nature portfolio

Peer Review File

Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys

Corresponding Author: Dr Abigail Ackerman

This file contains all editorial decision letters in order by version, followed by all author rebuttals in order by version.

Version 0:

Decision Letter:

** Please ensure you delete the link to your author homepage in this email if you wish to forward it to your coauthors **

Dear Dr Ackerman,

Thank you again for submitting your manuscript "Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys" to Communications Materials. We have now received reports from 2 reviewers and, based on their comments, we have decided to invite a revision of your work. You will find the reviewers' reports below. While they find your work of interest, they have raised important points which must be addressed in a revised manuscript.

To allow us to move forward with your work, we also ask that you edit your manuscript according to the attached table. **Please read this document carefully as we will be unable to further assess your revised paper until these important points are addressed.**

Please outline all revisions made in the right-hand column and return the completed table with your updated manuscript files as a Related Manuscript file.

When resubmitting, please also include:

- A point-by-point response to the reviewers' comments. If you are unable to address specific reviewer requests or find any points invalid, please explain
- A clean version of your revised manuscript with no mark-ups
- A marked-up version of your paper with all changes highlighted in a different colour

Please use the link below to submit your revised files:
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** This url links to your confidential home page and associated information about manuscripts you may have submitted or that your are reviewing for us. If you wish to forward this email to co-authors, please delete the link to your homepage first **

We hope to receive your revised paper within six weeks, but we understand that the revisions may take longer. Please let us know if you find that the revision process will take substantially more time.

We are committed to providing a fair and constructive peer-review process. Please do not hesitate to contact me if you have any questions or would like to discuss these revisions further. We look forward to seeing the revised manuscript and thank you for the opportunity to review your work.

Best regards,

John Plummer, PhD
Chief Editor
orcid.org/0000-0003-4824-8497
Communications Materials

Reviewers' comments:

Reviewer #1 (Remarks to the Author):

The manuscript describes an interesting study aiming at refining the size of secondary alpha precipitates in a prototype titanium alloy with a multi-modal alpha distribution. State-of-the-art characterization techniques are employed to provide convincing improvements in the fatigue performance of the investigated alloy, as compared to the as-received state. The manuscript is short, as recommended for this journal, well-written, and easy to read. Overall, I think that it can be a good contribution to the journal, as this topic, and the reported findings, are of interest to the titanium community.
Upon reading the article, I have several comments which should be addressed for publication. The authors completely eluded prior studies on so-called bi-lamellar microstructures, or even tri-modal microstructures. In particular, extensive data have been reported by Lütjering, and most trends reported in the present article are in line with his findings: lower crack growth rate, higher tensile strength, higher fatigue strength, lower ductility. Prior knowledge should be more clearly reported. Moreover, it will give more strength to the results from this manuscript. However, I am puzzled by the lack of deformation introduced in this prior work. Have the authors of the present manuscript made a trial without any rolling step to confirm the role of dislocations? Is it possible that alpha precipitation proceeds preferentially on omega precipitates rather than on dislocations? From the high temperature heat treatment, residual beta may be sufficiently enriched in beta stabilizers for omega to form during heating. From the TEM images, it also appears that alpha phase precipitates away from the dislocations contained in the slip band, which I do not understand unless other nucleation sites exist. I think that it is an essential point to discuss and clarify in the article, as the role of omega phase is now well established in basketweave alpha precipitation.
Other minor comments:
- Some sentences are very long (e.g., there are only 3 sentences in the abstract). Shorter sentences would make the paper easier to read.
- Time constraints are probably not a relevant reason for lacking experimental data in an article of a high impact journal.
- There is a typo L52P4.
- Key information about experiments is missing. For example, the composition of the investigated alloy is not given, as well as details of prior thermo-mechanical processing. The strain rate for tensile tests is missing as well. Of critical importance is the heating rate of the ageing treatment, which may, or may not, promote the formation of omega phase. This is well-known from previous studies on metastable beta alloys. In particular, omega precipitation may occur in situ in the TEM experiment. Do the authors have information to provide? Different heating rates may completely change precipitation routes.
- I was surprised that the cold rolling route disappeared along the way. I guess that cold rolling high strength material like Ti6246 is a challenge that prevented tensile and fatigue specimens to be prepared. I may have missed this information.
- In situ TEM heating conditions are very different from conditions applied in conventional experiments, as highlighted by the authors. Can the authors elaborate on this: is 850°C measured away from the foil?
- I do not see the drop in strength near the yield point in figure 5.
- With no lifetime lower than 105 cycles, there is no LCF data reported in this article.
- There is a typo L90P6: K1c is not a fatigue crack growth threshold, thus it has not been measured previously.
- I do not understand why the paragraph L7P7 exists. It contains very vague and general statements, not really connected to findings from this work. In particular, I would not say that dislocations are evenly distributed in these alloys.

