and wrinkles of MXene nanosheets) and poor

interlayer interactions between MXene nanosheets in existing fibers. Some efforts have been proposed to form compact layered structures between MXene nanosheets using wet spinning. Despite the enhanced mechanical and electrical performance, major challenges remain to reach a balance between electrical conductivity, strength, and toughness so as to achieve the full potential of MXene fibers. In particular, there are difficulties to further compact the loose MXene layers. Thus, in order to fundamentally address this challenge, a process that can achieve ultra-compact yet long MXene fibers in a controllable manner may be required.

[0013] After fibers with high mechanical and electrical performance are achieved, they can be used to construct functional textiles for large-scale coverage such as on the human body. However, performance degradation of these fibers is the common reason to limit their long-term usage, due to the fact that they are fully exposed to the environment and skin, and may suffer from physical impact applied directly on them due to body movements. In order to address these issues, an effective method may be to form a protective layer on the outer surfaces of these fibers. Achieving this may not only require additional process steps, but may also bring up uncertainties on controlling the interaction between the resulting fibers and coatings. Furthermore, it may be necessary to conduct an accurate assessment on the properties of flexible fibers with protective layers, for the consideration of practical scenarios.

[0014] Various embodiments may seek to meet the abovementioned requirements. Various embodiments may seek to address the abovementioned issues/shortcomings facing current fibers. Various embodiments may seek to address issues facing fibers without protective layers. Various embodiments may relate to a continuous and controllable route to fabricate ultra-compact MXene-based fibers.

[0015] FIG. 1 shows a schematic of a method of forming a fiber according to various embodiments. The method may include, in 102, forming a wet spun fiber from a plurality of MXene sheets using a wet spinning process. The method may also include, in 104, forming the

fiber by heating the wet spun fiber using a thermal drawing process. The fiber may be a cross-linked MXene-based fiber including a MXene core including the plurality of MXene sheets which are cross-linked. The fiber may further include a protective layer surrounding the MXene core.

[0016] In other words, the method may include wet-spinning MXene sheets into a wet-spun fiber, and subjecting the wet spun fiber to a thermal drawing process. The resultant fiber may have a MXene core, and a protective layer covering the circumferential surface of the core. The MXene core may include the plurality of cross-linked MXene sheets.

[0017] The fibers (alternatively referred to as a MXene-based fibers or cross-linked MXene based fibers) having the MXene cores and the protective layers surrounding the MXene cores may be referred to as MGP-T fibers, while the wet spun fibers prior to the thermal drawing process (alternatively referred to as wet spun MXene fibers) may be referred to as MGP fibers.

[0018] In various embodiments, a MXene sheet may include a sheet including a MXene material. Likewise, a MXene core/fiber may be a core/fiber including a MXene material.

[0019] MXene sheets or fibers/cores may include transition metal carbides, nitrides or carbonitrides. In various embodiments, the MXene sheets may be $\text{Ti}_3\text{C}_2\text{T}_x$ sheets, wherein Ti is titanium, C is carbon, T is a functional group (e.g. -OH, -O, or -F) and x is any number. Likewise, the MXene fibers/cores may be or include $\text{Ti}_3\text{C}_2\text{T}_x$. The $\text{Ti}_3\text{C}_2\text{T}_x$ sheets may be formed from Ti_3AlC_2 . In various other embodiments, the MXene sheets or fibers/cores may, for instance, be V_2CT_x , wherein V is vanadium, C is carbon, T is a functional group and x is any number. In yet various other embodiments, the MXene sheets or fibers/cores may, for instance, be Nb_2CT_x , wherein Nb is niobium, C is carbon, T is a functional group and x is any number.

[0020] In various embodiments, the one or more MXene sheets may be nanosheets. The MXene sheets may have a thickness of a few atoms.

[0021] In various embodiments, forming the wet spun fiber from the plurality of MXene sheets may include forming an intermediate fiber by extruding a spinning dispersion including the plurality of MXene sheets into a coagulant bath. Forming the wet spun fiber from the plurality of MXene sheets may also include forming the wet spun fiber by passing the intermediate fiber through a cross-linking solution.

[0022] In various embodiments, the intermediate fiber, the wet spun fiber and/or the fiber including the MXene core and the protective layer may be nanofibers.

[0023] As mentioned above, in various embodiments, the MXene core may include a plurality of cross-linked sheets, e.g. nanosheets. The term “cross-linked MXene sheets” or the term “the plurality of MXene sheets which are cross-linked” may refer to the plurality of MXene sheets of the MXene core, in which each MXene sheet of the MXene core is cross-linked to one or more other MXene sheets of the MXene core. Similarly, the term “cross-linked MXene nanosheets” or the term “the plurality of MXene nanosheets which are cross-linked” may refer to the plurality of MXene nanosheets of the MXene core, in which each MXene nanosheet of the MXene core is cross-linked with one or more other MXene nanosheets of the MXene core. In various embodiments, there may be cross-linking between fibers. For instance, in situations such as when the fibers are in wet condition, there may also be crosslinking between the fibers in addition to cross-linking between sheets of each fiber. Various embodiments may relate to a plurality of fibers (i.e. plurality of MXene-based cross-linked fibers) which may be cross-linked to one another. Various other embodiments may relate to a plurality of fibers (i.e. plurality of MXene-based cross-linked fibers) which may not be cross-linked to one another. In various embodiments, the spinning dispersion may include glutaraldehyde (GA). The intermediate fiber formed may be a cross-linked MXene- glutaraldehyde (GA) fiber. In other words, GA may function as a cross-linker. GA may form covalent bonds (titanium-oxygen-carbon or Ti-O-C bonds) between different sheets or nanosheets of an intermediate fiber, a wet spun fiber, or a

fiber including the protective layer. The GA molecules may form the covalent bond(s) via a nucleophilic substitution and dehydration reaction. In various other embodiments, the spinning dispersion may include nanocrystalline cellulose such that the intermediate fiber formed is a cross-linked MXene-nanocrystalline cellulose fiber. In other words, nanocrystalline cellulose may function as a cross-linker.

[0024] In various embodiments, the cross-linking solution may include polyvinyl alcohol (PVA). PVA may form hydrogen bonds between different sheets or nanosheets of a wet spun fiber, or a fiber including the protective layer. For instance, the wet spun fiber formed may be a cross-linked MXene- glutaraldehyde (GA) – polyvinyl alcohol (PVA) fiber. In other words, PVA may also function as a cross-linker. In various other embodiments, the cross-linking solution may include chitosan or sodium alginate.

[0025] In various embodiments, the coagulant bath may include ammonium ions.

[0026] In various embodiments, the method may further include passing the intermediate fiber through a first washing bath before forming the wet spun fiber. In various embodiments, the method may also include passing the wet spun fiber through a second washing bath before forming the cross-linked MXene-based fiber. The first washing bath and the second washing bath may include deionized water.

[0027] The intermediate fibers may be referred to as MG fibers.

[0028] In various embodiments, the protective layer may be a polymer layer. For instance, the protective layer may include polycarbonate, cyclo olefin polymer (COP), chlorinated polyethylene (CPE), polyethylene (PE), or polyetherimide (PEI). In various embodiments, the protective layer may cover a circumferential surface of the MXene core. In various embodiments, the protective layer may also cover end surfaces of the MXene core.

[0029] In various embodiments, the cross-linked MXene-based fiber may have a tensile strength selected from a range of between about 350 MPa and about 600 MPa, e.g. between

about 372.5 MPa and about 585.5 MPa. In various embodiments, the cross-linked MXene-based fiber may have a tensile strength selected from a range of between 350 MPa and 600 MPa, e.g. between 372.5 MPa and 585.5 MPa.

[0030] In various embodiments, the cross-linked MXene-based fiber may have a toughness selected from a range of between about 60 MJ m⁻³ and about 80 MJ m⁻³, e.g. between about 66.7 MJ m⁻³ and about 77.9 MJ m⁻³. In various embodiments, the cross-linked MXene-based fiber may have a toughness selected from a range of between 60 MJ m⁻³ and 80 MJ m⁻³, e.g. between 66.7 MJ m⁻³ and 77.9 MJ m⁻³.

[0031] In various embodiments, the cross-linked MXene-based fiber may have an electrical conductivity selected from a range of between about 8,000 S cm⁻¹ and about 9,000 S cm⁻¹, e.g. between about 8,344.5 S cm⁻¹ and about 8,802.4 S cm⁻¹. In various embodiments, the cross-linked MXene-based fiber may have an electrical conductivity selected from a range of between 8,000 S cm⁻¹ and 9,000 S cm⁻¹, between 8,344.5 S cm⁻¹ and 8,802.4 S cm⁻¹.

[0032] In various embodiments, the thermal drawing process may include feeding the wet spun fiber into a tube passing through a furnace. The furnace may have a hot zone having a temperature of about 200 °C, e.g. 200 °C.

[0033] In various embodiments, a ratio of pull to feed speed of the thermal drawing process may be of any value selected from a range of between about 1.2 and about 1.5, e.g. about 1.26, about 1.34 and about 1.41. In various embodiments, a ratio of pull to feed speed of the thermal drawing process may be of any value selected from a range of between 1.2 and 1.5, e.g. 1.26, 1.34 and 1.41.

[0034] In various embodiments, the cross-linked MXene-based fiber may show a porosity selected from a range of between about 5.5 % to about 9 %, between about 5.7% to about 8.6%. In various embodiments, the cross-linked MXene-based fiber may show a porosity selected from a range of between 5.5 % to 9 %, between 5.7% to 8.6%.

[0035] In various embodiments, the cross-linked MXene-based fiber may show an orientation order selected from a range of between about 0.8 to about 0.9, e.g. between about 0.84 to about 0.89. In various embodiments, the cross-linked MXene-based fiber may show an orientation order selected from a range of between 0.8 to 0.9, e.g. between 0.84 to 0.89.

[0036] In various embodiments, the cross-linked MXene-based fiber may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 40 dB to about 55 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 42 dB to about 51 dB (e.g. at about 8.2GHz). In various embodiments, the cross-linked MXene-based fiber may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 40 dB to 55 dB, e.g. a range of between 42 dB to 51 dB.

[0037] In various embodiments, the cross-linked MXene-based fiber may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 50 dB to about 65 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 52 dB to about 61 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 50 dB to 65 dB, e.g. a range of between 52 dB to 61 dB.

