nature portfolio

Peer Review File

Continuous MXene fibers with near-gigapascal tensile strength via radial confinement and axial stretching

Corresponding Author: Professor Qunfeng Cheng

This file contains all reviewer reports in order by version, followed by all author rebuttals in order by version.

Version 0:

Reviewer comments:

Reviewer #1

(Remarks to the Author)
This manuscript by Prof. Cheng and co-authors presents a continuous strategy for preparing MXene fibers through coaxial-wet-spinning-assisted radial confining integrated with roll-to-roll-assisted axial stretching. The wet-spun MXene fibers were firstly bridged with calcium ions and then radially confined to reduce the voids through transverse wrinkles by an in-situ bridged sodium alginate encapsulation layer through stretching. The resultant MXene fibers having strong interlayer interactions are highly aligned and densified. The record tensile strength of 958 MPa and electrical conductivity of 13,692 S cm-1 are achieved.
This manuscript is well-written, and the experimental data is well organized and robustly supports the conclusions. The general readers of Nature Communications will be interested in this new fabrication strategy and the outstanding properties of MXene fibers. Thus, this manuscript is strongly recommended for publication in Nature Communication after the following minor revision:
1. Much more details should be added in the figure captions for easily understanding the figures.
2. Much more discussions on fabrication and performance of reported MXene fibers should be added in the introduction, which is important for readers to understand the development of MXene fibers.
3. The format of references should be consistent with requirements of Nature Communications, please carefully double check main text.

Reviewer #2

(Remarks to the Author)
This work mainly reported a continuous fabrication strategy of high-performance MXene fibers by coaxial-wet-spinning-assisted radial confining integrated with roll-to-roll-assisted axial stretching. The reported data are solid, experimental methods is detailed, and the manuscript was well written and structured logically. I recommend this work for publication in Nature Communications. Some minor suggestions are listed below.

1. It is appreciated that the continuous fabrication process of CSM fibers has been recorded in the Supplementary Video 1. The manuscript can further improve its clarity by describing the diameter of spinneret channel, the extrusion speed (V0), and the collection speed (V1) in the caption of Supplementary Video 1.

2. It would be beneficial to specify the frequency range in the Supplementary Table 7 when comparing the EMI shielding performance of CSM textiles with that of the previous textiles woven using polymer, metal, carbon, and MXene composite fibers.

3. What is the density of CSM fibers? The CSM fibers are largely likely lighter than traditional metal wires, such as copper. Thus, comparing the mechanical properties and density of CSM fibers with those of copper wires is helpful to highlight the superiority of CSM fibers used as electrical wires.

4. The CSM fibers seems to be fabricated under near room temperature, which should be highlighted considering that many high-performance fibers, such as graphene fibers, are usually fabricated under high temperature (>2000 ºC).

