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4th Dec 20

Dear Dr Trindade,

Thank you for submitting your manuscript, "Residual polymer stabiliser causes anisotropic conductivity in metal nanoparticle 2D and 3D printed electronics", to Communications Materials. It has now been seen by 3 referees, whose comments are appended below. You will see that while they find your work of interest, some important points are raised. We are interested in the possibility of publishing your study in Communications Materials, but would like to consider your response to these concerns in the form of a revised manuscript before we make a decision on publication.

You will see in particular that Reviewer 1 and 2 are questioning the vertical resistivity measurement. We would expect to see this question addressed in the revised paper. Reviewer 3 is also asking about measurement artifacts. We would naturally expect the other comments and requests from all the referees to be responded to also. We therefore invite you to revise and resubmit your manuscript, taking into account the points raised.

We are committed to providing a fair and constructive peer-review process. Please don't hesitate to contact us if you wish to discuss the revision in more detail.

When submitting your revised manuscript, please include the following:

- A response letter with a point-by-point reply to each of the referee comments and a description of changes made. Please include the complete referee report in the response letter. Please note that the response letter must be separate to the cover letter to the editors.

- A marked-up version of the manuscript with all changes to the text in a different colored font. Please do not include tracked changes or comments. Please select the file type 'Revised Manuscript - Marked Up' when uploading the manuscript file to our online system.

- A clean version of the manuscript. Please select the file type 'Article File'.

- An updated <https://www.nature.com/documents/nr-editorial-policy-checklist.zip> Editorial Policy checklist, uploaded as a 'Related Manuscript File' type. This checklist is to ensure your paper complies with all relevant editorial policies. If needed, please revise your manuscript in response to these points. Please note that this form is a dynamic 'smart pdf' and must therefore be downloaded and completed in Adobe Reader. Clicking this link will download a zip file containing the pdf.

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We hope to receive your revised paper within three months; please let us know if you aren't able to submit it within this time so that we can discuss how best to proceed. If we don't hear from you, and the revision process takes significantly longer, we will close your file. In this event, we will still be happy to reconsider your paper at a later date, as long as nothing similar has been accepted for publication at Communications Materials or published elsewhere in the meantime.

We understand that due to the current global situation, the time required for revision may be longer than usual. We would appreciate it if you could keep us informed about an estimated timescale for resubmission, to facilitate our planning. Of course, if you are unable to estimate, we are happy to accommodate necessary extensions nevertheless.

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,

Jie Xu, PhD
Editorial Board Member
Communications Materials
orcid.org/0000-0001-7842-5496

Reviewers' comments:

Reviewer #1 (Remarks to the Author):

The authors demonstrated the mechanism of conductivity degradation by residual PVP in inkjet printed AgNPs. Although the conductivity degradation by PVP is well known and obvious, the authors characterized this in depth, using various characterization tools including ToF-SIMS, XPS, SEM, TEM, and OrbiSIMS. The residual PVP mitigates to the top and bottom surface of the printed trace, and affects the conductivity. Furthermore, the descriptions (texts and figures) are very clear. Therefore, the reviewer can recommend the publication of this manuscript after the revisions of the following concerns to verify the claim by the authors.

1. The accuracy of the vertical resistivity measurement is doubtful. ITO does not show low resistance, and Ag paste also contains organic residues which can influence the resistivity measurement. The authors should verify the accuracy by, for example, discussing the effect of thickness in the measured value.

2. The functionality to sense strain by the strain sensor in Figure 5 is not demonstrated even in reference 4 and 43. Thus, the description like “Fig. 5 Presence of residual stabiliser in a multi-material inkjet 3D printed encapsulated strain sensor.” is misleading. The descriptions in “Presence of residual stabiliser in a multi-material inkjet 3D printed encapsulated strain sensor.” in Page 11 are a kind of obvious without the other major part of the manuscript. The authors should demonstrate the importance of their findings in a different way.