[0038] In various embodiments, the cross-linked MXene-based fiber including the protective layer may be configured to reach an equilibrium temperature selected from a range of between about 90 °C to about 100 °C within about 300 s upon applying a direct current (DC) voltage of about 6 V. In various embodiments, the cross-linked MXene-based fiber including the protective layer may be configured to reach an equilibrium temperature selected from a

range of between 90 °C to 100 °C within 300 s upon applying a direct current (DC) voltage of 6V.

[0039] Various embodiments may relate to a fiber formed by a method as described herein. Various embodiments may relate to a fiber formed by a method including forming a wet spun fiber from a plurality of MXene sheets using a wet spinning process, and forming the fiber by heating the wet spun fiber using a thermal drawing process. The fiber may be a cross-linked MXene-based fiber including a MXene core including the plurality of MXene sheets which are cross-linked. The fiber may further include a protective layer surrounding the MXene core.

[0040] Various embodiments may relate to a method of forming one or more fibers. The method may include forming one or more wet spun fibers, each of the one or more wet spun fibers from a plurality of MXene sheets, using a wet spinning process. The method may also include forming one or more fibers by heating the one or more wet spun fibers using a thermal drawing process. The one or more fibers may be cross-linked MXene-based fibers, each of the one or more fibers including a MXene core including the plurality of MXene sheets which are cross-linked. The fiber may further include a protective layer surrounding the MXene core(s). In particular, various embodiments may relate to a method of forming a fiber bundle containing a plurality of fibers. The method may include forming a plurality of wet spun fibers, each of the plurality of wet spun fibers from a plurality of MXene sheets, using a wet spinning process. The method may also include forming the fiber bundle containing the plurality of fibers by heating the plurality of wet spun fibers using a thermal drawing process. The plurality of fibers may be cross-linked MXene-based fibers, each of the plurality of fibers including a MXene core including the plurality of MXene sheets which are cross-linked. The fiber bundle may further include a protective layer surrounding the plurality of fibers or the MXene cores.

[0041] FIG. 2 shows a schematic of a fiber 202 according to various embodiments. The fiber 202 may include a MXene core 204. The fiber 202 may also include a protective layer 206

surrounding the MXene core 204. The fiber 202 may be a cross-linked MXene-based fiber such that the MXene core includes a plurality of MXene sheets which are cross-linked.

[0042] In other words, various embodiments may relate to a cross-linked MXene-based fiber including a MXene core which has multiple cross-linked sheets. The cross-linked MXene-based fiber may also have a protective layer covering the circumferential surface of the MXene core.

[0043] For avoidance of doubt, FIG. 2 is intended to illustrate some features according to some embodiments, and is not intended to limit, for instance, the dimensions, aspect ratios, arrangement, orientation etc. of the fibers.

[0044] In various embodiments, the fiber 202 may be a cross-linked MXene- glutaraldehyde (GA) – polyvinyl alcohol (PVA) fiber. In various embodiments, besides GA, the sheets or nanosheets of the MXene core 204 may alternatively be cross-linked by nanocrystalline cellulose. In various embodiments, besides PVA, the sheets or nanosheets of the MXene core 204 may alternatively be cross-linked by chitosan or sodium alginate.

[0045] In various embodiments, the protective layer 206 may be a polymer layer. In various embodiments, the protective layer may be a polymer layer. For instance, the protective layer may include polycarbonate, cyclo olefin polymer (COP), chlorinated polyethylene (CPE), polyethylene (PE), or polyetherimide (PEI).

[0046] In various embodiments, the cross-linked MXene-based fiber 202 may have a tensile strength selected from a range of between about 350 MPa and about 600 MPa, e.g. between about 372.5 MPa and about 585.5 MPa. In various embodiments, the cross-linked MXene-based fiber 202 may have a tensile strength selected from a range of between 350 MPa and 600 MPa, e.g. between 372.5 MPa and 585.5 MPa.

[0047] In various embodiments, the cross-linked MXene-based fiber 202 may have a toughness selected from a range of between about 60 MJ m⁻³ and about 80 MJ m⁻³, e.g. between about 66.7 MJ m⁻³ and about 77.9 MJ m⁻³. In various embodiments, the cross-linked MXene-

based fiber 202 may have a toughness selected from a range of between 60 MJ m^{-3} and 80 MJ m^{-3} , e.g. between 66.7 MJ m^{-3} and 77.9 MJ m^{-3} .

[0048] In various embodiments, the cross-linked MXene-based fiber 202 may have an electrical conductivity selected from a range of between about $8,000 \text{ S cm}^{-1}$ and about $9,000 \text{ S cm}^{-1}$, between about $8,344.5 \text{ S cm}^{-1}$ and about $8,802.4 \text{ S cm}^{-1}$. In various embodiments, the cross-linked MXene-based fiber 202 may have an electrical conductivity selected from a range of between $8,000 \text{ S cm}^{-1}$ and $9,000 \text{ S cm}^{-1}$, between $8,344.5 \text{ S cm}^{-1}$ and $8,802.4 \text{ S cm}^{-1}$.

[0049] In various embodiments, the cross-linked MXene-based fiber 202 may show a porosity selected from a range of between about 5.5 % to about 9 %, between about 5.7% to about 8.6%. In various embodiments, the cross-linked MXene-based fiber 202 may show a porosity selected from a range of between 5.5 % to 9 %, between 5.7% to 8.6%.

[0050] In various embodiments, the cross-linked MXene-based fiber 202 may show an orientation order selected from a range of between about 0.8 to about 0.9, e.g. between about 0.84 to about 0.89. In various embodiments, the cross-linked MXene-based fiber 202 may show an orientation order selected from a range of between 0.8 to 0.9, e.g. between 0.84 to 0.89.

[0051] In various embodiments, the cross-linked MXene-based fiber 202 may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 40 dB to about 55 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber 202 may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 42 dB to about 51 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber 202 may show an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 40 dB to 55 dB, e.g. selected from a range of between 42 dB to 51 dB.

[0052] In various embodiments, the cross-linked MXene-based fiber 202 may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between

about 50 dB to about 65 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber 202 may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between about 52 dB to about 61 dB (e.g. at about 8.2 GHz). In various embodiments, the cross-linked MXene-based fiber 202 may show a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 50 dB to 65 dB, a range of between 52 dB to 61 dB.

[0053] In various embodiments, the cross-linked MXene-based fiber 202 including the protective layer may be configured to reach an equilibrium temperature selected from a range of between about 90 °C to about 100 °C within about 300 s upon applying a direct current (DC) voltage of about 6 V. In various embodiments, the cross-linked MXene-based fiber 202 including the protective layer may be configured to reach an equilibrium temperature selected from a range of between 90 °C to 100 °C within 300 s upon applying a direct current (DC) voltage of 6 V.

[0054] Various embodiments may relate to an object including one or more fibers as described herein. The object may, for instance, be a textile cloth, an electromagnetic shield or a clothing such as a sweater.

[0055] Various embodiments may relate to one or more fibers, each of the one or more fibers including a MXene core. The one or more fibers may also include a protective layer surrounding the MXene core(s). Each of the one or more fibers may be a cross-linked MXene-based fiber such that the MXene core includes a plurality of MXene sheets which are cross-linked. In particular, various embodiments may relate to a fiber bundle containing a plurality of fibers, each of the plurality of fibers include a MXene core. The fiber bundle may include a protective layer surrounding the MXene cores or the plurality of fibers. Each of the plurality of fibers may be a cross-linked MXene-based fiber such that the MXene core includes a plurality of MXene sheets which are cross-linked.

[0056] Various embodiments may relate to a continuous and controllable route to fabricate ultra-compact MXene fibers. To start, interfacial interactions in the wet spinning may enable the transfer from MXene nanosheets to compact MXene fibers. Then, these fibers may continuously be fed into a polymer tube in thermal drawing, resulting in (1) ultra-compact MXene fibers due to the drawing-induced stresses, and (2) in-situ generated protective layer. Owing to such a synergy of interfacial interactions and thermal drawing-induced stresses, the resulting ultra-compact MXene fibers with protective layers may not only offer remarkable tensile strength of ~ 585.5 MPa and ultra-high toughness of ~ 66.7 MJ m⁻³, but may also exhibit high electrical conductivity of $\sim 8,802.4$ S cm⁻¹ and excellent long-term mechanical durability and stability. Two representative applications based on large-scale MXene textiles fabricated using these fibers are also explored herein.

[0057] One relates to electromagnetic interference shielding with high shielding efficiency of ~ 57 dB and $\sim 87.8\%$ retention of performance after 5×10^4 bending cycles. The other one relates to electrothermal effect with the demonstration of a sweater generating heat, with temperatures reaching ~ 35 °C ~ 70 °C. The demonstrated synergy of interfacial interactions and thermal drawing-induced stresses may attract interests for other applications of the compact MXene-based fibers.

[0058] Results

[0059] The flexible MXene fibers with high compactness may be fabricated by a continuous and controllable synergy of wet spinning with interfacial interactions and thermal drawing-induced stresses, as schematically illustrated in FIG. 3A. FIG. 3A is a schematic illustrating the fabrication of ultra-compact MXene-based fibers with the protective layer (MGP-T) via continuous wet spinning and thermal drawing, and the formation of MXene-based textiles according to various embodiments. Large-area multi-functional fabrics can be constructed by the resulting fibers for applications in electromagnetic interference (EMI) shielding and thermal

management based on electrothermal (ET) effect. Compared to the pure MXene fibers with poor interfacial interaction, the resulting wet spun MXene fibers (MGP) may achieve increased orientation orders (f) from ~ 0.82 to ~ 0.87 , and low porosity due to synergistic interfacial interactions. This may result in the enhancement of the tensile strength from ~ 167.1 MPa to ~ 565.2 MPa with increase in toughness from ~ 0.4 MJ m⁻³ to ~ 19.2 MJ m⁻³. FIG. 3B shows (top left) scanning electron microscopy (SEM) images of pure MXene fibers (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the pure MXene fibers according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa) as a function of strain (in percent or %) showing the stress-strain curve of the pure MXene fibers according to various embodiments. FIG. 3C shows (top left) a scanning electron microscopy (SEM) images of resulting wet spun MXene fibers (MGP) (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the resulting wet spun MXene fibers (MGP) according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa) as a function of strain (in percent or %) showing the stress-strain curve of the resulting wet spun MXene fibers (MGP) according to various embodiments. After sequentially suffered from the effectively thermal drawing-induced stresses, the ultra-compact MXene-based fibers with protective layer (MGP-T) may be obtained with the significantly promoted orientation orders (f) of ~ 0.90 , accompanied by the further reduction of the porosity. As a result, the obtained MGP-T fibers with the protective layer may offer the highest strength of ~ 585.5 MPa and toughness of ~ 66.7 MJ m⁻³, thanks to the enhancement of alignment and reduction of voids. FIG. 3D shows (top left) a scanning electron microscopy (SEM) images of resulting MXene-based fibers with protective layers (MGP-T) (normal and zoomed in) according to various embodiments; (top right) a wide-angle X-ray scattering (WAXS) pattern of the resulting fibers with protective layers (MGP-T) according to various embodiments; and (below) a plot of the stress (in mega Pascals or MPa)

as a function of strain (in percent or %) showing the stress-strain curve of the resulting fibers with protective layers (MGP-T)) according to various embodiments. The results show that the MGP-T fibers may offer excellent mechanical properties with high alignment and low porosity, compared to pure MXene and MGP fibers.