Reviewer #2 (Remarks to the Author):

The authors investigated changes in the thermomechanical processing of Ti-6246 alloy by employing cold or warm rolling to introduce dislocations into the β matrix using high-resolution characterization techniques, mechanical testing and 4D-STEM. The experimental findings reveal that cold and warm working can induce dislocations, leading to the precipitation of fine scale secondary α. In addition, the authors proposed that the formation of fine secondary α phase contributes significantly to the alloy's enhanced mechanical properties. However, this paper has weaknesses in experimental specifics, results and data analysis presented in the manuscript. The authors should address these issues in the following specific comments.
Considering the above, I cannot recommend this work for publication in Communications Materials in its present form.
1. The authors attribute the improvement in strength and hardness of the alloy primarily to the refinement of the secondary α phase. However, after rolling deformation, lattice distortion and dislocation density increase significantly, leading to a strong work-hardening effect (i.e., enhanced strength and hardness). The authors should clarify whether the observed mechanical enhancement is dominated by the secondary α refinement or the work-hardening effect.
2. Regarding the 4D-STEM strain maps in Figure 4, It is not clear why the authors chose 1050 °C as the in-situ heating temperature? This temperature exceeds the β-transus temperature of Ti-6246 alloy. Under this condition, it would be expected that the dissolution of the α phase has occurred, accompanied by transformation into the β phase.
3. On page 4, line 65–67, the authors mention that "The warm rolled and aged material shows a slight drop in strength towards its yield point." However, this feature is not clearly shown in Figure 5. The authors should provide clarification.
4. The authors should clarify how the 0.2% yield strength values were determined for the two stress-strain curves in Figure 5.

Based on the stress-strain curves, the two curves exhibit nearly overlapping behavior (virtually linear) within the 0–1000 MPa range, indicating that their yield strengths should both exceed 1000 MPa.
5. The authors propose that the microstructure resulting from the edited processing route contains all 12 secondary α variants without apparent variant selection. However, titanium alloys typically develop pronounced crystallographic textures following rolling deformation. The authors should clarify whether there are differences in the quantity or proportion of these 12 variants. Furthermore, the authors should discuss whether the resulting textures influence the mechanical properties of the alloy.
6. Please add label (a) to the upper-left corner of the leftmost image in Figure 3. Scale bars should be provided into both Figures (a) and (b). Additionally, the caption for Figure 3 contains an incorrect term: “Bright Field Transmission Electron Diffraction,” which requires correction.
7. On page 6, line 42, “112” should be corrected to “{112}”.
8. The authors should check the language and grammar of their manuscript. Examples are as follows:
1) Page 4, line 52: “pahses”.
2) Page 6, line 23 and 24: “850C” and “250C”.
3) Page 6, line 27: “foilan”.
4) Page 6, line 42: “planesindicating”.
5) Page 6, line 63: “Qui”.
6) Page 7, line 12: “defectssimilar”.
9. There should be a space between the numerical value and the unit. For instance, “850°C” should be corrected to “850 °C” (with a space between the numerical value and the degree Celsius symbol).

Version 1:

Decision Letter:

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Dear Dr Ackerman,

Thank you again for submitting your revised manuscript "Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys" to Communications Materials. It has been seen again by the two original reviewers, who have raised some further points that require revision. You will find the reviewers’ reports below. We therefore ask that you address these points in a revised manuscript.

To allow us to move forward with your work, we also ask that you edit your manuscript according to the attached table. **Please read this document carefully as we will be unable to further assess your revised paper until these important points are addressed.**

Please outline all revisions made in the right-hand column and return the completed table with your updated manuscript files as a Related Manuscript file.