Reviewer #2 (Remarks to the Author):

This is a very interesting paper, that attempts addressing well known and important points in the field of inkjet printing of conductive materials. The attempt is laudable, though I see several "dangerous" claims, that are too superficially cast by the authors.

1) I do not agree on the fact that 90 °C is sufficient for pinning without triggering sinterization. Particularly comparing this claim with your own admission that the planar resistivity of the sample decreased dramatically after processing at 100 °C. If 100 is enough to produce sinterization, you achieve 90% of that temperature, which is already too much. Two thirds are already enough, so you should stay below 65 °C.

2) You never discuss about surface tension, which is fundamental, particularly considering your substrate choice (silicon). The structures I see in figure 4c at 500 °C are clearly showing a de-wetting phenomenon. I bet using glass instead of silicon would dramatically change your results. Polyimide as well. So you should be very careful in casting conclusions to the generic application, as what was found is a very particular case.

3) Measuring the vertical resistivity should not be done with the particular setup shown in figure 4b. As far as I understand you are collecting the resistivity of the whole device, and correlating measurements with the thickness of the sample (normalized with a special sample having 50 layers, as reported in the methods section). The problem is that the device is printed on ITO, which is not silicon. Their surface tension is different, therefore the morphology of layers cannot be identical. Furthermore, ITO has particular crystalline structure, that can induce conformal growth during sintering. The measurements cannot be compared. To measure vertical component, I think the only technique that could be implemented is a in situ characterization with nanoprobe, positioned for example on a section of the printed and sintered device.

Besides this, some minor point are also evidenced:

1) for the reviewer's sake, and you own, please provide numbered lines

2) Introduction, page 2, line 4: also intrinsically conductive polymers are printable colloids, see e.g. [Adv. Funct. Mater. 2014, 24, 3375–3383], and magnetic nanoparticles [Eur. Phys. J. B (2013) 86: 173]

3) Introduction, page 2, line 22: not only depends on temperature, but also on time, or more specifically the thermal profile (heating time), see e.g. figure 6 [Microelectronic Engineering 97 (2012) 8–15]

4) Results, page 3, line 1: "pinning"

5) Figure 1, I suggest to include the Ag NP diameter distribution because it is a fundamental part of

your study

- 6) page 5: so the average layer is around 300 nm, the average NP is 60 nm in diameter, can we deduce that your layer is approximately composed by 6 monolayers of stacked NPs?
- 7) page 9, line 4: was the oven treatment in air? vacuum?
- 8) figure 5b: scale bar missing
- 9) page 14: is the ink coming in TGME without other solvents? No water? No ethanol?
- 10) Supporting Information page 3 figure S3a: I see some nanorods there! This is important, could you please provide also a histogram showing the aspect ratio distribution of your particles? (1=sphere, 2=rod whose length is twice the diameter)

Reviewer #3 (Remarks to the Author):

In this manuscript, Trindade et al. presented a study about the effect of organic residuals from inkjet inks on the property of printed metal nanoparticles. The authors utilized multiple chemical and functional imaging tools to understand the distribution of organic residuals in the printed nanoparticles and electronic devices. This work is helpful to the development of printed electronics. Overall, the manuscript is well written and valuable. Therefore, I recommend publish it on Communications Materials after the authors address the below comments.

1. In Figure 2b, the organic species (red curve) show a very high signal near the surface. Could the authors comment on the origin of the high signal: is it the real PVP signal or surface hydrocarbon contamination?
2. The authors observed higher PVP concentration near-surface and lower PVP concentration near-bottom in ToF-SIMS results. Since Harvey et al. revealed that, in SIMS measurement, the gradient of organic species could be a measurement artifact [Harvey, Steven P., et al. "Investigating the Effects of Chemical Gradients on Performance and Reliability within Perovskite Solar Cells with TOF-SIMS." *Advanced Energy Materials* (2020): 1903674.; Harvey, Steven P., et al. "Understanding Measurement Artifacts Causing Inherent Cation Gradients in Depth Profiles of Perovskite Photovoltaics with TOF-SIMS." 2019 IEEE 46th Photovoltaic Specialists Conference (PVSC). IEEE, 2019.], could the authors comment on if they considered measurement artifacts in this study? It will be better if the author can perform some measurement to rule out the effect of measurement artifacts. For example, Liu et al. [Liu, Yongtao, et al. "Strain–Chemical Gradient and Polarization in Metal Halide Perovskites." *Advanced Electronic Materials* 6.4 (2020): 1901235.] ruled out the effect of measurement artifacts by changing the distribution of organic species.
3. In the TEM micrograph, the authors observed penetration of AgNPs into the dielectric material, so how this interpenetration affects the performance and stability of electronics?