[0060] Synthesis and characterization of MXene fibers via wet spinning

[0061] To start, MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets may be prepared by etching and shaking from preliminary titanium aluminum carbide (Ti_3AlC_2) and accordion-like MXene. FIG. 4A shows a scanning electron microscopy (SEM) image of titanium aluminum carbide (Ti_3AlC_2) according to various embodiments. FIG. 4B shows a scanning electron microscopy (SEM) image of accordion-like MXene according to various embodiments.

[0062] The obtained MXene nanosheets have a lateral size of $\sim 10\ \mu\text{m}$ with a thickness of $\sim 1.5\ \text{nm}$ according to the scanning electron microscopy (SEM) and atomic force microscopy (AFM) images. FIG. 5A shows a scanning electron microscopy (SEM) image of exfoliated MXene nanosheets with a lateral size of about $10\ \mu\text{m}$ according to various embodiments. FIG. 5B shows a plot of count as a function of lateral size (in micrometers or μm) illustrating the size distribution for the exfoliated MXene nanosheets shown in FIG. 5A according to various embodiments. FIG. 5C shows an atomic force microscopy (AFM) image of exfoliated MXene nanosheets with a thickness of about $10\ \mu\text{m}$ according to various embodiments. FIG. 5D shows a plot of height (in nanometers or nm) as a function of lateral size (in micrometers or μm) illustrating the thickness of the exfoliated MXene nanosheets shown in FIG. 5C according to various embodiments.

[0063] Moreover, the exfoliated MXene nanosheets may have the expected high crystallinity hexagonal structures without defects, confirmed by the high-resolution transmission electron microscopy (HR-TEM) images and the selected area electron diffraction patterns. FIG. 6A shows a transmission electron microscopy (TEM) image of exfoliated

MXene nanosheets according to various embodiments. FIG. 6B shows the corresponding high-resolution transmission electron microscopy (HR-TEM) image of the exfoliated MXene nanosheets; and (inset) selected area electron diffraction (SAED) pattern of the exfoliated MXene nanosheets according to various embodiments. The SAED patterns indicates hexagonal single crystals structure without obvious defects.

[0064] FIG. 7 is a plot of intensity (in arbitrary units or a.u.) as a function of angle (2θ) showing the X-ray diffraction (XRD) patterns of primitive titanium aluminum carbide (Ti_3AlC_2) and exfoliated MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets according to various embodiments. The X-ray diffraction (XRD) patterns of exfoliated MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) nanosheets do not show the 104 and 105 peaks characteristics of Ti_3AlC_2 , demonstrating the complete etching of the aluminum (Al) layer and indicating that the MXene nanosheets have been successfully prepared from Ti_3AlC_2 . The disappearance of the (002) and (004) peaks of Ti_3AlC_2 and a new peak at the 2θ of $\sim 6^\circ$ may also indicate that the MXene nanosheets were successfully fabricated from the Ti_3AlC_2 .

[0065] According to Onsager's theoretical prediction model, MXene nanosheets in the MXene-glutaraldehyde (GA) spinning dispersion may present lyotropic liquid-crystalline properties in the range of $\sim 15 \text{ mg mL}^{-1}$ to $\sim 30 \text{ mg mL}^{-1}$. FIG. 8 show polarizing optical microscope (POM) images of MXene-glutaraldehyde (GA) spinning dispersion with concentrations from $\sim 5 \text{ mg mL}^{-1}$ to $\sim 30 \text{ mg mL}^{-1}$ according to various embodiments. As shown in FIG. 8, optical birefringence may be observed in concentrations of $\sim 15 \text{ mg mL}^{-1}$ to $\sim 30 \text{ mg mL}^{-1}$. This may indicate the formation of liquid-crystalline phase as a result of the local orientation without aggregation.

[0066] Meanwhile, the spinning dispersion with high concentration of 30 mg mL^{-1} may form a viscous ink with a viscosity of $\sim 4.1 \text{ kPa s}$ without aggregates.

[0067] FIG. 9A shows a plot of viscosity (in Pascals seconds or Pa.s) as a function of shear rate (in per second or 1/s) illustrating the variation of viscosity with shear rate of different concentrations of the spinning dispersion according to various embodiments. Similar to most complex fluid systems with rigid polymer chains, the viscosity of MXene nanosheets in spinning dispersions may decrease with an increasing shear rate, and may increase with an increasing concentration.

[0068] FIG. 9B shows a plot of shear stress (in Pascals or Pa) as a function of shear rate (in per second or 1/s) illustrating the variation of shear stress with shear rate of different concentrations of the spinning dispersion according to various embodiments. The shear stress of the spinning dispersion may decrease sharply at the initial stage and may then gradually increase with the shear rate. It suggests that the randomly oriented MXene nanosheets may turn into an ordered state because of the shear-induced deformation.

[0069] FIG. 9C shows a plot of modulus (in Pascals or Pa) as a function of frequency (in per second or 1/s) illustrating the variation of modulus with frequency of different concentrations of the spinning dispersion according to various embodiments. As shown in FIG. 9C, the ratio of the storage modulus to the loss modulus (G'/G'') of a spinning dispersion may be in the range from about 1.86 to about 6.01 with the concentration of about 30 mg mL^{-1} to about 15 mg mL^{-1} , acting as an indicator for the spinnability of liquid-crystalline MXene nanosheets colloidal dispersions. In this process, MXene nanosheets may initially crosslink with GA at a concentration of 30 mg mL^{-1} to form MXene-GA (MG) fibers, and sequentially crosslinked with polyvinyl alcohol (PVA) to fabricate several meters long MXene-GA-PVA (MGP) fibers with a diameter of $\sim 60 \text{ }\mu\text{m}$ by wet spinning. FIG. 10A shows a line drawing of several meter long wet spun (MGP) fibers according to various embodiments; and (inset) a magnified line drawing showing the axial SEM morphology of a wet spun (MGP) fiber according to various embodiments.

[0070] Fourier transform infrared spectroscopy (FTIR) spectra show a new peak at $\sim 842.1 \text{ cm}^{-1}$ for the MG and MGP fibers, indicating the formation of Ti-O-C bond between MXene nanosheets and GA molecules. FIG. 10B shows a plot of intensity (in arbitrary units or a.u.) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the obtained fibers according to various embodiments, glutaraldehyde (GA), polyvinyl alcohol (PVA) and pure MXene. FIG. 10C shows a plot of reflectance (in percent or %) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the obtained fibers according to various embodiments, glutaraldehyde (GA), polyvinyl alcohol (PVA) and pure MXene. FIG. 10D shows a plot of reflectance (in percent or %) as a function of wavenumbers (per centimeter or cm^{-1}) showing the Fourier transform infrared (FTIR) spectra of the wet spun (MGP) fibers with different weight ratios of polyvinyl alcohol (PVA) according to various embodiments.

[0071] The disappearance of peak of $\sim 1,717.5 \text{ cm}^{-1}$ for the aldehyde group ($-\text{CHO}$) of GA molecules may confirm the formation of the Ti-O-C bond. The peak of $-\text{OH}$ of MXene nanosheets may have shifted from $\sim 3,651.2 \text{ cm}^{-1}$ to the low wavenumber of $\sim 3,515.6 \text{ cm}^{-1}$ and becomes wider due to the hydrogen bond between $-\text{OH}$ of MXene nanosheets and $-\text{OH}$ of PVA molecules. Furthermore, X-ray photoelectron spectroscopy (XPS) spectra exhibit no existence of Al element for MXene nanosheets in pure MXene, MG, and MGP fibers compared to that of Ti_3AlC_2 , suggesting the successful preparation of MXene nanosheets. FIG. 11 shows a plot of intensity (in arbitrary units or a.u.) as a function of wavenumbers (per centimeter or cm^{-1}) showing the X-ray photoelectron spectroscopy (XPS) spectra of the fabricated fibers according to various embodiments, pure MXene and MAX (Ti_3AlC_2). The peaks for Ti and the disappeared Al peak may indicate that MXene nanosheets have been successfully etched from the primitive MAX (Ti_3AlC_2).

[0072] FIG. 12A shows a plot of intensity (in arbitrary units or a.u.) as a function of binding energy (in electron volts or eV) showing the titanium (Ti) 2p spectra of the fabricated fibers according to various embodiments and pure MXene fibers. FIG. 12B shows a plot of intensity (in arbitrary units or a.u.) as a function of binding energy (in electron volts or eV) showing the oxygen (O) 1s spectra of the fabricated fibers according to various embodiments and pure MXene fibers. FIG. 12C shows a table illustrating the atomic percentage of O-Ti-O, C-Ti-OX, O-C, and C-Ti-OH according to the oxygen (O) 1s peak in the obtained X-ray photoelectron spectroscopy (XPS) spectra of the obtained fibers according to various embodiments and pure MXene fibers.

[0073] As shown in FIG. 12A, a new peak at ~456.4 eV for MGP and MG fibers indicates the formation of Ti-O-C between MXene nanosheets and GA compared with pure MXene fibers according to Ti 2p spectra. The atomic percentage for O-C for MG and MGP fibers is increased to 16.4% and 22.0%, compared to pure MXene fibers of 6.2% in O 1s spectra, indicating the formation of Ti-O-C bond between MXene nanosheets and GA, and hydrogen bonding between MXene nanosheets and PVA. Hence, compact MXene fibers may be fabricated via interfacial interactions, which are firstly crosslinked with GA via Ti-O-C bond of nucleophilic substitution (FIG. 13A), and then crosslinked with PVA via hydrogen bonding between MXene nanosheets and PVA (FIG. 13B)

[0074] FIG. 13A illustrates one possible mechanism for the formation of Ti-O-C covalent bond between MXene nanosheets and glutaraldehyde molecules according to various embodiments. In FIG. 13A(a), the aldehyde group (-CHO) of glutaraldehyde (GA) molecules may react with hydroxyl functional group (OH) of a MXene nanosheet to form Ti-O-C covalent bond via nucleophilic substitution and dehydration reaction. FIG. 13A(b) shows the corresponding structure schematic of formed Ti-O-C bond between MXene nanosheets and glutaraldehyde molecules. FIG. 13B shows a structure graph of the covalent bond between

MXene nanosheets and glutaraldehyde (GA) and the hydrogen bond between MXene nanosheets and polyvinyl alcohol (PVA) according to various embodiments.