5. What is the molecular weight or viscosity of SA powder? It should be described in the Methods section.

6. SR should be defined using full name upon first appearance in the Supplementary Information.

Reviewer #3

(Remarks to the Author)
This study provides a strategy for fabricating highly aligned and densely packed MXene-based fibers by combining coaxial wet spinning with sodium alginate bridging, followed by axial stretching. The reported mechanical strength, electrical conductivity, and EMI shielding performance of the MXene fibers are certainly impressive. However, there are several issues including discussion, experimental method, and format that must be solved before publication. After careful review, I suggest major revision subjected to the corrections for the publication in Nature communications.
1. The manuscript suggests that the MXene-infused fiber maintains electrical conductivity even under tensile deformation, implying a strong mechanical-electrical coupling. However, the reviewer is concerned that an embedded MXene network inside a CSA fiber could be disrupted under strain, potentially cracking the internal structure and breaking conductive pathways. Did the authors perform simultaneous tensile testing (e.g. on a UTM) with in situ resistance measurements to verify that conductivity is preserved during stretching? If not, on what basis do they claim that the fiber remains conductive under load? Demonstrating real-time electrical continuity during elongation is essential to validate the claimed electromechanical coupling.
2. The authors combine radial confinement with axial stretching to produce MXene fibers, describing this as a new synergistic strategy. Yet inserting a 2D nanomaterial into a tubular polymer structure and stretching it for alignment has been explored in prior studies[R1,R2]. Given that coaxial wet-spinning and post-drawing are established techniques for producing aligned composite fibers, what specifically is novel here? How does the proposed method fundamentally differ from or improve upon earlier MXene fiber fabrication approaches (e.g. core-sheath fibers or stretched composites)? The reviewer requests a clearer delineation of the unique innovations and advantages of this work, to convincingly establish its novelty beyond the existing literature.
3. Supplementary Fig S7, the TGA of the MXene shows virtually no weight loss, which is surprising. Even pristine $Ti_3C_2T_x$ MXene typically exhibits a mass loss due to absorbed water and decomposition of surface terminations upon heating. How do the authors explain the absence of any weight change in their MXene sample? Was the material pre-dried or treated in a way that eliminates adsorbed species, or might there be an issue with the TGA measurement (e.g. calibration or baseline drift)? This unexpected result raises concerns about sample preparation or instrument accuracy.
4. The introduction notes that introducing binders or crosslinkers can strengthen MXene fibers at the cost of electrical conductivity (since added agents disrupt inter-sheet contact). In this work, the MXene fibers are encapsulated with a calcium-crosslinked alginate shell intended to densify and reinforce the structure. How do the authors ensure that this encapsulation (and the presence of Ca^{2+} ions) does not impede electron transport along the fiber? The reported conductivity (13692 S cm^{-1}) is exceptionally high for a composite fiber, suggesting minimal electrical compromise. Did the authors compare conductivity between fibers with and without the alginate sheath, or measure whether the alginate layer adds any contact resistance at the MXene interface? A discussion of how the fiber maintains such high conductivity despite the polymer encapsulation would be valuable.
5. A central claim of the manuscript is that radial confinement through alginate/Ca^{2+} encapsulation densifies the MXene structure by eliminating transverse wrinkles and voids. While SAXS and TEM images are presented to support this, the evidence remains largely qualitative. Have the authors measured the bulk density (mass per unit volume) of the fibers with and without radial confinement? Quantitative density analysis is essential to validate the claimed densification. Without such data, it is difficult to objectively assess how much structural compaction was achieved.
6. The manuscript reports a tensile strength of 958 MPa, which is among the highest values for MXene-based fibers. However, from the shape of the S-S curve, it is unclear where and how the internal MXene network fails during elongation. To ensure practical applicability, it is important to determine whether the electrical pathways persist up to or beyond the mechanical failure point. Could the authors clarify at what strain level the electrical conductivity is lost, and whether the fracture originates from disruption of the MXene stack or from the surrounding matrix?
7. Although the manuscript provides cycling and sonication experiments in the SI, the reviewer finds the validation incomplete. Specifically, after sonication, the study does not clearly show whether the electrical conductivity is retained or how the internal structure is affected (e.g., cracks, delamination, or collapse). Can the authors provide comparative post-sonication conductivity data, or high-resolution structural images to assess damage? Given that sonication imposes significant mechanical agitation, the fiber's performance post-treatment is critical to prove its durability.

[R1] Zhou, Tianzhu, et al. "Ultra-compact MXene fibers by continuous and controllable synergy of interfacial interactions and thermal drawing-induced stresses." Nature Communications 13.1 (2022): 4564.
[R2] Zhou, Tianzhu, et al. "Interlocking-governed ultra-strong and highly conductive MXene fibers through fluidics-assisted thermal drawing." Advanced Materials 35.51 (2023): 2305807.

Version 1:

Reviewer comments:

Reviewer #1

(Remarks to the Author)

This version is fine to be accepted.

Reviewer #2

(Remarks to the Author)
As my comments were considered during their revision process, the revised version can be accepted,