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- A clean version of your revised manuscript with no mark-ups
- A marked-up version of your paper with all changes highlighted in a different colour

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** This url links to your confidential home page and associated information about manuscripts you may have submitted or that your are reviewing for us. If you wish to forward this email to co-authors, please delete the link to your homepage first **

We hope to receive your revised paper within six weeks, but we understand that the revisions may take longer. Please let us know if you find that the revision process will take substantially more time.

We are committed to providing a fair and constructive peer-review process. Please do not hesitate to contact me if you have any questions or would like to discuss these revisions further. We look forward to seeing the revised manuscript and thank you for the opportunity to review your work.

Best regards,

John Plummer, PhD
Chief Editor
orcid.org/0000-0003-4824-8497
Communications Materials

Reviewers' comments:
Reviewer #1 (Remarks to the Author):

The authors provided a revised version of the manuscript to address the comments of the reviewers. Overall, changes and responses to my comments are satisfactory. I am somewhat disappointed by the lack of acknowledgement of a possible role of omega, although no conclusive evidence of its absence is provided in the rebuttal. Given the differences in heat treatments between the original and the edited routes (i.e., annealing temperatures and cooling rates), a possible role cannot be ruled out so easily in my opinion, but OK.
Also the measured composition of the investigated alloy remains missing. The rotation is given as a percentage in figure 4: is it a fraction of radians? (I missed it in the first round of reviews: sorry for that). A number of typos remain (e.g., page 4 line 65, page 6 line 28, page 8 line 8), they might be corrected in the proofs.

Reviewer #2 (Remarks to the Author):

I appreciate the authors for their responses to the reviewer comments and for their efforts to revise the manuscript. The authors have carefully addressed the majority of the issues raised in the review comments and made corresponding revisions, which have improved the overall quality of the manuscript. However, several key issues still remain inadequately resolved, which are critical to ensuring the rigor of the research conclusions and the reliability of the data presented.

1. Regarding issue 1: The inset table in Figure 5 indicates that the hardness value of the "No αs" sample is lower than that of the "As-received" sample, suggesting that the presence of αs has an impact on the material's hardness. However, for the "Cold/Warm Rolled & Aged" samples, although their hardness has indeed increased compared to both the "No αs" and "As-received" samples, deformation and the presence of fine αs are simultaneously introduced. These factors make it difficult to exclude the influence of work-hardening effects induced by deformation on the material's properties. In other words, it cannot be concluded that work-hardening is absent in the "Cold Rolled & Aged" and "Warm Rolled & Aged" samples. Therefore, the authors should further clarify the issue.
2. Regarding issue 4: The authors are requested to provide an enlarged view of the stress-strain curves in the manuscript to further illustrate the detailed determination of the yield strength.
3. Regarding issue 5: α-Ti, with its hexagonal close-packed structure, exhibits marked deformation anisotropy. If a pronounced texture develops after rolling, it will inevitably affect the mechanical properties of the alloy. The authors need to clarify this issue.
4. There is still a typo in the revised manuscript. Page 4, line 65: “TStress”.

Version 2:

Decision Letter:

** Please ensure you delete the link to your author homepage in this email if you wish to forward it to your coauthors **

Dear Dr Ackerman,

Thank you once again for submitting your manuscript, “Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys," to Communications Materials. Your manuscript has been seen again by the referees, whose comments are appended below. I am happy to say that the concerns of our reviewers have now been addressed, and that we only require some minor amendments before we can accept your paper.

Our remaining requests are:
- We recommend that author first names be written out in full rather than provided as initials.
- Affiliations must be numbered in the order of their first appearance in the author list.
- At least one corresponding author must be designated, and an e-mail address must be provided for each corresponding author (with a limit of one e-mail address per author). E-mail addresses should be supplied directly below the affiliations.

- Please provide the name and/or email address of the person who will be responsible for replying to this request in the data availability statement.
- Please provide a heading for the “Competing interests” statement in the manuscript.
- Please provide a heading for “Author Contributions” section.

This will be the final revision of your manuscript. We ask that you carefully review all files associated with your paper and follow the link below to upload the final version of all files, including display items and supplementary material. Please ensure that these files are clean, without any markups or comments, as they will be sent for publication.

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We hope to receive this updated version of your paper within 1 week, but please let us know if you find that you need more time.