[0075] Mechanical properties of MXene fibers via wet spinning

[0076] As a result of the interfacial interactions, the alignment of MXene fibers is significantly enhanced, and the porosity is largely reduced, according to the wide-angle X-ray scattering/small-angle X-ray scattering (WAXS/SAXS) patterns. FIG. 14A shows (a) wide-angle X-ray scattering (WAXS) patterns of the fabricated intermediate (MG) fibers with different weight ratios of glutaraldehyde molecules from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments; and (b) small-angle X-ray scattering (SAXS) patterns of the fabricated intermediate (MG) fibers with different weight ratios of glutaraldehyde molecules from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments.

[0077] FIG. 14B shows a plot of orientation order (f) as a function of percentage of glutaraldehyde (in weight percent or wt%) showing the variation of the orientation of the intermediate (MG) fibers with different amounts of glutaraldehyde based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments. FIG. 14C shows a plot of density (in grams per cubic centimeter) / porosity (in percent or %) as a function of percentage of glutaraldehyde (in weight percent or wt%) showing the variation of density and porosity of the intermediate (MG) fibers with different amounts of glutaraldehyde according to various embodiments. The results show that MG fibers with 5 wt% glutaraldehyde may have achieved the highest orientation order (f) and the lowest porosity.

[0078] FIG. 15 shows the wide-angle X-ray scattering (WAXS) and (inset) small-angle X-ray scattering (SAXS) patterns of the fabricated wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments.

[0079] FIG. 16A shows a plot of orientation order (f) as a function of percentage of polyvinyl alcohol (PVA) (in weight percent or wt%) showing the variation of the orientation of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments. FIG. 16B shows a plot of density (in grams per cubic centimeter) / porosity (in percent or %) as a function of percentage of polyvinyl alcohol (in weight percent or wt%) showing the variation of density and porosity of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) according to various embodiments. FIG. 16C shows a plot of tensile strength (in mega-Pascals or MPa) / toughness (in mega-Joules or MJ m⁻³) as a function of percentage of polyvinyl alcohol (PVA) (in weight percent or wt%) showing the variation of strength and toughness of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) according to various embodiments.

[0080] FIG. 17A shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or Å⁻¹) illustrating the variation of intensity of the intermediate (MG) fibers with different amounts of glutaraldehyde from 0 weight percent (wt%) to 20 weight percent (wt%) according to various embodiments. FIG. 17B shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or Å⁻¹) illustrating the variation of intensity of the wet spun (MGP) fibers with different amounts of polyvinyl alcohol (PVA) from 1 weight percent (wt%) to 15 weight percent (wt%) according to various embodiments. FIG. 17C shows a plot of intensity (in arbitrary units or a.u) as a function of scattering vector q (per Angstrom or Å⁻¹) illustrating the variation of intensity of the various fabricated fibers according to various embodiments.

[0081] FIG. 18 shows a plot of weight loss (in percent or %) as a function of temperature (in degrees Celsius or °C) illustrating the thermogravimetric analysis (TGA) results of polyvinyl alcohol (PVA), glutaraldehyde (GA) and various fabricated fibers according to various

embodiments. FIG. 19 shows a table illustrating the orientation order (f) and porosity (in percent or %) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments. FIG. 20 shows a table illustrating the orientation order (f) and porosity (in percent or %) of the fabricated wet spun (MGP) fibers with different weight percentage of polyvinyl alcohol (PVA) molecules according to various embodiments. FIG. 21 shows a table illustrating the tensile strengths (in mega-Pascals or MPa), strain (in percent or %) and toughness (in mega-Joules per cubic meter or MJ m⁻³) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments.

[0082] As shown in FIGS. 13, 14A-B, 18 and 19, when MXene nanosheets are crosslinked with GA via Ti-O-C covalent bond, the orientation orders (f) of the obtained MG fibers may increase to ~ 0.85 with ~ 5 wt% GA and may decrease to ~ 0.72 with 20 wt% GA compared with the orientation order of ~ 0.82 for pure MXene fibers. Moreover, the porosity of fibers reduces to 14.1% with ~ 5 wt% GA lower than that of $\sim 17.2\%$ for pure MXene fibers. This is because that more GA molecules are hindered in the inter-layer, resulting in the appearing wrinkles and voids according to the increasing intensity of SAXS patterns (FIG. 17A).

[0083] After PVA cross-linking with MXene nanosheets into MGP fibers, the f is indeed enhanced to ~ 0.87 , higher than that of pure MXene fibers and MG fibers (FIGS. 15, 16A, 20). The orientation orders of MGP fibers are increased with the increment of PVA up to 5%, while are decreased to ~ 0.85 with further increasing of PVA. Furthermore, the porosity is significantly reduced to $\sim 7.4\%$ to achieve a high density of ~ 3.68 g cm⁻³ via hydrogen bond of PVA (FIG. 16B). PVA with more than 5 wt% may lead to the increase of wrinkles of MXene nanosheets and voids between MXene nanosheets, which is testified by the increasing intensity of SAXS patterns for MGP fibers (FIG. 17B). Therefore, the compact MGP fibers may be achieved with

low porosity than MG and MXene fibers according the decreased intensity of SAXS patterns (FIG. 17C).

[0084] FIG. 22A is a plot comparing the tensile strengths and toughness of fabricated intermediate (MG) fibers with different weight percentages of glutaraldehyde molecules from 0 wt% to 20 wt% according to various embodiments. FIG. 22B is a plot comparing the electrical conductivity of fabricated intermediate (MG) fibers with different weight percentages of glutaraldehyde molecules from 0 wt% to 20 wt% according to various embodiments. FIGS. 22A-B show that the MG fibers containing 5 wt% glutaraldehyde molecules may have maximum tensile strengths, toughness, and conductivity.

[0085] FIG. 23A shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of pure MXene fibers according to various embodiments. FIG. 23B shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 2 weight percent (2 wt%) glutaraldehyde molecules according to various embodiments. FIG. 23C shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 5 weight percent (5 wt%) glutaraldehyde molecules according to various embodiments. FIG. 23D shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 10 weight percent (10 wt%) glutaraldehyde molecules according to various embodiments. FIG. 23E shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 15 weight percent (15 wt%) glutaraldehyde molecules according to various embodiments. FIG. 23F shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 20 weight percent (20 wt%) glutaraldehyde molecules according to various embodiments.

[0086] Due to the high alignment and low porosity achieved by the interfacial interactions, the prepared MG fibers have a higher tensile strength of ~ 335.6 MPa with the toughness of ~ 4.0 MJ m⁻³, compared to that of ~ 167.1 MPa and ~ 0.4 MJ m⁻³ for pure MXene fiber via Ti-O-C covalent bond between MXene nanosheets and GA molecules (FIGS. 21, 22A, 23A-F). When the GA weight percentage is more than 5 wt%, both tensile strength and toughness decrease for MG fibers, which may be attributed to hindering stress transfer with a higher weight percentage of GA between MXene nanosheets. FIG. 24 shows a table illustrating the electrical conductivity (in siemens per centimeter or S cm⁻¹) of the fabricated intermediate (MG) fibers with different weight percentage of glutaraldehyde (GA) molecules according to various embodiments. FIGS. 22B, 24 show that the conductivity of MG fibers decreases from $\sim 11,360.4$ S cm⁻¹ to $\sim 4,536.4$ S cm⁻¹, due to the introduction of non-conductive GA molecules into the MXene nanosheets. However, MG fibers with 5 wt% GA can still keep a high conductivity of $\sim 9,860.6$ S cm⁻¹, because of the high alignment and low porosity of MG fibers.

[0087] FIG. 25A shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of intermediate (MG) fibers with 5 weight percent (5 wt%) glutaraldehyde molecules according to various embodiments. FIG. 25B shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 1 weight percent (1 wt%) polyvinyl alcohol (PVA) according to various embodiments. FIG. 25C shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 2 weight percent (2 wt%) polyvinyl alcohol (PVA) according to various embodiments. FIG. 25D shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 5 weight percent (5 wt%) polyvinyl alcohol (PVA) according to various embodiments. FIG. 25E shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %)

illustrating the stress-strain curves of wet spun (MGP) fibers with 10 weight percent (10 wt%) polyvinyl alcohol (PVA) according to various embodiments. FIG. 25F shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of wet spun (MGP) fibers with 15 weight percent (15 wt%) polyvinyl alcohol (PVA) according to various embodiments. FIG. 25G shows a plot of stress (in mega-Pascals or MPa) as a function of strain (in percent or %) illustrating the stress-strain curves of pure polyvinyl alcohol (PVA) fibers. FIG. 26 shows a table illustrating the tensile strengths (in mega-Pascals or MPa), strain (in percent or %) and toughness (in mega-Joules per cubic meter or MJ m⁻³) of various fibers with different weight percentage of polyvinyl alcohol (PVA) according to various embodiments.

[0088] After being subsequently crosslinked with PVA molecules, the tensile strength and toughness are further enhanced to ~565.2 MPa and ~19.2 MJ m⁻³ due to the significant improvement of alignment and decrease of porosity via the synergistic interfacial interactions of the covalent bonds and hydrogen bonds, shown in FIGS. 6C, 25A-G, 26. In addition, the mechanical properties of MGP fibers may be weakened by increasing the weight percentage more than ~5 wt% PVA due to the hindering effect.

[0089] FIG. 27 is a plot comparing the electrical conductivity of MXene fibers, intermediate (MG) fibers, and wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments. FIG. 28 shows a table illustrating the electrical conductivity (in siemens per centimeter or S cm⁻¹) of intermediate fibers and wet spun (MGP) fibers with different weight percentages of polyvinyl alcohol (PVA) according to various embodiments.

[0090] Although the conductivity of MGP fibers slightly decreases with the increment of non-conductive PVA, MGP fiber with ~5 wt% PVA still offers a high conductivity of ~8,110.4 S cm⁻¹, thanks to the compactness via synergistically interfacial interactions (FIGS. 27 - 28).