Reviewer #4

(Remarks to the Author)
I am reviewing the manuscript “Continuous ultrastrong MXene fibers via the synergy of radial confining and axial stretching” in response to the editor’s request. The authors have meticulously and clearly addressed all the concerns raised by the Reviewer 3 in the previous round of peer review. The authors have measured the real-time electrical conductance during tensile testing, TGA curve, bulk densities, the in-situ SEM of stretching process, and the electrical conductivity and cross-sectional SEM after sonicating. Additionally, the revised manuscript has provided a convincing discussion on high conductivity and a clear description of its unique innovations and advantages, which are very impressive on me. The supplemented data are solid and further verify the conclusions, as follows:
1. The real-time electrical conductance results clearly demonstrate that CSM fibers preserve high conductivity and the inner MXene layer does not break prematurely during stretching.
2. The low energy-consuming fabrication process under near room temperature, strong core-shell interfacial interactions, and synergistic densification effect induced by axial stretching and radial confining are presented as the novelties and advantages of this work that can fully differentiate from the prior studies.
3. The re-tested TGA curve is fine and consistent with the previous reports.
4. The authors have compared the conductivity of MXene fibers without SA sheath and CM fibers with SA sheath, firmly verifying that the SA encapsulation promotes the electron transport along the fiber. A reasonable explanation strongly supported by structural characterization has also been provided that the radial confining induced by bridged SA encapsulation effectively improves the alignment of MXene nanosheets and removes the voids resulting from transverse wrinkles.
5. The bulk density of the MXene layer in MXene, SM, CM, and CSM fibers has been measured. The result is consistent with SAXS and TEM characterizations, further demonstrating the densification induced by radial confining and axial stretching.
6. The stretching process of CSM fibers has been observed by in-situ SEM, clearly demonstrating that the bridged SA encapsulation layer does not break prematurely during elongation. Together with real-time electrical conductance results, it is convincing to claim that the inner MXene and bridged SA encapsulation layers are broken almost simultaneously at the mechanical failure point of CSM fibers, partly because of the strong core-shell interfacial interactions.
7. After sonicating, CSM fibers are still compact and have a high conductivity retention, well demonstrating their excellent durability.
In short, the revised manuscript has good enough data for verifying the conclusions, and this work will be an good advance in the field of MXene, 2D materials and fibers. I recommend that this work can be published in Nature Communications.

RESPONSES TO REVIEWERS' COMMENTS FOR MANUSCRIPT NCOMMS-25-45809

We would like to express our appreciation to the three reviewers for their very helpful comments and constructive suggestions. We realize that all of you spent a considerable amount of time carefully reading the manuscript and formulating a strategy of action for us to make this paper better. We are very grateful for this.

We have attempted to address all the issues raised in the review and to revise the paper exactly along the lines suggested. Below we provide the reviewers' comments in **black** and our responses in **blue**.

Reviewer 1:

This manuscript by Prof. Cheng and co-authors presents a continuous strategy for preparing MXene fibers through coaxial-wet-spinning-assisted radial confining integrated with roll-to-roll assisted axial stretching. The wet-spun MXene fibers were firstly bridged with calcium ions and then radially confined to reduce the voids through transverse wrinkles by an in-situ bridged sodium alginate encapsulation layer through stretching. The resultant MXene fibers having strong interlayer interactions are highly aligned and densified. The record tensile strength of 958 MPa and electrical conductivity of 13,692 S cm^{-1} are achieved.
This manuscript is well-written, and the experimental data is well organized and robustly supports the conclusions. The general readers of Nature Communications will be interested in this new fabrication strategy and the outstanding properties of MXene fibers. Thus, this manuscript is strongly recommended for publication in Nature Communication after the following minor revision:

Reply: We appreciate the reviewer's positive comments. We have tried to revise the manuscript exactly along the following suggestions.

1. Much more details should be added in the figure captions for easily understanding the figures.

Reply: We appreciate the reviewer's insightful suggestions. The details have been added in the captions of Figures 1~4 of the revised manuscript with highlighted in yellow.

2. Much more discussions on fabrication and performance of reported MXene fibers should be added in the introduction, which is important for readers to understand the development of MXene fibers.

Reply: We appreciate the reviewer's insightful suggestions. The discussions on the fabrication and performance of reported MXene fibers have been added in the introduction section of the revised manuscript with highlighted in yellow.

3. The format of references should be consistent with requirements of Nature Communications, please carefully double check main text.

Reply: We appreciate the reviewer's kind reminding. The format of references has been checked and corrected in the revised manuscript according to the requirements of Nature Communications.

Reviewer 2:

This work mainly reported a continuous fabrication strategy of high-performance MXene fibers by coaxial-wet-spinning-assisted radial confining integrated with roll-to-roll-assisted axial stretching. The reported data are solid, experimental methods is detailed, and the manuscript was well written and structured logically. I recommend this work for publication in Nature Communications. Some minor suggestions are listed below.

Reply: We appreciate your encouraging comments. We have tried to revise the manuscript exactly along the following suggestions.

1. It is appreciated that the continuous fabrication process of CSM fibers has been recorded in the Supplementary Video 1. The manuscript can further improve its clarity by describing the diameter of spinneret channel, the extrusion speed (V_0), and the collection speed (V_1) in the caption of Supplementary Video 1.