Re **"Residual polymer stabiliser causes anisotropic conductivity in metal nanoparticle 2D and 3D printed electronics"**, for publication in *Communications Materials*. (COMMSMAT-20-0326A).

Reviewers' comments:

Reviewer #1 (Remarks to the Author):

The authors demonstrated the mechanism of conductivity degradation by residual PVP in inkjet printed AgNPs. Although the conductivity degradation by PVP is well known and obvious, the authors characterized this in depth, using various characterization tools including ToF-SIMS, XPS, SEM, TEM, and OrbiSIMS. The residual PVP mitigates to the top and bottom surface of the printed trace, and affects the conductivity. Furthermore, the descriptions (texts and figures) are very clear. Therefore, the reviewer can recommend the publication of this manuscript after the revisions of the following concerns to verify the claim by the authors.

1. The accuracy of the vertical resistivity measurement is doubtful. ITO does not show low resistance, and Ag paste also contains organic residues which can influence the resistivity measurement. The authors should verify the accuracy by, for example, discussing the effect of thickness in the measured value.

R: To measure vertical resistance of our printed AgNP layers, we use silver paint ($R < 1 \Omega$) and ITO coated glass ($R \sim 10 \Omega/\text{sq}$) with resistance lower than that of the measured sample. We have also measured vertical resistance on the printed samples with different number of printed layers (100 and 200 layers) and obtained comparable values of resistivity. We have included these results in the revised manuscript on page 12 (In the section "Residual PVP in a multi-material 3D printed device") and in Methods (page 16) to clarify this point.

2. The functionality to sense strain by the strain sensor in Figure 5 is not demonstrated even in reference 4 and 43. Thus, the description like "Fig. 5 Presence of residual stabiliser in a multi-material inkjet 3D printed encapsulated strain sensor." is misleading. The descriptions in "Presence of residual stabiliser in a multi-material inkjet 3D printed encapsulated strain sensor." in Page 11 are a kind of obvious without the other major part of the manuscript. The authors should demonstrate the importance of their findings in a different way.

R: We revised that manuscript and refer to the device as "a multi-material inkjet 3D printed encapsulated conductive pattern". We believe this change is appropriate as it focuses on the properties of materials in this structure. With regards to the text in Page 11, our intention is to illustrate the use of advanced characterisation technique to gain valuable insights into the morphology and composition of inkjetted samples. Here we confirmed the presence of trapped PVP residues at the interface between the printed layers. We demonstrated that photoreactive acrylate ink did not allow the diffusion of the PVP at the interface to either into the metallic phase or into the polymeric phase. The understanding of this not only informs how PVP affects the interface between the two materials, but also whether it could lead to material properties change.

Such a description on its own would be sufficient to prove the presence of PVP but the results provided in the previous, more systematic sections of the manuscript go beyond identification of the residues and provide insights into the reason and mechanism of which PVP migrates to surfaces/interfaces. In more complex cases such as inkjet printing of optoelectronic devices, a controlled level of interface chemistry/morphology is essential to achieve the desired performance for example in our recent work where we inkjet-printed a graphene transistor and observed very different conductivity and transport behaviours between printed graphene layer alone and printed graphene layer with silver electrodes on top (*Adv. Funct. Mater.* 2020, 2007478). As observed by SEM and suggested by modelling, the penetration has doped the graphene layer which changes the transport property in graphene. Upon revision, we have now included sentences to highlight the relevance of having those results (Page 12, line 31).