Consequently, compact MGP fibers are successfully achieved with the compact morphology, exhibiting higher performance than pure MXene and MG fibers. FIG. 29 shows scanning electron microscopy images of the cross-sections of intermediate (MG) fibers with different weight percentages (wt%) of polyvinyl alcohol (PVA) from 0 wt% to 20 wt% according to various embodiments. FIG. 30 shows scanning electron microscopy images of the cross-sections of wet spun (MGP) fibers with different weight percentages (wt%) of polyvinyl alcohol (PVA) from 1 wt% to 15 wt% according to various embodiments.

[0091] In-situ characterization of MXene fibers being heated

[0092] Then, the heating process induced property change of MGP fibers was studied at various heating temperatures by in-situ XRD patterns. When an MGP fiber is heated from 25 °C to 350 °C, the 002 pattern is shifted to high q with the d-spacing of MXene nanosheets down to 11.61 Å, according to the in-situ X-ray diffraction (XRD) patterns. FIG. 31A shows a plot of temperature (in degree Celsius or °C) as a function of scattering vector (per Angstrom or Å⁻¹) illustrating the in-situ X-ray diffraction (XRD) patterns of a wet spun (MGP) fiber according to various embodiments. FIG. 31B shows a plot of d-spacing (in Angstroms or Å) as a function of temperature (in degree Celsius or °C) illustrating the d-spacing curve according to the in-situ X-ray diffraction (XRD) pattern according to various embodiments. FIG. 31C shows a plot of intensity (in arbitrary units or a.u.) as a function of scattering vector q (per Angstrom or Å⁻¹) showing the in-situ X-ray diffraction (XRD) patterns of fabricated wet spun (MGP) fibers being heated under various temperatures from 25 °C to 350 °C according to various embodiments. The results showed the d-spacing between MXene nanosheets was decreased with the increase of heating temperature.

[0093] Moreover, as shown in FIG. 31D, the porosity increases sharply from 7.7% to 16.7% when increasing the heating temperature, indicating that the voids and wrinkles between MXene nanosheets are generated when heating. FIG. 31D shows a plot of porosity (in percent

or %) as a function of temperature (in degree Celsius or °C) illustrated the porosity of wet spun (MGP) fibers being heated according to various embodiments. FIG. 32A shows a schematic diagram of wet spun (MGP) fibers being heated according to various embodiments.

[0094] In addition, the in-situ SAXS patterns were also conducted to study the heating process of fibers. FIG. 32B shows the in-situ small-angle X-ray scattering (SAXS) patterns of fabricated wet spun (MGP) fibers (with 5% polyvinyl alcohol (PVA)) being heated under variable temperatures from 25°C to 350 °C according to various embodiments. FIG. 32C shows a plot of intensity as a function of scattering vector q (per Angstrom or $\text{\AA}^{-1}$) illustrating the intensity curves of wet spun fibers under different temperatures based on the small-angle X-ray scattering (SAXS) patterns according to various embodiments.

[0095] The increasing intensity of SAXS patterns for MGP fibers may confirm the generation of voids and wrinkles of MXene nanosheets when increasing annealing temperature. The voids may mainly be generated via the removal of intercalated water and partial hydroxyl surface terminations at elevated temperatures.

[0096] Fabrication of ultra-compact MXene fibers via thermal drawing

[0097] In order to further enhance the compactness of MXene fibers, thermal drawing enabled physical compression was developed. More importantly, such a continuous drawing process may offer a path to protect the resulting ultra-compact MXene fibers with an in-situ formed polymer layer, resulting in a packaged fiber device to directly engage various applications. In the process of thermal drawing, a hollow polycarbonate (PC) tube passed through the hot zone of the drawing furnace, while a MGP fiber was continuously fed into the PC tube from the top of it (see FIG.3A). FIG. 33A shows a schematic diagram of forming the cross-linked MXene-based (MGP-T) fibers with protective layers from wet spun (MGP) fibers with axial stress and perpendicular stress during thermal drawing according to various embodiments. As shown in FIG. 33A, the MGP fibers in the PC tube may generate voids and

wrinkles when the whole tube entered the hot zone at the central temperature of ~ 200 °C, which was proved by the in-situ XRD and SAXS patterns. However, when thermal drawing started, the generated voids and wrinkles were significantly reduced by the drawing-induced stresses, including both axial stress and perpendicular stress. Meanwhile, the MGP fibers with an outer protective layer (MGP-T) were formed in-situ to protect the resulting ultra-compact MXene fibers. As a result, several meters long MGP-T fibers were successfully fabricated by this strategy with a diameter of ~ 75 μm . FIG. 33B shows a line drawing of several meters long cross-linked MXene-based fibers (MGP-T) fibers with protective layers according to various embodiments. FIG. 33C shows another line drawing of cross-linked MXene-based (MGP-T) fibers with protective layers according to various embodiments.

[0098] Under this controllable and continuous process, various MGP-T fibers were fabricated by increasing the draw-down ratio of pull to feed speed of 1.26, 1.34, and 1.41, forming MGP-T₁, MGP-T₂, and MGP-T₃, respectively. FIG. 34A is a plot comparing the orientation orders (f) of three cross-linked MXene-based (MGP-T) fibers with protective layers fabricated by various draw-down ratios according to various embodiments, with the inset showing the wide-angle X-ray scattering (WAXS) pattern of the various fibers according to various embodiments. FIG. 34B shows a plot of intensity (in arbitrary units or a.u.) as a function of azimuthal angle (in degrees or °) showing the variation across fibers formed by different draw-down ratios based on wide-angle X-ray scattering (WAXS) patterns according to various embodiments. FIG. 34C shows a table comparing the orientation order (f) and porosity (in percent or %) of the fabricated MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios according to various embodiments.

[0099] As a result of the induced stress parallel to the axial direction in the thermal drawing process, the alignment of MGP fibers may be enhanced from ~ 0.84 to ~ 0.89 when increasing the draw-down ratio, according to the WAXS patterns presented in FIGS. 34A-C.

Simultaneously, the stress perpendicular to the axis may effectively compress the MGP fibers and reduce the porosity from ~8.6% to ~5.7% during the thermal drawing. FIG. 35A shows a plot of porosity (in percent or %)/conductivity (in $\times 10^3$ siemens per centimeter or $S\text{ cm}^{-1}$) comparing the porosity and electrical conductivity of the various fabricated MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios according to various embodiments. FIG. 35B shows the scanning electron microscopy (SEM) cross-sectional images of the MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios (a: MGP-T₁, b: MGP-T₂, c: MGP-T₃) with energy-dispersive X-ray spectroscopy (EDX) according to various embodiments. The results showed that MGP-T fibers get more compact with the increase of the draw-down ratio from MGP-T₁ to MGP-T₃, which may significantly reduce the porosity and enhance the alignment of fibers. The EDX mapping of the cross-sections of the MGP-T fibers clearly show that the fibers become more compact with low porosity when increasing the draw-down ratio.

[00100] In addition, the high resolution transmission electron microscopy (HR-TEM) images of MGP-T fibers confirm the high alignment of alternative MXene layers and PVA/GA polymer layers. FIG. 36A shows a high resolution transmission electron microscopy (HR-TEM) image of a MXene-based fiber with protective layer (MGP-T) according to various embodiments. FIG. 36B shows a zoomed in view of the area indicated in FIG. 36A according to various embodiments. The results showed that the PVA/GA polymers (light color) were successfully introduced into the $Ti_3C_2T_x$ layers (dark color) to form MGP-T fibers with high alignment. FIG. 36C shows the EDX spectrum of the selected area shown in FIG. 36A according to various embodiments. The EDX spectrum suggests the existence of O, C, and Ti element of MXene ($Ti_3C_2T_x$) nanosheets, PVA, and GA polymers.

[00101] Therefore, the ultracompact MGP-T fibers with in-situ generated protective layer were achieved with a large enhancement of the alignment and reduction of the porosity, which

was induced by the controllable thermal drawing stresses. As a result, the conductivity of the inner ultra-compact MGP fibers is promoted from $\sim 8,344.5 \text{ S cm}^{-1}$ to $\sim 8,802.4 \text{ S cm}^{-1}$ in FIG. 35A and FIG. 37. FIG. 37 shows a table illustrating the electrical conductivity of the fabricated MXene-based fibers with protective layers (MGP-T) formed using various draw-down ratios according to various embodiments.

[00102] Meanwhile, MGP-T fibers may exhibit ultra-high tensile strength of $\sim 585.5 \text{ MPa}$ and toughness of $\sim 66.7 \text{ MJ m}^{-3}$ due to the promoted alignment and reduction of porosity during the thermal drawing process with increasing stresses. FIG. 38A shows a plot of tensile strength (in mega-Pascals or MPa)/toughness (in mega-Joules per cubic meter or MJ m^{-3}) comparing the tensile strength and toughness of the various fabricated MXene-based fibers with protective layers (MGP-T) formed using different draw-down ratios according to various embodiments. FIG. 38B shows a table illustrating the tensile strength (in mega-Pascals or MPa) and toughness (in mega-Joules per cubic meter or MJ m^{-3}) of the fabricated MXene-based fibers with protective layers (MGP-T) formed using various draw-down ratios according to various embodiments and polycarbonate (PC).

[00103] FIG. 39A shows a plot of tensile strength (in mega-Pascals or MPa) as a function of electrical conductivity (in $\times 10^3$ siemens per centimeter or S cm^{-1}) comparing the tensile strength and electrical conductivity of the obtained compact MXene-based fibers according to various embodiments with other reported MXene-based fibers. FIG. 39B shows a table comparing the tensile strength (in mega-Pascals or MPa), toughness (in mega-Joules per cubic meter or MJ m^{-3}) and electrical conductivity (in siemens per centimeter or S cm^{-1}) of the obtained compact MXene-based fibers according to various embodiments with that of other reported MXene-based fibers. As summarized in FIGS. 39A-B, various embodiments may offer the highest performance on both tensile strength and conductivity, compared with the reported MXene fibers without protective layers. Especially, the toughness of the ultra-compact

MGP-T fibers may reach $\sim 66.7 \text{ MJ m}^{-3}$, higher than that of the reported MXene-based fibers, graphene-based fibers, and CNT fibers via different preparation strategies (FIGS. 39B, 40A-B).

[00104] FIG. 40A shows a plot of toughness (in mega-Joules per cubic meter or MJ m^{-3}) as a function of electrical conductivity ((in siemens per centimeter or S cm^{-1}) comparing the toughness and electrical conductivity of the ultra-compact MXene fibers according to various embodiments with that for other reported MXene-based fibers, graphene fibers, and carbon nanotube (CNT) fibers. FIG. 40B shows a table comparing the tensile strength (in mega-Pascals or MPa), toughness (in mega-Joules per cubic meter or MJ m^{-3}) and electrical conductivity (in siemens per centimeter or S cm^{-1}) of the obtained compact MXene-based fibers according to various embodiments with that of other reported carbon -based fibers.