Reply: We appreciate the reviewer's insightful suggestions. The following description has been added in the caption of Supplementary Movie 1 of the revised manuscript with highlighted in yellow: The coaxial spinneret consists of an inner 28-gauge needle having an inner diameter of 0.19 mm and an outer diameter of 0.35 mm and an outer 21-gauge needle having an inner diameter of 0.5 mm and an outer diameter of 0.8 mm. The extrusion (V_0) and collection (V_1) speeds are 7.2 and 13.0 cm min^{-1}, respectively.

2. It would be beneficial to specify the frequency range in the Supplementary Table 7 when comparing the EMI shielding performance of CSM textiles with that of the previous textiles woven using polymer, metal, carbon, and MXene composite fibers.

Reply: We appreciate the reviewer's insightful suggestions. The frequency range has been specified in the Supplementary Table 7 of the revised manuscript with highlighted in yellow.

3. What is the density of CSM fibers? The CSM fibers are largely likely lighter than traditional metal wires, such as copper. Thus, comparing the mechanical properties and density of CSM fibers with those of copper wires is helpful to highlight the superiority of CSM fibers used as electrical wires.

Reply: We appreciate the reviewer's insightful questions and suggestions. The density of CSM fibers is 2.07 ± 0.03 g cm^{-3}, which is lower than for copper wires (9.7 g cm^{-3}, *Nano-Micro Lett.* 2022, 14, 111). Additionally, the tensile strength and toughness of CSM fibers (958 ± 29 MPa and 66.3 ± 3.7 MJ m^{-3}, *Nano-Micro Lett.* 2022, 14, 111) are much higher than for copper wires (256 MPa and 51.8 MJ m^{-3}), respectively. The following comparison has been made in the revised manuscript with highlighted in yellow: Moreover, the density of CSM fibers (2.07 ± 0.03 g cm^{-3}) is much lower than for copper wires (9.7 g cm^{-3}), while their tensile strength and toughness are much higher than for copper wires (256 MPa and 51.8 MJ m^{-3})[42], suggesting promising application in flexible electrical wires.

4. The CSM fibers seems to be fabricated under near room temperature, which should be highlighted considering that many high-performance fibers, such as graphene fibers, are usually fabricated under high temperature (>2000 ºC).

Reply: We appreciate the reviewer’s insightful suggestions. The “near-room-temperature fabrication process” has been emphasized in the sections of Abstract, Introduction, Fabrication of CSM fibers, and Discussion of the revised manuscript with highlighted in yellow.

5. What is the molecular weight or viscosity of SA powder? It should be described in the Methods section.

Reply: We appreciate the reviewer’s kind reminding. The viscosity of SA powder is 300 mPa·s, which has been added in the Methods section of the revised manuscript with highlighted in yellow.

6. SR should be defined using full name upon first appearance in the Supplementary Information.

Reply: We appreciate the reviewer’s kind reminding. Stretching ratio has been defined as SR upon first appearance in Supplementary Figure 6 of the revised Supplementary Information with highlighted in yellow.

Reviewer 3:

This study provides a strategy for fabricating highly aligned and densely packed MXene-based fibers by combining coaxial wet spinning with sodium alginate bridging, followed by axial stretching. The reported mechanical strength, electrical conductivity, and EMI shielding performance of the MXene fibers are certainly impressive. However, there are several issues including discussion, experimental method, and format that must be solved before publication. After careful review, I suggest major revision subjected to the corrections for the publication in Nature communications.

Reply: We appreciate the reviewer's comments. We have performed additional experiments and tried to clarify the following questions.

1. The manuscript suggests that the MXene-infused fiber maintains electrical conductivity even under tensile deformation, implying a strong mechanical-electrical coupling. However, the reviewer is concerned that an embedded MXene network inside a CSA fiber could be disrupted under strain, potentially cracking the internal structure and breaking conductive pathways. Did the authors perform simultaneous tensile testing (e.g. on a UTM) with in situ resistance measurements to verify that conductivity is preserved during stretching? If not, on what basis do they claim that the fiber remains conductive under load? Demonstrating real-time electrical continuity during elongation is essential to validate the claimed electromechanical coupling.