Reviewer #2 (Remarks to the Author):

This is a very interesting paper, that attempts addressing well known and important points in the field of inkjet printing of conductive materials. The attempt is laudable, though I see several "dangerous" claims, that are too superficially cast by the authors.

1) I do not agree on the fact that 90 °C is sufficient for pinning without triggering sinterization. Particularly comparing this claim with your own admission that the planar resistivity of the sample decreased dramatically after processing at 100 °C. If 100 is enough to produce sinterization, you achieve 90% of that temperature, which is already too much. Two thirds are already enough, so you should stay below 65 °C.

R: We chose 90 °C based on a systematic analysis that we carried out on the sintering behaviour of this specific AgNPs ink and observed that sintering does not significantly start until higher temperatures (Supporting Figure S6). and pinning with minimal sintering is observed. We have revised the manuscript throughout to refer to this temperature as T at which minimal sintering is observed. We have updated Figure 4 with an inset to show the resistivity measurements in more detail and included more micrographs for samples treated at different temperatures.

2) You never discuss about surface tension, which is fundamental, particularly considering your substrate choice (silicon). The structures I see in figure 4c at 500 °C are clearly showing a de-wetting phenomenon. I bet using glass instead of silicon would dramatically change your results. Polyimide as well. So you should be very careful in casting conclusions to the generic application, as what was found is a very particular case.

R: As the reviewer commented, the wetting property of the melted silver would be different depending on substrates and this will influence the final conductivity. What we intended to show is that when the post treatment temperature stays below the melting point of AgNPs (in the sintering temperature range we tested), only interconnection and bridging will form between particles instead of complete coalescence. Therefore, conductivity will improve. However, if the temperature reaches the melting range, melted

particles can fully coalesce and cause de-wetting, which may lead to significant conductivity reduction. The de-wetting phenomenon will vary depending on substrate properties. To avoid this speculation, we have now made clearer in the manuscript that the conclusions are with regards to silicon wafer substrates only (Page 9, line 21).

3) Measuring the vertical resistivity should not be done with the particular setup shown in figure 4b. As far as I understand you are collecting the resistivity of the whole device, and correlating measurements with the thickness of the sample (normalized with a special sample having 50 layers, as reported in the methods section). The problem is that the device is printed on ITO, which is not silicon. Their surface tension is different, therefore the morphology of layers cannot be identical. Furthermore, ITO has particular crystalline structure, that can induce conformal growth during sintering. The measurements cannot be compared. To measure vertical component, I think the only technique that could be implemented is a in situ characterization with nanoprobe, positioned for example on a section of the printed and sintered device.

R: We agree with the reviewer that the measurements of vertical resistance on these samples are challenging. To ensure that the ITO is suitable as bottom electrode we first confirmed that the deposition of the AgNP ink is comparable on ITO and on Si/SiO₂. We compared the width of a AgNP line deposited with the same printing parameters onto Si/SiO₂ and onto glass/ITO substrates and found the width of the printed lines was ~ 45 µm for both substrates. We have also obtained comparable values of vertical resistivity from samples with different numbers of printed layers, confirming suitability of ITO for these measurements. This information has been added to page 16 of the revised manuscript.

The in-situ assessment of conductivity during annealing could indeed provide an interesting insight, however since we anneal our samples in Argon, this measurement is currently not possible in our lab. We will consider nanoprobe measurement in our future work, though achieving good bottom contact will remain a challenge.

Besides this, some minor point are also evidenced: 1) for the reviewer's sake, and you own, please provide numbered lines

R: The revised version has now numbered lines.

2) Introduction, page 2, line 4: also intrinsically conductive polymers are printable colloids, see e.g. [Adv. Funct. Mater. 2014, 24, 3375–3383], and magnetic nanoparticles [Eur. Phys. J. B (2013) 86: 173]

R: We thank the reviewer for these references which have now been included.