We hope to receive this updated version of your paper **within 1-week,** but please let us know if you find that you need more time.

Best regards,
John Plummer, PhD
Chief Editor
orcid.org/0000-0003-4824-8497
Communications Materials

Version 3:

Decision Letter:

Dear Dr Ackerman,

We are delighted to accept your manuscript titled "Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys" for publication in Communications Materials. Thank you for choosing to publish your interesting work with us.

Acceptance of your manuscript is conditional on all authors' agreement with our publication policies. In particular, your manuscript must not be published elsewhere and there must be no announcement of the work in the media until the publication date.

Please carefully read the information below for information on what to expect from the next steps of the publishing process:

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When your article is published as the Version of Record, you will receive a notification email. **If you are planning an embargoed press release or require a specific publication date, please complete our scheduling requests form, or contact commsproduction@springernature.com, as soon as possible after acceptance and we will endeavour to accommodate your request.**

For further information on the journey of your article from acceptance to publication, please see our Author FAQs.

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Providing great service is very important to us. We would greatly appreciate any comments you have about your experience at Communications Materials. We look forward to publishing your paper, and we hope to work with you again in the future.

Best regards,
John Plummer, PhD
Chief Editor
orcid.org/0000-0003-4824-8497
Communications Materials

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Department of Materials,
Imperial College London

South Kensington Campus, London SW7 2AZ, UK

a.ackerman14@imperial.ac.uk
www.imperial.ac.uk/materials

Dr. Abigail K Ackerman BSc, PhD
Royal Academy of Engineering Research Fellow

25 September 2025

Dr John Plummer
Nature Communications Materials

Dear Dr Plummer,

Thank you for the editorial response and decision. As you have suggested in the e-mail dated 14th Aug 2025, my co-authors and I have carefully revised the paper and addressed the comments made by the reviewers. For your reference, the submission title is 'Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys', Ms. No. COMMSMAT-25-0549-T, by Ackerman, Savitzky, Ophus, Danaie, Karamched and Dye.

The response to the reviewers is attached.

I hope this corrected paper satisfies both the reviewers and editor, and is now suitable for publication in Nature Communications Materials.

Yours Respectfully,

Abigail Ackerman

Response to reviewers

We would like to thank the reviewers for their careful work in looking over our manuscript, and for their comments. Changes to the manuscript are in red; the review comments are *italicised* and our responses are in **bold** text.

First Referee:

Major Comments:

- *The authors completely eluded prior studies on so-called bi-lamellar microstructures, or even tri-modal microstructures. In particular, extensive data have been reported by Lütjering, and most trends reported in the present article are in line with his findings: lower crack growth rate, higher tensile strength, higher fatigue strength, lower ductility. Prior knowledge should be more clearly reported.*

  Thank you for this insight, this has now been added to the text. Indeed, the extensive work by Lütjering in this area should be included.

- *However, I am puzzled by the lack of deformation introduced in this prior work. Have the authors of the present manuscript made a trial without any rolling step to confirm the role of dislocations?*

  The authors have not completed this step as part of this study, as this is assumed to be the as received material. In the standard processing route, the alloy goes directly from the alpha phase processing to ageing to nucleate and grow the secondary alpha. Therefore the as received material here can be assumed to be the material without deformation. This has now been clarified in the text.

- *Is it possible that alpha precipitation proceeds preferentially on omega precipitates rather than on dislocations? From the high temperature heat treatment, residual beta may be sufficiently enriched in beta stabilizers for omega to form during heating. From the TEM images, it also appears that alpha phase precipitates away from the dislocations contained in the slip band, which I do not understand unless other nucleation sites exist. I think that it is an essential point to discuss and clarify in the article, as the role of omega phase is now well established in basketweave alpha precipitation.*

  Our TEM examinations over the years have not provided any incidental evidence of omega formation in the retained beta, and nor is there evidence in the literature that Ti-6246 forms omega, that we are aware of. For nearly 50 years, there has been evidence that more heavily beta stabilised ("near-beta") landing gear alloys such as Ti-10-2-3 and Ti-5553 form omega and can retain beta on quenching, but we aren't aware of such as suggestion in a conventional alpha-beta alloy such as Ti-6246.