Reply: We appreciate the reviewer's insightful comments and suggestions. The real-time electrical conductance of CSM fibers was measured during tensile testing, as shown in Figure R1. Upon tensile loading, the electrical conductance of CSM fibers is slightly decreased because of the slippage of MXene nanosheets and can maintain more than 90.4% of its initial value. At the moment of fracture of CSM fibers, their electrical conductance retention is sharply decreased from 90.4% to 0.00012%, indicating that the inner MXene layer does not break prematurely during stretching. Thus, CSM fibers remain highly conductive during stretching. The above result and discussion have been added in the revised manuscript with highlighted in yellow. Figure R1 is Supplementary Figure 36 in the revised manuscript. Additionally, CSM fibers remain highly conductive during cyclic bending (Supplementary Figure 28) or knotting (Supplementary Figures 34 and 35b).

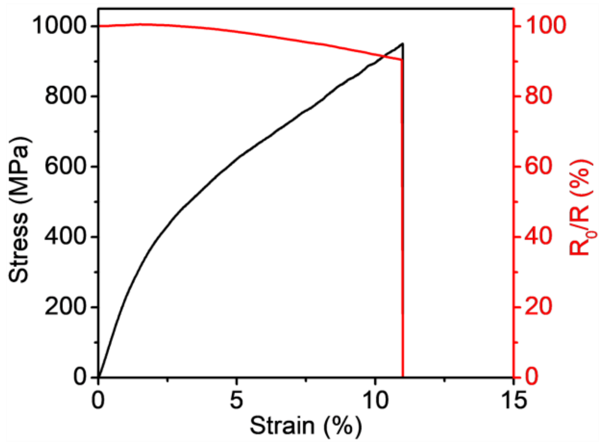

Figure R1. Tensile stress-strain curve of CSM fibers and their electrical conductance retention percentage during tensile testing.

2. The authors combine radial confinement with axial stretching to produce MXene fibers, describing this as a new synergistic strategy. Yet inserting a 2D nanomaterial into a tubular polymer structure and stretching it for alignment has been explored in prior studies [R1,R2]. Given that coaxial wet-spinning and post-drawing are established techniques for producing aligned composite fibers, what specifically is novel here? How does the proposed method fundamentally differ from or improve upon earlier MXene fiber fabrication approaches (e.g. core-sheath fibers or stretched composites)? The reviewer requests a clearer delineation of the unique innovations and advantages of this work, to convincingly establish its novelty beyond the existing literature.

Reply: We appreciate the reviewer's insightful comments. The mentioned previous studies (R1: *Nat. Commun.* 2022, 13, 4564; R2: *Adv. Mater.* 2023, 35, 2305807) mainly fabricated core-sheath and aligned MXene fibers by thermal drawing a molten polymer tube under a temperature typically higher than 200 ºC. Very recently, the coaxial wet-spinning and post-drawing have also been developed to fabricate core-sheath and aligned MXene fibers under near room temperature (*Nano-Micro Lett.* 2022, 14, 111; *Adv. Funct. Mater.* 2025, 25, 2419923; *Small* 2025, 21, 2411735; *Adv. Mater.* 2025, 35, 2501485). Improving the properties of MXene fibers by designing the core-sheath and stretch-induced aligned structure seems like the similarity between these previous studies and our current work. However, the interfacial interactions between core and shell layers are usually weak and even neglected in the previous studies, largely decreasing the properties of MXene fibers. Additionally, the thermal drawing process is complex and energy-consuming. What differentiates the current work from these previous studies mainly includes the following aspects:

(1) The fabrication process of CSM fibers in our current work is under near room temperature, which is lower energy-consuming compared with thermal drawing process.
(2) The inner MXene layer is strongly crosslinked with the outer bridged SA layer by hydrogen and ionic bonding (please see the Structural characterization of CSM fibers

section of the revised manuscript) in our current work, which is largely differ from the weak core-shell interfacial interactions of the MXene fibers fabricated by thermal drawing or coaxial wet-spinning and post-drawing in the previous studies. The strong core-shell interfacial interactions is responsible for the excellent mechanical properties of CSM fibers and their good structural integrity and high electrical conductivity retention during deformation (please see the detailed response to your comments 1 and 6).

(3) Wet-spun MXene fibers usually show loose assembly of MXene nanosheets in transverse direction because of the nanosheet wrinkling induced by the capillary contraction during drying. We found in the current work that axial stretching (can induce the shrinkage of bridged SA layer) and radial confining can synergistically inhibit the capillary contraction of MXene nanosheets and reduce the voids resulting from transverse wrinkles (please see the Structural characterization of CSM fibers section of the revised manuscript), which has not yet been reported in the previous studies.