3) Introduction, page 2, line 22: not only depends on temperature, but also on time, or more specifically the thermal profile (heating time), see e.g. figure 6 [Microelectronic Engineering 97 (2012) 8–15]

R: The text has been updated with the information.

4) Results, page 3, line 1: "pinning"

R: Fixed.

5) Figure 1, I suggest to include the Ag NP diameter distribution because it is a fundamental part of your study

R: We now use the caption of Figure 1 to refer the reader to Supplementary Figure S3.

6) page 5: so the average layer is around 300 nm, the average NP is 60 nm in diameter, can we deduce that your layer is approximately composed by 6 monolayers of stacked NPs?

R: We believe this is a fair approximation. In fact, we have further recent results with such information from a cross-sectional analysis of an inkjet printed graphene transistor (*Adv. Funct. Mater.* 2020, 2007478). The results show that one layer is approximately composed of 6 to 8 monolayers of stacked NPs. We have now included a reference to the paper with these results in (Page 5, line 16).

7) page 9, line 4: was the oven treatment in air? vacuum?

R: The oven treatment was done in argon. This information has now been added to the manuscript.

8) figure 5d: scale bar missing

R: Fixed.

9) page 14: is the ink coming in TGME without other solvents? No water? No ethanol?

R: Based on the manufacturer, the ink contains TGME only as a solvent (http://anapro.com/eng/product/silver_inkjet_ink.html).

10) Supporting Information page 3 figure S3a: I see some nanorods there! This is important, could you please provide also a histogram showing the aspect ratio distribution of your particles? (1=sphere, 2=rod whose length is twice the diameter)

R: We have now provided the aspect ratio distribution as an inset in Figure S3.

Reviewer #3 (Remarks to the Author):

In this manuscript, Trindade et al. presented a study about the effect of organic residuals from inkjet inks on the property of printed metal nanoparticles. The authors utilized multiple chemical and functional imaging tools to understand the distribution of organic residuals in the printed nanoparticles and electronic devices. This work is helpful to the development of printed electronics. Overall, the manuscript is well written and valuable. Therefore, I recommend publish it on Communications Materials after the authors address the below comments.

1. In Figure 2b, the organic species (red curve) show a very high signal near the surface. Could the authors comment on the origin of the high signal: is it the real PVP signal or surface hydrocarbon contamination?

R: Due to the high specificity of the ToF/Orbi SIMS technique, this is definitely a real PVP signal ($C_6H_{10}NO^+$ ion). Based on the XPS results, before temperature treatment, the top surface is richer in a non-interacting form of PVP, so we attribute the higher SIMS signal to surface segregation of “free” PVP. This is then again observed in the multi-material device with a high intensity of PVP at the organic/metal interface. This information is in the manuscript on Page 3, line 17: “As a result, we have found polyvinylpyrrolidone (PVP) on the surface of printed layers of AgNPs by means of the unambiguous identification of characteristic PVP signal [36,52]”.

2. The authors observed higher PVP concentration near-surface and lower PVP concentration near-bottom in ToF-SIMS results. Since Harvey et al. revealed that, in SIMS measurement, the gradient of organic species could be a measurement artifact [Harvey, Steven P., et al. "Investigating the Effects of Chemical Gradients on Performance and Reliability within Perovskite Solar Cells with TOF-SIMS." Advanced Energy Materials (2020): 1903674.; Harvey, Steven P., et al. "Understanding Measurement Artifacts Causing Inherent Cation Gradients in Depth Profiles of Perovskite Photovoltaics with TOF-SIMS." 2019 IEEE 46th Photovoltaic Specialists Conference (PVSC). IEEE, 2019.], could the authors comment on if they considered measurement artifacts in this study? It will be better if the author can perform some measurement to rule out the effect of measurement artifacts. For example, Liu et al. [Liu, Yongtao, et al. "Strain-Chemical Gradient and Polarization in Metal Halide Perovskites." Advanced Electronic Materials 6.4 (2020): 1901235.] ruled out the effect of measurement artifacts by changing the distribution of organic species.