More generally, this is an A/B study examining precipitation in warm rolled vs annealed material; the difference between the samples is the dislocation density, not aspects of the heat treatment that would produce omega. Most studies eg in Ti-555 that make very refined alpha using omega as a nucleant use some kind of quench (and possibly a low temperature age) cycle to produce the omega, whereas the material in the present study is cooled conventionally. Of course, Ti-10-2-3 in a conventional cooling and ageing cycle also produces fine alpha, and there are hints that this is might be nucleated from isothermal omega. But Beta-C, the spring alloy, develops very high strengths by cold rolling and ageing, which is more the concept we are following here.

To be more bold: there has been lots of work in the literature recently on the use of omega as a nucleant. It has yet to produce a ductile product, and its not yet clear if it will be industrially useful as a concept. We are more interested here in industrial processing following on from the old Beta-C work that is in the Titanium handbook, and work done in Louvain-la-Neuve on dislocations as a nucleant in Ti-555.

- *Some sentences are very long (e.g., there are only 3 sentences in the abstract). Shorter sentences would make the paper easier to read.*

  The authors have endeavoured to edit the manuscript to include shorter sentences. This has also been edited in the abstract.

- *Time constraints are probably not a relevant reason for lacking experimental data in an article of a high impact journal.*

  This sentence has now been removed.

- *There is a typo L52P4.*

  This has now been corrected.

- *Key information about experiments is missing. For example, the composition of the investigated alloy is not given, as well as details of prior thermo-mechanical processing. The strain rate for tensile tests is missing as well. Of critical importance is the heating rate of the ageing treatment, which may, or may not, promote the formation of omega phase. This is well-known from previous studies on metastable beta alloys. In particular, omega precipitation may occur in situ in the TEM experiment. Do the authors have information to provide? Different heating rates may completely change precipitation routes.*

  Some of these points are explained thoroughly in the methods section and some sections have now been clarified. We thank the reviewer for noticing some missing experimental details such as the tensile strain rate. The heating rate used was that which would be found in an industrial furnace, e.g. in a relatively thin section die forged aerofoil, rather than anything exotic.

- *I was surprised that the cold rolling route disappeared along the way. I guess that cold rolling high strength material like Ti6246 is a challenge that prevented tensile and fatigue specimens to be prepared. I may have missed this information.*

  Initial investigations were on the cold rolling route to investigate the viability and fundamental materials science of this processing route. But, it isn't viable to make, eg compressor aerofoils this way, so its only a toy process route for lab investigations (although of course, there are sheet components like nozzles...). To be able to use in industrial processing for example for compressor blades (and maybe, for small discs and big forging presses, rotor discs), this was then done warm. Therefore, to test the viability for this processing route for large scale jet engine components, mechanical testing focussed on this processing route. This has now been clarified in the text.

- *In situ TEM heating conditions are very different from conditions applied in conventional experiments, as highlighted by the authors. Can the authors elaborate on this: is 850°C measured away from the foil?*

  An explanation of the in situ heating system can be found here: link. The centre of the DENS chip is where the peak measured temperature is, and as the foil is lying flat on the chip, it is assumed that the temperature is an accurate reading. As shown in the manuscript, the literature has shown that due to thin film effects, it is common to observe temperature differences from the bulk behaviour of the material. Over the years since we first did this work, there has been a lot more work on characterising the gradients in DENS in situ heating chips, but eg changes in phase transformations suggest that they aren't too terrible.

- *I do not see the drop in strength near the yield point in figure 5.*

  This was a confusing bit of language and has now been removed.

- *With no lifetime lower than 105 cycles, there is no LCF data reported in this article.*

  The sentence on p4 line 65 has been shortened and clarified. Of course, strict definitions probably mean something like "LCF = plasticity in cycle 1" whereas "HCF = life greater than than 1E6," but these definitions are still fuzzy, so we just present S-N and step test / lifetime HCF results.

- *There is a typo L90P6: K1c is not a fatigue crack growth threshold, thus it has not been measured previously.*

  Thank you, this has now been corrected.

- *I do not understand why the paragraph L7P7 exists. It contains very vague and general statements, not really connected to findings from this work. In particular, I would not* *say that dislocations are evenly distributed in these alloys.*

This has now been removed.