We strongly believe that the above novelties and advantages can fully differentiate our current work from the mentioned previous studies (R1 and R2) and other reported works. The following description has been added in the revised manuscript with highlighted in yellow: (1) under near room temperature; (2) Thus, the inner MXene layer in CSM fibers is strongly crosslinked with the outer bridged SA layer by hydrogen and ionic bonding; (3) synergistically reducing the voids resulting from transverse wrinkles.

3. Supplementary Fig S7, the TGA of the MXene shows virtually no weight loss, which is surprising. Even pristine $Ti_3C_2T_x$ MXene typically exhibits a mass loss due to absorbed water and decomposition of surface terminations upon heating. How do the authors explain the absence of any weight change in their MXene sample? Was the material pre-dried or treated in a way that eliminates adsorbed species, or might there be an issue with the TGA measurement (e.g. calibration or baseline drift)? This unexpected result raises concerns about sample preparation or instrument accuracy.

Reply: We appreciate the reviewer's kind reminding. We have re-tested the TGA curve of MXene powder under a nitrogen atmosphere with a temperature rising from room temperature to 550 ºC at a rate of 10 ºC min^{-1} (Figure R2). The mass loss of MXene powder is about 3.16% because of the removal of absorbed water and decomposition of surface terminations upon heating from room temperature to 550 ºC. Figure R2 is Supplementary Figure 7 in the revised manuscript.

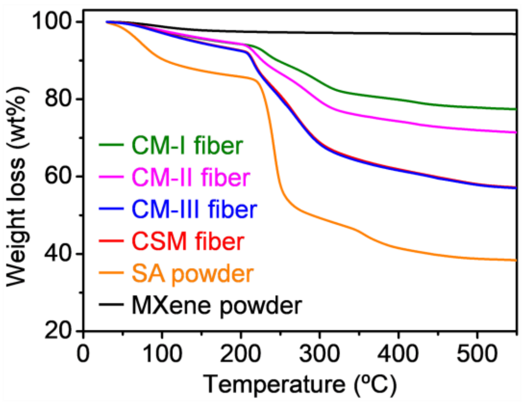

Figure R2. TGA curves of MXene powder, CM-I to CM-III fibers, CSM fibers, and SA powder.

4. The introduction notes that introducing binders or crosslinkers can strengthen MXene fibers at the cost of electrical conductivity (since added agents disrupt inter-sheet contact). In this work, the MXene fibers are encapsulated with a calcium-crosslinked alginate shell intended to densify and reinforce the structure. How do the authors ensure that this encapsulation (and the presence of Ca^{2+} ions) does not impede electron transport along the fiber? The reported conductivity (13692 S cm^{-1}) is exceptionally high for a composite fiber, suggesting minimal electrical compromise. Did the authors compare conductivity between fibers with and without the alginate sheath, or measure whether the alginate layer adds any contact resistance at the MXene interface? A discussion of how the fiber maintains such high conductivity despite the polymer encapsulation would be valuable.

Reply: We appreciate the reviewer's insightful comments and suggestions. Compared with MXene fibers (9,325 ± 107 S cm^{-1}) without SA sheath, CM fibers (10,818 ± 112 S cm^{-1}) with SA sheath have higher electrical conductivity (Supplementary Table 6), demonstrating that the SA encapsulation promotes the electron transport along the fiber. This is because the radial confining induced by SA encapsulation effectively improves the alignment of MXene nanosheets (Supplementary Table 3) and removes the voids resulting from transverse wrinkles (Supplementary Table 2). Note that both MXene and CM fibers fabricated using calcium chloride as coagulant contain Ca^{2+} ions. The following discussion has been added in the revised manuscript with highlighted in yellow: The electrical conductivity of CM fibers is higher than for MXene fibers, demonstrating that the bridged SA encapsulation promotes the electron transport along the fiber. This is because the radial confining induced by bridged SA encapsulation effectively improves the alignment of MXene nanosheets and removes the voids resulting from transverse wrinkles.

5. A central claim of the manuscript is that radial confinement through alginate/Ca2+ encapsulation densifies the MXene structure by eliminating transverse wrinkles and voids. While SAXS and TEM images are presented to support this, the evidence remains largely qualitative. Have the authors measured the bulk density (mass per unit volume) of the fibers with and without radial confinement? Quantitative density analysis is essential to

validate the claimed densification. Without such data, it is difficult to objectively assess how much structural compaction was achieved.