R: We thank the reviewer for these important remarks and, since we propose that this method can be applied to complex samples, we have decided to include a few sentences about our awareness of measurement artifacts. (Page 7, line 1). However, specifically for this work, we conclude that the PVP depth distribution is not influenced by such measurement artefacts. This is based on a conjunction of consistent measurements that include: orbiSIMS data; ToF-SIMS data of single droplets before and after etching; ToF-SIMS depth profiling data of single layer samples without treatment and treated under various temperatures (Figure 4 and Figure S2) and XPS data of various samples before and after GCIB etching (surface vs. bulk, Figures 2 and 4). Moreover, differently to more complex samples (such as PSCs), there is no significant variation of chemical matrix across the profiles.

3. In the TEM micrograph, the authors observed penetration of AgNPs into the dielectric material, so how this interpenetration affects the performance and stability of electronics?

R: AgNPs interpenetration can affect printed electronics in several ways. A high level of interpenetration may affect cohesion between nanoparticles and consequently electrical conductivity. In more complex cases such as inkjet printing of optoelectronic devices, a controlled level of interpenetration is essential to achieve the desired performance for example in our recent work where we inkjet-printed a graphene transistor and observed very different conductivity and transport behaviours between printed graphene layer alone and printed graphene layer with silver electrodes on top (*Adv. Funct. Mater.* 2020, 2007478). As observed by SEM and suggested by modelling, the penetration has doped the graphene layer which changes the transport property in graphene. Moreover, since the devices are made in an additive fashion, top and bottom interfaces may present different levels of adhesion not only due to possible different materials on each side, but also (specifically for the device in this manuscript) related to the previously reported fact that the top surface of photocurable inkjet printed resins will have a lower degree of conversion monomer-to-polymer, thus potentially facilitating NPs interpenetration. We have now included this discussion in the manuscript (Page 12, line 31).

Yours faithfully

Dr Gustavo F Trindade

29th Mar 21

Dear Dr Trindade,

Thank you for submitting your revised manuscript, "Residual polymer stabiliser causes anisotropic conductivity in metal nanoparticle 2D and 3D printed electronics", to Communications Materials and please accept our apologies for the delay in getting back to you. It has now been seen again by Reviewer 1 and 2, whose comments appear below. Reviewer 3 did not respond to our requests to look at the revised paper (causing the delay) and we therefore checked with one of the other referees that the manuscript had been revised appropriately per the comments of Reviewer 3.

In light of their advice I am delighted to say that we are happy, in principle, to publish a suitably revised version in Communications Materials under the open access CC BY license (Creative Commons Attribution v4.0 International License).

We therefore invite you to edit your manuscript to comply with our journal policies and formatting style in order to maximise the accessibility and therefore the impact of your work.

EDITORIAL REQUESTS

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* An updated editorial policy checklist that verifies compliance with all required editorial policies must be completed and uploaded with the revised manuscript. All points on the policy checklist must be addressed; if needed, please revise your manuscript in response to these points. Please note that this form is a dynamic 'smart pdf' and must therefore be downloaded and completed in Adobe Reader. Clicking this link will download a zip file containing the pdf.

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We hope to hear from you within two weeks; please let us know if the process may take longer.

Best regards,

Jie Xu, PhD
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orcid.org/0000-0001-7842-5496

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

REVIEWERS' COMMENTS:

Reviewer #1 (Remarks to the Author):

The authors properly addressed the reviewer's concerns. I recommend the publication.

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The authors did a good job to reply to all of the comments. I am happy with the paper in its current

state.

Re " ***Residual polymer stabiliser causes anisotropic electrical conductivity during inkjet printing of metal nanoparticles***", for publication in *Communications Materials*. (COMMSMAT-20-0326B).

REVIEWERS' COMMENTS:

We thank the reviewers for their comments and believe the final version is an improvement over the original submission

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