[00105] Finite element analysis for thermal drawing process

[00106] With the combined effects of the stress parallel to the axial and compressing stress perpendicular to the axial of fibers induced by the thermal drawing, MGP-T fibers with high alignment and low porosity have been achieved, which further enhance their mechanical and electrical properties. In order to understand the role of drawing-induces stresses in formation of ultra-compact MGP-T fibers, finite element analysis was conducted by the Abaqus software.

[00107] During the full procedure of thermal drawing, the compressing effect existed when the polycarbonate (PC) hollow tube just started to contact the MGP-T fibers with a diameter of $\sim 60 \text{ }\mu\text{m}$. Therefore, the compressed mechanical behavior of PC hollow tube was studied when it passed through the drawing furnace. A PC hollow tube model with an inner diameter of 0.06 mm, an outer diameter of 0.08 mm, and 0.50 mm long was constructed to study the mechanical behavior of PC hollow tube.

[00108] FIG. 41 shows (a) a schematic diagram of the apparatus used for the thermal drawing according to various embodiments; and (b) a polycarbonate (PC) hollow tube model with an

inner diameter of 0.06 mm, an outer-diameter of 0.08 mm, and a length of 0.50 mm long constructed to study the mechanical behavior of the PC hollow tube via finite element analysis according to various embodiments.

[00109] When the drawing force was applied on the bottom of the PC hollow tube during the thermal drawing, the PC model was stretched with the reduction of the diameter of the PC hollow tube. FIG. 42A shows a simulation of the stress distribution of the polycarbonate (PC) hollow tube model according to various embodiments. According to the simulation results as shown in FIG. 42A, the stress parallel to the axial (Y) may be generated accompanied by compressing stress perpendicular to the axial (Z) of PC hollow tube. Therefore, the alignment of inner MGP fibers may be promoted due to axial stress, while the porosity may be reduced because of the perpendicular stress.

[00110] When increasing the draw-down ratio in simulation, both Y drawing stress and Z compressing stress may be increased, according to the results of finite element analysis shown in FIG. 42B. FIG. 42B shows plots of stress (in mega-Pascals or MPa) as a function of draw-down ratio illustrating the Y and Z stress curves with the increment of the draw-down ratio according to finite element analysis according to various embodiments. The increasing stresses may further enhance the alignment and reduce the porosity, resulting in ultra-compact MGP-T fibers with high mechanical and electrical properties. These results agree well with the results of the experiments.

[00111] Large-scale wearable MXene textiles for electromagnetic interference shielding and electrothermal applications

[00112] The rapid development of telecommunication and the ever-increasing usage of portable electronic devices may result in serious electromagnetic problems and impact on health of human beings. Therefore, it may be important to fabricate functional textiles to protect humans from electromagnetic waves. Due to the excellent properties of MXene nanosheets, the

obtained ultra-compact MGP-T fibers may not only offer high toughness, tensile strength, and electrical conductivity, but may also provide potentially useful electromagnetic interference (EMI) shielding performance with high shielding efficiency (SE). FIG. 43A shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) as a function of frequency (in gigahertz or GHz) illustrating the EMI reflection shielding efficiency (SE_R), absorption shielding efficiency (SE_A) and total efficiency (SE_T) of the textiles according to various embodiments at the frequencies of 8.2 GHz – 12.4 GHz.

[00113] FIG. 43B is a schematic illustrating plain-weaved textiles based on MXene-based fibers according to various embodiments for electromagnetic interference (EMI) measurements. The as-synthesized MGP and MGP-T fiber textiles show a SE_T of ~50 dB and ~57 dB, compared to that of pure MXene fiber textile (~74 dB) at the frequency of 8.2 GHz, because the electrical conductivity of MGP and MGP-T fibers is lower than that of pure MXene fibers. However, these results show that the SE_A (such as ~60 dB, ~47 dB, and ~40 dB) may be higher than SE_R (~15 dB, ~10 dB, and ~10 dB) for pure MXene, MGP fiber, and MGP-T fiber textiles, respectively. Therefore, the EMI shielding may mainly be dependent on the absorption mechanism. The EMI SE of MGP-T fibers textiles fabricated via increasing draw-down ratios is also summarized in FIGS. 44A-C.

[00114] FIG. 44A shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) comparing the reflection shielding efficiency (SE_R), absorption shielding efficiency (SE_A) and total efficiency (SE_T) of textiles fabricated from the MXene-based fibers with protective layers (MGP-T) obtained using various draw-down ratios according to various embodiments at the frequency of 8.2 GHz. FIG. 44B shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) as a function of frequency (in gigahertz or GHz) illustrating the electromagnetic interference (EMI) properties of 3-layered textiles fabricated using various MXene-based fibers with protective layers (MGP-T) according

to various embodiments at frequencies from 8.2 GHz to 12.4 GHz. FIG. 44C shows a plot of electromagnetic interference shielding efficiency (EMI SE, in decibels or dB) comparing the electromagnetic interference (EMI) properties of 3-layered textiles fabricated using various fabricated and pure MXene fibers according to various embodiments at the frequency of 8.2 GHz. The MGP-T fibers textiles may exhibit a higher SE_A than SE_R . The SE_T of MGP-T₁ changes from ~52 dB to ~61 dB of MGP-T₃, accompanied with an increment of SE_A from ~42 dB to ~51 dB at the shielding frequency of 8.2 GHz. The increase may be attributed to the reduction of porosity and enhancement of alignment of MGP-T fibers when applying the high draw-down ratio. However, there is no obvious increment of SE_R for MGP-T fibers textiles, as the major EMI mechanism is absorption. Also, the MGP-T fibers textiles may show higher SE_A and SE_T , compared with these of MGP fiber textiles, due to the enhanced compactness of fibers during the thermal drawing procedure.

[00115] FIG. 45A is a schematic illustrating the electromagnetic interference (EMI) mechanism of textiles fabricated using the MXene-based fibers with protective layers (MGP-T) according to various embodiments.

[00116] As shown in FIG. 45A, the EMI shielding mechanism of MGP-T fibers may be explained as follows: when electromagnetic waves (EMWs) strike the surface of an MGP-T fiber, some EMWs may be immediately reflected at the surface because of enormous free electrons of the highly conductive MXene nanosheets. Then, the remaining waves may go through the lattice structure of MXene nanosheets, which interact with the high electron density of MXene nanosheets and induce currents to reduce the energy of the EMWs with the ohmic losses. After going through the first layer (i.e. first nanosheet) of MXene, the surviving EMWs may encounter the next MXene barrier layer with the repetition of EMW attenuation. Meanwhile, the second layer (i.e. the second nanosheet) as the surface reflects the surviving EMWs and results in multiple internal reflections. Finally, the EMWs may be reflected back-

and-forth and completely absorbed within the compact layers. Therefore, the compact structure may provide the MGP-T fibers with the ability to behave as a multi-level EMI shield. In addition, the textiles based on MGP-T fibers may maintain the remarkable bending stability with ~87.8% retention of EMI SE performance after 5×10^4 bending cycles. FIG. 45B shows a plot of electromagnetic interference shielding efficiency (EMI SE) retention (in percent or %) as a function of number of bending cycles illustrating the electromagnetic interference shielding efficiency retention of the textile according to various embodiments after 5×10^4 bending cycles.

[00117] Other than the high EMI shielding performance, the obtained MGP-T fibers may also show excellent electrothermal (ET) performance for human thermal management in wearable textiles. FIG. 46A shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating the temperature-time curves of single MXene-based fibers with protective layers (MGP-T) according to various embodiments when a direct current (DC) voltage of 6 V is applied. When applying the working DV voltage of 6 V, the MGP-T fibers may immediately generate thermal energy and reach the equilibrium temperature from ~90 °C to ~100 °C (from MGP-T₁ to MGP-T₃), owing to the mechanism of Joule heat. It may be attributed to the high compactness of MGP-T fibers with the low porosity and high alignment induced by thermal drawing under the high stress.

[00118] FIG. 46B shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating the temperature-time curves of a MXene-based fiber with protective layer (MGP-T) according to various embodiments when voltages from 2 V to 8 V are applied. FIG. 46B shows that after being applied with different voltages at a broad range of 2 V to 8 V, MGP-T₃ fibers can generate the heat with the temperature increasing up to ~130 °C, suggesting that the maximum temperature generated by the MGP-T fiber may be tunable by applying various DC voltages.

[00119] These results indicate that MGP-T fibers may provide excellent ET behavior for their Joule heating. Furthermore, a single MGP-T fiber may also show an excellent cycling life with performance retention of ~99% after 5,000 cycles as shown in FIG. 46C. FIG. 46C shows a plot of performance retention (in percent or %) as a function of the number of cycles illustrating the cycling life of a MXene-based fiber with protective layer (MGP-T) according to various embodiments, while the inset is a plot of temperature (in degrees Celsius or °C) as a function of time (in seconds or s) illustrating the temperature variation profile in the first and last few cycles according to various embodiments.

[00120] FIG. 46D shows images of various MXene-based fibers with protective layers (MGP-T) arranged in shapes of different letters upon application of various direct current (DC) voltages of 2 V to 8 V according to various embodiments. FIG 46D shows that, as an example to achieve the practical applications of MGP-T fibers for thermal management, fabrics with different shapes of the letters based on MGP-T fibers may effectively generate the heat by varying the applied voltages.

[00121] The mechanical durability of the MGP-T fibers may be considered for further applications. FIG. 46E shows a plot of temperature (in degree Celsius or °C) as a function of time (in seconds or s) illustrating temperature-time curves of a single MXene-based fiber with protective layer (MGP-T) according to various embodiments at the bending angles from 0° to 180°. As shown in FIG. 46E, the mechanical stability of a single MGP-T fiber was conducted when bending at the bending angle of 0° to 180°. The obtained stable temperature-time curves with the equilibrium temperature may be the same as the initial curve, according to the infrared (IR) images. Meanwhile, the MGP-T fibers may be tied into knots of various diameters from ~23.93 mm to ~2.17 mm, and the performance retention may be measured as shown in FIG. 46F. FIG. 46F shows a plot of performance retention (in percent or %) as a function of diameter (in millimeters or mm) illustrating the temperature performance retention of the knots of

MXene-based fibers with protective layers (MGP-T) according to various embodiments. The performance of ET behavior remained stable evaluated through IR images. Particularly, the obtained MGP-T fibers could keep the temperature performance retention of ~81% after suffering from $\sim 1.1 \times 10^5$ bending cycles as shown in FIG. 46G. FIG. 46G shows a plot of performance retention (in percent or %) as a function of the number of bending cycles illustrating the temperature performance retention of a single MXene-based fiber with protective layer (MGP-T) according to various embodiments after 1.1×10^5 bending cycles from the bending angles of 0° to 180° .