Reply: We appreciate the reviewer's insightful comments and suggestions. The bulk density of the MXene layer in MXene, SM, CM, and CSM fibers has been measured, as shown in Figure R3. The magnitude of the density for MXene layer in fibers increases as follows: MXene < SM < CM < CSM, which is consistent with SAXS and TEM characterizations. This result further demonstrates the densification induced by radial confining and axial stretching. The density analysis has been added in the revised manuscript with highlighted in yellow. Figure R3 is Supplementary Figure 14 in the revised manuscript.

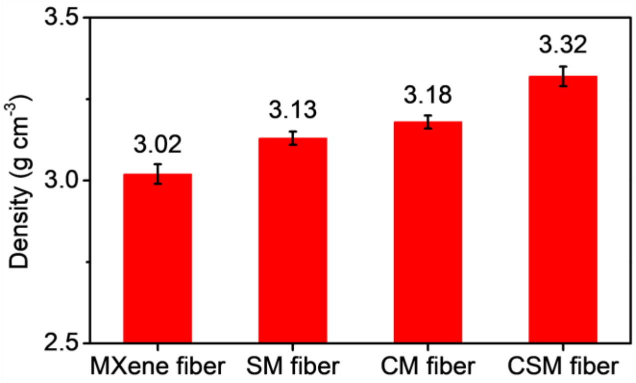

Figure R3. Density of the MXene layer in MXene, SM, CM, and CSM fibers.

6. The manuscript reports a tensile strength of 958 MPa, which is among the highest values for MXene-based fibers. However, from the shape of the S-S curve, it is unclear where and how the internal MXene network fails during elongation. To ensure practical applicability, it is important to determine whether the electrical pathways persist up to or beyond the mechanical failure point. Could the authors clarify at what strain level the electrical conductivity is lost, and whether the fracture originates from disruption of the MXene stack or from the surrounding matrix?

Reply: We appreciate the reviewer's insightful questions. The real-time conductance measurements during tensile testing show that the inner MXene layer does not break prematurely during elongation and the electrical conductivity is lost at the mechanical failure point (please see the detailed response to your comment 1). Additionally, the stretching process of CSM fibers was observed by in-situ SEM (Supplementary Movie 7), demonstrating that the bridged SA encapsulation layer does not break prematurely during elongation. The fracture surface also shows that the inner MXene layer is closely boned with the outer SA encapsulation layer (Supplementary Figure 12h). Thus, the inner MXene and bridged SA encapsulation layers are broken almost simultaneously at the mechanical failure point of CSM fibers, partly because of the strong core-shell interfacial interactions. The following discussion has been added in the revised manuscript with highlighted in yellow: CSM fibers also remain highly conductive during stretching (Supplementary Figure 36) and their inner MXene and bridged SA encapsulation layers are broken almost simultaneously at the mechanical failure point (Supplementary Movie 7), partly because of the strong interfacial interactions.

7. Although the manuscript provides cycling and sonication experiments in the SI, the reviewer finds the validation incomplete. Specifically, after sonication, the study does not clearly show whether the electrical conductivity is retained or how the internal structure is affected (e.g., cracks, delamination, or collapse). Can the authors provide comparative post-sonication conductivity data, or high-resolution structural images to assess damage? Given that sonication imposes significant mechanical agitation, the fiber's performance post-treatment is critical to prove its durability.

Reply: We appreciate the reviewer's insightful suggestions. The electrical conductivity of CSM fibers after sonicating was measured (Figure R4) and their cross-sectional structure was also characterized (Figure R5). After sonicating for 60 minutes, the retention percentage of the electrical conductivity for CSM fibers is 98.1 ± 0.4%. Additionally, the internal structure of CSM fibers after sonicating is similar to that before sonicating (Supplementary Figure 11b), demonstrating their excellent durability. The above results and discussion have been added in the revised manuscript with highlighted in yellow. Figures R4 and R5 are Supplementary Figures 30 and 31 in the revised manuscript, respectively.

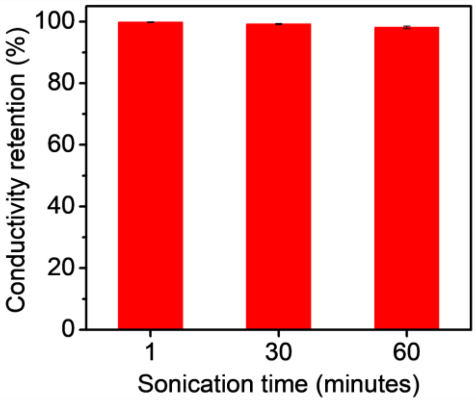

Figure R4. Electrical conductivity retention percentage of CSM fibers after sonicating for 1, 30, and 60 minutes.