Second Referee:

- *The authors attribute the improvement in strength and hardness of the alloy primarily to the refinement of the secondary alpha phase. However, after rolling deformation, lattice distortion and dislocation density increase significantly, leading to a strong work-hardening effect (i.e., enhanced strength and hardness). The authors should clarify whether the observed mechanical enhancement is dominated by the secondary alpha refinement or the work-hardening effect.*

  As shown in the inset table in Figure 5, hardness testing was completed prior to ageing to assess the strength. No increase in hardness occurred, suggesting that this is not a work hardening effect. This has been clarified in the text.

- *Regarding the 4D-STEM strain maps in Figure 4, It is not clear why the authors chose 1050 C as the in-situ heating temperature? This temperature exceeds the beta-transus temperature of Ti-6246 alloy. Under this condition, it would be expected that the dissolution of the alpha phase has occurred, accompanied by transformation into the beta phase.*

  Thank you for this observation. This has been more thoroughly clarified in both the discussion and methods sections. As we are observing a nanoscale surface area TEM foil, thin film effects have much more significant impacts than would be observed in the bulk material, typically requiring much higher temperatures than expected.

- *On page 4, line 65–67, the authors mention that 'The warm rolled and aged material shows a slight drop in strength towards its yield point.' However, this feature is not clearly shown in Figure 5. The authors should provide clarification.*

  This was incorrect and has now been removed.

- *The authors should clarify how the 0.2% yield strength values were determined for the two stress-strain curves in Figure 5. Based on the stress-strain curves, the two curves exhibit nearly overlapping behaviour (virtually linear) within the 0–1000 MPa range, indicating that their yield strengths should both exceed 1000 MPa.*

  The 0.2% yield strength was determined using ASTM E8M, the conventional standard for calculating offset stress. This involves plotting a straight line of the 0.2% offset and finding where this intersect with the tensile data. This can be seen by zooming in on the figure and observing where the as received begins to tail off just before 1000 MPa and therefore this value is correct

- *The authors propose that the microstructure resulting from the edited processing route contains all 12 secondary* α *variants without apparent variant selection. However, titanium alloys typically develop pronounced crystallographic textures following rolling deformation. The authors should clarify whether there are differences in the quantity or proportion of these 12 variants. Furthermore, the authors should discuss whether the resulting textures influence the mechanical properties of the alloy.*

  Indeed, rolling can influence crystallographic texture in titanium alloys. However, the purpose of this study and the EBSD results was to show the presence of all variants, and therefore there was no significant change in the crystallography resulting in the mechanical performance improvement. It could be argued that doing this kind of statistical analysis to show to overall texture of the secondary alpha would only be valid over a much larger area which would not be realistic due to the small scale of the phase.

- *Please add label (a) to the upper-left corner of the leftmost image in Figure 3. Scale bars should be provided into both Figures (a) and (b). Additionally, the caption for Figure 3 contains an incorrect term: "Bright Field Transmission Electron Diffraction," which requires correction.*

  This has now been corrected.

- *On page 6, line 42, "112" should be corrected to "112".*

  This has now been corrected.

- *The authors should check the language and grammar of their manuscript. Examples are as follows: 1) Page 4, line 52: "pahses".* **Corrected.** *2) Page 6, line 23 and 24: "850C" and "250C".* **Corrected.** *3) Page 6, line 27: "foilan* **Corrected.** *4) Page 6, line 42: "planesindicating".* **Corrected.** *5) Page 6, line 63: "Qui".* **Corrected.** *6) Page 7, line 12: "defectssimilar".* **Corrected.**

  The Authors would like to apologise that these issues were not spotted prior to submission - they were a result of formatting issues in LaTeX, but care has now been taken to fix these.

- *There should be a space between the numerical value and the unit. For instance, "850°C" should be corrected to "850 °C" (with a space between the numerical value and the degree Celsius symbol).*

  This has now been corrected throughout the text.