[00122] Furthermore, 18-meter long ultra-compact MGP-T fibers were woven into a piece of cotton cloth with a length of 2.0 meters and width of 0.6 meters, enabling the large-scale applications, especially acting as wearable functional textiles for thermal management and EMI shielding. FIG. 47A shows a line drawing of the piece of cotton cloth with the size of 0.6 m by 2.0 m woven by 18 meter-long ultra-compact MXene-based fibers with protective layers (MGP-T) according to various embodiments. FIG. 47B shows an enlarged line drawing of a portion of the cloth shown in FIG. 47A according to various embodiments.

[00123] Also, some designed patterns were easily woven into the cotton cloth using the flexible MGP-T fibers and remained the performance even under complex deformations, indicating the good stitchability for the applications. FIG. 47C shows a line drawing of designed patterns woven into the cotton cloth according to various embodiments; and (inset) the cloth according to various embodiments being subjected to complex deformation.

[00124] FIG. 47D shows a line drawing of a sweater woven with several MXene-based fibers with protective layers (MGP-T) according to various embodiments to generate heat.

[00125] FIG. 47E shows a thermal image of the sweater in FIG. 47D according to various embodiments when a potential difference of 2 V is applied. FIG. 47F shows a thermal image of the sweater in FIG. 47D according to various embodiments when a potential difference of 4V

is applied. FIG. 47G shows an enlarged line drawing of a portion of the sweater shown in FIG. 47D according to various embodiments.

[00126] FIGS. 47D – G show a sweater with several MGP-T fibers that could quickly generate the heat with the temperature of ~35 °C and ~70 °C by applying the DC voltages of 2 V and 4 V for wearable human thermal management. Each fiber may be ~30 cm in length. The obtained results indicate that the ultra-compact MGP-T fibers may be very suitable for constructing smart textiles with excellent ET behavior and remarkable mechanical durability.

[00127] Various embodiments may relate to an effective and continuous strategy to fabricate ultra-compact MXene fibers with an in-situ generated protective layer. The fibers may be developed via the combination of wet spinning and thermal drawing. Due to the interfacial interactions and drawing-induced stresses, the alignment of MGP-T fibers may be significantly enhanced together with the effective reduction of porosity of MXene nanosheets. Consequently, the resulting ultra-compact MGP-T fibers may show excellent mechanical and electrical properties. In addition, the MGP-T fibers may not only offer remarkable electromagnetic interference shielding performance but may also have excellent electrothermal performance with ultra-stable mechanical durability. Meanwhile, the woven textiles based on MGP-T fibers (for large scale applications) may work properly even under complex deformations. These results demonstrate that the strategy may provide an effective means to fabricate compact fibers with multiple functions, paving a way for smart textiles. Various embodiments may generally be applied to the fabrication of other materials to construct the compact fibers with remarkable properties.

[00128] Methods

[00129] Materials

[00130] Ti_3AlC_2 powders (particle size ~ 400 mesh) were obtained from Jilin 11 Technology Co., Ltd. Lithium fluoride, ammonium chloride, ammonium hydroxide solution (28.0-30.0

wt%), and glutaraldehyde solution (25 wt%) were purchased from Sigma-Aldrich Co., Ltd. Polyvinyl alcohol (PVA) with a molecular weight of $MW \approx 130,000$ was purchased from Sigma-Aldrich. All materials were used as received. PVA solutions used for wet spinning were prepared by dissolving 15 g of PVA powder in 1 L of 95 °C deionized water under vigorous stirring for 12 hours. Polycarbonate (PC) was purchased from Goodfellow Co., Ltd.

[00131] Preparation of MXene nanosheets dispersion

[00132] The solutions with MXene nanosheets were prepared as follows. 1.8 g Ti_3AlC_2 powders were added to 40 mL solution (9 M HCl) that contained 3.8 g of lithium fluoride (LiF). Then the solution was stirred at 45 °C for 30 hours. After complete reaction, the accordion-like MXene product was washed with 9 M HCl solution for three cycles and deionized water for about eight cycles. Each cycle involved 5 minutes of centrifugation at 3500 revolutions per minute (rpm). Until a supernatant solution of MXene nanosheets reached at $pH \approx 7$, the resulting sediments were dispersed into 200 mL deionized water with continuous vibration for 13 minutes. Next the solution was centrifuged at 1500 rpm for 30 minutes and the obtained supernatant solution was then centrifuged at 3500 rpm for 30 minutes to get sediments. Finally, the sediments were dispersed into deionized water to prepare various concentration dispersions of MXene nanosheets.

[00133] Preparation of MXene fibers via wet-spinning

[00134] MXene ($Ti_3C_2T_x$) nanosheets with different weight percentages of glutaraldehyde solution were used as spinning dispersion. The MXene spinning dispersion was placed in a syringe and extruded through the nozzle with the diameter of 250 μm into the prepared coagulant bath at a velocity of 300 $\mu L \text{ min}^{-1}$. The coagulant bath included ammonium chloride (12.5 g), ammonium hydroxide solution (5 mL), and deionized water (1 L). After that, the extruded MG fibers in the coagulate bath solution were transferred to a washing bath of deionized water, PVA solutions, and another washing bath of deionized water by rollers.

Especially, the MG fibers were prepared via adjusting the weight ratios of MXene nanosheets and glutaraldehyde. Furthermore, the MGP fibers with different weight of PVA were obtained by adjusting the rates of roller. Pure PVA fibers were prepared by the same wet spinning with the coagulant of acetone.

[00135] Preparation of ultra-compact MGP-T fibers via thermal drawing

[00136] The PC hollow column was prepared via the wrapped PC on the ceramic rod with the diameter of ~6.35 mm and stored in the vacuum oven at 160 °C for 2 hours. Then the compactness MXene-based fibers were fabricated by the thermal drawing process via placing the preform in a two-zone heating furnace, where the top and bottom zones were heated to 150 °C and 350 °C, respectively. The perform with PC hollow column was fed into the furnace with various draw-down ratio (τ) when the MGP fibers were fed into the top of hollow column. Finally, the ultra-compact fibers were collected labeled as MGP-T. Here, the draw-down ratio (τ) could be defined as $\tau = \sqrt{\frac{v_d}{v_f}}$, where v_d and v_f are the drawing speed and feeding speed respectively.

[00137] Characterization

[00138] SEM images were collected using a JEOL JSM-7600F Schottky field emission scanning electron microscopes at a voltage of 5 kV. High-resolution transmission electron microscope (HR-TEM) images were obtained using a JEOL 2010HR instrument and an acceleration voltage of 200 kV. AFM images were tested by a Bruker Dimension Icon. TGA results were obtained under nitrogen using a Q500 SDT from Thermal Instruments and a heating rate of 10 K min⁻¹ between 35 °C and 800 °C. XRD patterns were conducted using Cu-K α radiation and an XRD Bruker D8 Advance. The rheological behaviors of the MXene-glutaraldehyde spinning dispersions were investigated with a rheometer (Anton Paar MCR 501) under both steady shear and dynamic oscillatory conditions. The viscoelastic properties of the spinning dispersions were measured by measuring the storage and loss modulus as a function

of frequency from 0.1 to 100 rad s⁻¹. The strain amplitude remained at 0.1% with a gap of 1 mm at 25 °C for the frequency sweep. Polarized optical microscopy (POM) images were recorded to exhibit optical birefringence via Olympus BX51. FTIR spectra were recorded at room temperature with Diamond ATR by an FTIR Frontier from PerkinElmer. XPS spectra were obtained using an XPS Kratos AXIS Supra. Electrical conductivities were tested using a standard two-probe method with a Keithley 2700 source meter. WAXS/SAXS measurements were conducted on a SWAXS Xenocs Nanoinxider, which was equipped with a Cu-K α source (operated at 30 W) with a beam diameter of 200 to 800 μ m on SAXS (200 K) and WAXS (100 K) detectors. In-situ XRD variable temperature tests were also conducted with the Linkam temperature stage by a SWAXS Xenocs Nanoinxider. The cross-section morphology of MGPT fibers was obtained by Focused Ion Beam (FIB) with the equipment of ZEISS Crossbeam 540 FIB-SEM. Tensile stress-strain curves were measured on 20 mm long, 3 mm wide samples using a SUNS EUT4103X Tester at a loading rate of 0.3 mm min⁻¹ with a 10 N sensor at room temperature. The area of fibers was measured by SEM. The results for each fiber were evaluated from the average value of at least three samples.

[00139] Finite element analysis of the thermal drawing

[00140] The finite element model was conducted by the commercial software Abaqus 2019 with Abaqus/explicit. During the thermal drawing, the PC tube did not contact the MGP fibers at hot zone at the central temperature of ~200 °C. Instead, the MGP fiber contacted the PC tube at a temperature of ~160 °C in the drawing furnace according to the experiments. Here, the compressed mechanical behavior of PC hollow tube was the focus of research when the PC hollow tube and MGP fiber came into contact until passing through the drawing furnace at the temperature of 160 °C. In the simulation, due to the diameter of ~60 μ m for MGP fibers, the PC hollow tube model with an inner diameter of 0.06 mm, an outer diameter of 0.08 mm, and 0.50 mm length was constructed to study the mechanical behavior of PC hollow tube during

thermal drawing. The PC hollow tube was simulated based on the isotropic bulk modulus (E) of 0.18 GP and Poisson ratio (ν) of 0.35. The mechanical property of PC at the temperature of 160 °C was characterized by the direct mechanical analyzer (DMA) instrument (DMA Q800). The fixed boundary conditions were set at the top of the PC hollow tube, while the pulling force was applied at the bottom of the hollow. In order to study the mechanical behavior of various draw-down ratios for thermal drawing, increasing the pulling force was used as same as the conditions of the experiments. The material model was defined with isotropic elasticity-plasticity of equations (1) and (2) as follows:

$$\begin{Bmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ \gamma_{12} \\ \gamma_{13} \\ \gamma_{14} \end{Bmatrix} = \begin{bmatrix} 1/E & -\nu/E & -\nu/E & 0 & 0 & 0 \\ -\nu/E & 1/E & -\nu/E & 0 & 0 & 0 \\ -\nu/E & -\nu/E & 1/E & 0 & 0 & 0 \\ 0 & 0 & 0 & 1/G & 0 & 0 \\ 0 & 0 & 0 & 0 & 1/G & 0 \\ 0 & 0 & 0 & 0 & 0 & 1/G \end{bmatrix} \begin{Bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{12} \\ \sigma_{13} \\ \sigma_{23} \end{Bmatrix} \quad (1)$$

$$G = \frac{E}{2 \times (1 + \nu)} \quad (2)$$

where G , E , and ν are the shear modulus, Young's modulus, and Poisson's ratio, respectively.