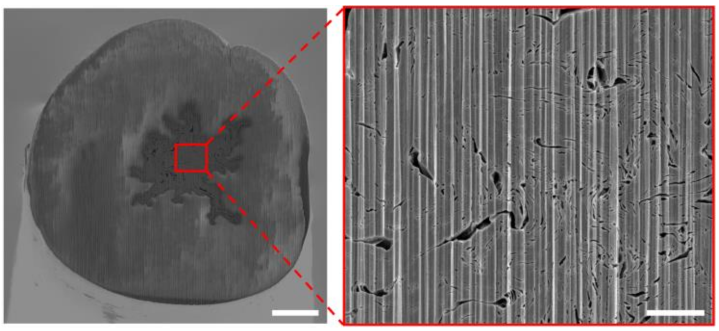

Figure R5. SEM images of the cross-section cut by focused ion beam for CSM fibers after sonicating for 60 minutes. Scale bars, 10 μm in low-resolution image and 1 μm in high-resolution image.

[R1] Zhou, Tianzhu, et al. "Ultra-compact MXene fibers by continuous and controllable synergy of interfacial interactions and thermal drawing-induced stresses." Nature Communications 13.1(2022): 4564.
[R2] Zhou, Tianzhu, et al. "Interlocking - governed ultra - strong and highly conductive MXene fibers through fluidics - assisted thermal drawing." Advanced Materials 35.51 (2023): 2305807.

RESPONSES TO REVIEWERS' COMMENTS FOR MANUSCRIPT NCOMMS-25-45809A

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This version is fine to be accepted.

Reply: We appreciate the reviewer's positive comments.

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As my comments were considered during their revision process, the revised version can be accepted.

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Reviewer 4:

I am reviewing the manuscript "Continuous ultrastrong MXene fibers via the synergy of radial confining and axial stretching" in response to the editor's request. The authors have meticulously and clearly addressed all the concerns raised by the Reviewer 3 in the previous round of peer review. The authors have measured the real-time electrical conductance during tensile testing, TGA curve, bulk densities, the in-situ SEM of stretching process, and the electrical conductivity and cross-sectional SEM after sonicating. Additionally, the revised manuscript has provided a convincing discussion on high conductivity and a clear description of its unique innovations and advantages, which are very impressive on me. The supplemented data are solid and further verify the conclusions, as follows:

1. The real-time electrical conductance results clearly demonstrate that CSM fibers preserve high conductivity and the inner MXene layer does not break prematurely during stretching.
2. The low energy-consuming fabrication process under near room temperature, strong core-shell interfacial interactions, and synergistic densification effect induced by axial stretching and radial confining are presented as the novelties and advantages of this work that can fully differentiate from the prior studies.
3. The re-tested TGA curve is fine and consistent with the previous reports.
4. The authors have compared the conductivity of MXene fibers without SA sheath and CM fibers with SA sheath, firmly verifying that the SA encapsulation promotes the electron

transport along the fiber. A reasonable explanation strongly supported by structural characterization has also been provided that the radial confining induced by bridged SA encapsulation effectively improves the alignment of MXene nanosheets and removes the voids resulting from transverse wrinkles.

5. The bulk density of the MXene layer in MXene, SM, CM, and CSM fibers has been measured. The result is consistent with SAXS and TEM characterizations, further demonstrating the densification induced by radial confining and axial stretching.

6. The stretching process of CSM fibers has been observed by in-situ SEM, clearly demonstrating that the bridged SA encapsulation layer does not break prematurely during elongation. Together with real-time electrical conductance results, it is convincing to claim that the inner MXene and bridged SA encapsulation layers are broken almost simultaneously at the mechanical failure point of CSM fibers, partly because of the strong core-shell interfacial interactions.

7. After sonicating, CSM fibers are still compact and have a high conductivity retention, well demonstrating their excellent durability.

In short, the revised manuscript has good enough data for verifying the conclusions, and this work will be an good advance in the field of MXene, 2D materials and fibers. I recommend that this work can be published in Nature Communications.

Reply: We appreciate the reviewer's encouraging comments.