Department of Materials,
Imperial College London

South Kensington Campus, London SW7 2AZ, UK

a.ackerman14@imperial.ac.uk
www.imperial.ac.uk/materials

Dr. Abigail K Ackerman BSc, PhD
Royal Academy of Engineering Research Fellow

19 December 2025

Dr John Plummer
Nature Communications Materials

Dear Dr Plummer,

Thank you for the editorial response and decision. As you have suggested in the e-mail dated 7th Nov 2025, my co-authors and I have carefully revised the paper and addressed the comments made by the reviewers. For your reference, the submission title is 'Defect-Assisted Refinement of Nanoscale Alpha in Titanium Alloys', Ms. No. COMMSMAT-25-0549-T, by Ackerman, Savitzky, Ophus, Danaie, Karamched and Dye.

The response to the reviewers is attached.

I hope this corrected paper satisfies both the reviewers and editor, and is now suitable for publication in Nature Communications Materials.

Yours Respectfully,

Abigail Ackerman

IMPERIAL

Response to reviewers

We would like to thank the reviewers again for their careful work in looking over our manuscript, and for their comments. Changes to the manuscript are in red; the review comments are *italicised* and our responses are in **bold** text.

First Referee:

Comments:

- *The authors provided a revised version of the manuscript to address the comments of the reviewers. Overall, changes and responses to my comments are satisfactory. I am somewhat disappointed by the lack of acknowledgement of a possible role of omega, although no conclusive evidence of its absence is provided in the rebuttal. Given the differences in heat treatments between the original and the edited routes (i.e., annealing temperatures and cooling rates), a possible role cannot be ruled out so easily in my opinion, but OK.*

  We thank the reviewer again for their insightful comments regarding the omega phase.

- *Also the measured composition of the investigated alloy remains missing. .*

  The composition has not been measured in this alloy as it has been produced using aerospace production methods and meets aerospace rotor grade specifications. The nominal composition is now given in the text.

- *The rotation is given as a percentage in figure 4: is it a fraction of radians? (I missed it in the first round of reviews: sorry for that)*

  As the rotation is given as a strain, it is dimensionless, as with the other strains shown. Therefore though it is originally measured in radians, this is not the unit displayed as it is given as a percentage shift in the diffraction.

- *A number of typos remain (e.g., page 4 line 65, page 6 line 28, page 8 line 8), they might be corrected in the proofs.*

  Thank you, these have now been corrected.

Second Referee:

- *Regarding issue 1: The inset table in Figure 5 indicates that the hardness value of the "No α_s" sample is lower than that of the "As-received" sample, suggesting that the presence of α_s has an impact on the material's hardness. However, for the "Cold/Warm Rolled & Aged" samples, although their hardness has indeed increased compared to* *both the "No α_s" and "As-received" samples, deformation and the presence of fine α_s are simultaneously introduced. These factors make it difficult to exclude the influence of work-hardening effects induced by deformation on the material's properties. In other words, it cannot be concluded that work-hardening is absent in the "Cold Rolled & Aged" and "Warm Rolled & Aged" samples. Therefore, the authors should further clarify the issue.*

In all likelihood the alloy is work hardening after deformation, whether warm or cold. We have intended to pile up and store dislocations within the matrix via this deformation. Indeed, within the TEM we can see the dislocations within the β matrix. However, the purpose of the age is to both relieve this stress and to nucleate and grow the secondary α phase. The in-situ TEM data in Figure 4 shows how the secondary α is using the dislocations as a nucleation point, and therefore we can assume that this is the dominant hardening effect, rather than work hardening. This has been further clarified in the text.

- *2. Regarding issue 4: The authors are requested to provide an enlarged view of the stress-strain curves in the manuscript to further illustrate the detailed determination of the yield strength.*

  This has now been added to Figure 5.

- *3. Regarding issue 5: α-Ti, with its hexagonal close-packed structure, exhibits marked deformation anisotropy. If a pronounced texture develops after rolling, it will inevitably affect the mechanical properties of the alloy. The authors need to clarify this issue.*

  We agree that the deformation anisotropy of α-Ti and the resulting texture evolution during rolling can significantly influence the alloy's mechanical behaviour. However, this is mainly observed in the primary α texture, which has not changed in this study. Traditional secondary α texture is influenced by the parent primary α texture, as explained in the introduction and reference 10. Here, we are showing that the deformation has not had an impact on the texture by the presence of all 12 variants and therefore the mechanical properties have been influenced solely by the size change of the secondary α. This has now been clarified in the text.

- *4. There is still a typo in the revised manuscript. Page 4, line 65: "TStress".*

  This has now been corrected.