[00141] Measurement for electromagnetic interference (EMI) shielding performance

[00142] The woven textiles, prepared by three layers of MXene-based fibers, were tested for EMI shielding in a DR-WX rectangular waveguide with using the ENA5071C network analyzer (USA) at the Xband frequency range of 8.2 GHz to 12.4 GHz. The MXene-based fibers were weaved into the rectangular shape textiles of 30 mm×16 mm for the test.

[00143] The textiles' ability to attenuate the energy of the incident electromagnetic waves was evaluated as EMI SE. As an electromagnetic radiation into electromagnetic interference shielding equipment, the absorption (A), reflection (R), and transmission (T) should add up to 1, exhibiting the shielding phenomenon, that is,

$$A + R + T = 1 \quad (3)$$

[00144] The corresponding reflection (R) and transmission (T) coefficients were directly obtained from the network analyzer in the form of scattering parameters (S_{22} , S_{11} , S_{21} , and S_{12}). Thereafter, the coefficients of R and T were calculated as follows:

$$R = |S_{22}|^2 = |S_{11}|^2 \quad (4)$$

$$T = |S_{21}|^2 = |S_{12}|^2 \quad (5)$$

[00145] As a result, the shielding reflection (SE_R) and absorption (SE_A) can be estimated according to the as coefficients of R and T follows:

$$SE_R = 10 \log \left(\frac{1}{1-R} \right) = 10 \log \left(\frac{1}{1-|S_{11}|^2} \right) \quad (6)$$

$$SE_A = 10 \log \left(\frac{1-R}{T} \right) = 10 \log \left(\frac{1-|S_{11}|^2}{|S_{21}|^2} \right) \quad (7)$$

[00146] In addition, the multiple internal reflections can be generally negligible as more than 15 dB. Hence, the total EMI SE_T is the contributions of reflection (SE_R) and absorption (SE_A), which can be evaluated as follows:

$$SE_T = SE_R + SE_A \quad (8)$$

[00147] Measurement for electrothermal effect

[00148] Electrothermal property of a single fabricated fibers and textiles was investigated using a DC power supply (MP5020D). Silver paste was used as a contact point to connect the MXene-based fibers and conductive copper wires to accelerate the test. The thermal images and temperature-time curves of the fibers were recorded by an IR thermal imaging instrument of FLIR A3255SC camera. Moreover, the electrical-thermal behavior of textiles was performed as same as that of single MXene-based fibers

[00149] Measuring electrical conductivity of fibers

[00150] The electrical conductivity of the fabricated MXene-based fibers was measured using a Keithley 2700 source meter with the two-point probe method. Silver paste was conducted as a contact point to connect the MXene fibers and conductive probes. The conductivity (ρ) of the

measured MXene fibers was calculated using $\rho = \frac{L}{SR}$, where L is the length of the MXene fibers, R is the electrical resistance, and S is the crosssection area of each measured fibers from SEM images.

[00151] Estimation of the orientation order parameters of MXene-based fibers

[00152] Quantification of the WAXS/SAXS patterns was conducted with scattering vector q (defined as $q=4\pi \sin\theta/\lambda$, where 2θ is the scattering angle) and azimuthal angle φ as coordinates. The orientation of the MXene nanosheets in fibers was quantified by the (002) reflection from the WAXS patterns. The MXene nanosheets orientation and fibers alignment were quantified by converting the orientation distribution into a Hermans order parameter (f), defined as:

$$f = \langle \frac{3}{2} \cos^2 \varphi - \frac{1}{2} \rangle \quad (9)$$

where φ is the azimuthal distribution, and the orientation order parameter can be expanded as:

$$f = \int_0^\pi I(\varphi) (\frac{3}{2} \cos^2 \varphi - \frac{1}{2}) \sin(\varphi) d(\varphi) \quad (10)$$

[00153] The intensity is normalized according to

$$\int_0^\pi I(\varphi) (\sin(\varphi) d(\varphi)) = 1 \quad (11)$$

[00154] Estimation of the porosity of MXene-based fibers

[00155] The porosity (ρ) of the fabricated MXene-based fibers were calculated according to the structural parameters via equation (12):

$$\rho = \left(1 - \frac{\rho_{MF} d_{002}}{\rho_M d_M} \right) \times 100 \quad (12)$$

where ρ_{MF} is the density of MXene-based fibers, ρ_M is the Ti_3C_2 density of 5.2 g cm^{-3} , d_{002} is the d-spacing (002) of the MXene-based fibers, and d_M is the d-spacing of Ti_3C_2 crystals (1.02 nm).

CLAIMS

1. A method of forming a fiber, the method comprising:

forming a wet spun fiber from a plurality of MXene sheets using a wet spinning process; and

forming the fiber by heating the wet spun fiber using a thermal drawing process;

wherein the fiber is a cross-linked MXene-based fiber comprising a MXene core comprising the plurality of MXene sheets which are cross-linked; and

wherein the fiber further comprises a protective layer surrounding the MXene core.
2. The method according to claim 1,

wherein forming the wet spun fiber from the plurality of MXene sheets comprises:

forming an intermediate fiber by extruding a spinning dispersion comprising the plurality of MXene sheets into a coagulant bath; and

forming the wet spun fiber by passing the intermediate fiber through a cross-linking solution.
3. The method according to claim 2,

wherein the spinning dispersion further comprises glutaraldehyde (GA) such that the intermediate fiber formed is a cross-linked MXene- glutaraldehyde (GA) fiber.

4. The method according to claim 3,
wherein the cross-linking solution comprises polyvinyl alcohol (PVA) such
that the wet spun fiber formed is a cross-linked MXene- glutaraldehyde (GA) –
polyvinyl alcohol (PVA) fiber.
5. The method according to any one of claims 2 to 4, further comprising:
passing the intermediate fiber through a first washing bath before forming the
wet spun fiber; and
passing the wet spun fiber through a second washing bath before forming the
cross-linked MXene-based fiber.
6. The method according to claim 5,
wherein the first washing bath and the second washing bath comprise
deionized water.
7. The method according to any one of claims 1 to 6, further comprising:
wherein the protective layer is a polymer layer.
8. The method according to any one of claims 1 to 7,
wherein the plurality of MXene sheets are $\text{Ti}_3\text{C}_2\text{T}_x$ sheets;
wherein Ti is titanium;
wherein C is carbon;
wherein T is a functional group; and
wherein x is any number.

9. The method according to claim 8,
wherein the $\text{Ti}_3\text{C}_2\text{T}_x$ sheets are formed from Ti_3AlC_2 .
10. The method according to any one of claims 1 to 9,
wherein the plurality of MXene sheets are nanosheets.
11. The method according to any one of claims 1 to 10,
wherein the cross-linked MXene-based fiber has an electrical conductivity
selected from a range of between $8,000 \text{ S cm}^{-1}$ and $9,000 \text{ S cm}^{-1}$.
12. The method according to any one of claims 1 to 11,
wherein the thermal drawing process comprises feeding the wet spun fiber into
a tube passing through a furnace.
13. The method according to claim 12,
wherein the furnace has a hot zone having a temperature of 200°C .
14. The method according to any one of claims 1 to 13,
wherein a ratio of pull to feed speed of the thermal drawing process is of any
value selected from a range of between 1.2 and 1.5.
15. The method according to any one of claims 1 to 14,
wherein the cross-linked MXene-based fiber shows a porosity selected from a
range of between 5.5 % to 9 %.

16. The method according to any one of claims 1 to 15,
wherein the cross-linked MXene-based fiber shows an orientation order
selected from a range of between 0.8 to 0.9.
17. The method according to any one of claims 1 to 16,
wherein the cross-linked MXene-based fiber shows an absorption
electromagnetic interference (EMI) shielding efficiency (SE) selected from a
range of between 40 dB to 55 dB.
18. The method according to any one of claims 1 to 17,
wherein the cross-linked MXene-based fiber shows a total electromagnetic
interference (EMI) shielding efficiency (SE) selected from a range of between
50 dB to 65 dB.
19. The method according to any one of claims 1 to 18,
wherein the cross-linked MXene-based fiber comprising the protective layer is
configured to reach an equilibrium temperature selected from a range of
between 90 °C to 100 °C within 300 s upon applying a direct current (DC)
voltage of 6 V.
20. A fiber comprising:
a MXene core; and
a protective layer surrounding the MXene core;
wherein the fiber is a cross-linked MXene-based fiber such that the MXene core
comprises a plurality of MXene sheets which are cross-linked.

21. The fiber according to claim 20,
wherein the fiber is a cross-linked MXene- glutaraldehyde (GA) – polyvinyl alcohol (PVA) fiber.
22. The fiber according to claim 20 or claim 21,
wherein the protective layer is a polymer layer.
23. The fiber according to any one of claims 20 to 22,
wherein the cross-linked MXene-based fiber has an electrical conductivity selected from a range of between $8,000 \text{ S cm}^{-1}$ and $9,000 \text{ S cm}^{-1}$.
24. The fiber according to any one of claims 20 to 23,
wherein the cross-linked MXene-based fiber shows a porosity selected from a range of between 5.5 % to 9 %.
25. The fiber according to any one of claims 20 to 24,
wherein the cross-linked MXene-based fiber shows an orientation order selected from a range of between 0.8 to 0.9.
26. The fiber according to any one of claims 20 to 25,
wherein the cross-linked MXene-based fiber shows an absorption electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 40 dB to 55 dB.

27. The fiber according to any one of claims 20 to 26,
wherein the cross-linked MXene-based fiber shows a total electromagnetic interference (EMI) shielding efficiency (SE) selected from a range of between 50 dB to 65 dB.
28. The fiber according to any one of claims 20 to 27,
wherein the cross-linked MXene-based fiber comprising the protective layer is configured to reach an equilibrium temperature selected from a range of between 90 °C to 100 °C within 300 s upon applying a direct current (DC) voltage of 6 V.