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Responses to Editor's and Reviewers' Comments

[The comments are in black, and authors' responses are in blue.]

The authors would like to thank the editor and reviewers for their valuable time and effort for the evaluation of the present manuscript.

Responses to Editorial Comments

[Editorial comments are in black, and authors' responses are in blue.]

1.Manuscript would benefit from copyediting for grammatical errors throughout. The discussion is particularly difficult to understand: the last sentence of the second paragraph is unclear, and the last sentence of the fifth paragraph consists of two phrases that do not equal a complete sentence.

The particularly mentioned grammatical errors have been corrected and the whole manuscript has been proof read.

2. Formatting:
-Please submit multipanel figures in a single file.
-Missing step – 1.5.2
-Section 1 refers almost exclusively to nanoparticles. Section 2 refers to micro-spheres. Are these the same thing? If so, one consistent term should be used throughout the protocol.

Step 1.5.2 is included. And, in the author's published paper (Ref [1]), the size of spheres ranges from 0.6 microns to 10 microns. Therefore, the terms that depend on the diameter of the sphere have been changed to the consistent term "micro/nano-sphere."

Did you request a single pdf file which contains all 15 figures?

-"Required" after "Acknowledgements" should be removed.

Removed.

-References: Please abbreviate all journal titles.

Done.

3. Visualization/Animation:
-The animated Figure 1 is a powerpoint file with multiple slides. To be included in the text manuscript, Figure 1 should be submitted in a static format (e.g. jpeg, tif, etc.). The PPT file can still be used to generate the animation in the video.

Animation 1 is still referenced in the text, but a static format has been added as two new figures, as Figure 10 and Figure 12.

-Please include steps 2.4.2 and 2.4.4 in the SW file with suggested visualization.

Steps 2.4.2 and 2.4.4 have been added to the SW file including Figure 10 and 12, which are the static format of Animation 1.

4. Additional detail is required:
-1.1.2 – What does “and now vapor” mean? Does this refer to the toluene or another component?

I have removed “and now vapor”; this was referencing the bubbled toluene.

1.1.2) When the reactor reaches 1000°C, add the bubbled argon at a rate of 240-300ml/min and bubbled toluene at a rate of 90-300ml/min. Allow this run for 60mins.

-1.5.1 – Which inset? Figure 5 does not appear to have one.

Figure 5a inset was the same representation on Figure 5. Figure 5a has been removed.

-What recipe is used to coat the nanoparticles in BN?

1.5.2) You can create any recipe needed depending on the parameters you want. An example of a recipe to obtain a very thick shell around 120nm would include N2 at a constant rate of 60sccm, B2H6 at 15sccm, an RF of 300mW/cm2, a temperature of 400°C, and a run time of 400mins.

5. Please keep the editorial comments from your previous revisions in mind as you revise your manuscript to address peer review comments. For instance, if formatting or other changes were made, commercial language was removed, etc., please maintain these overall manuscript changes.

6. Please take this opportunity to thoroughly proofread your manuscript to ensure that there are no spelling or grammar issues. Your JoVE editor will not copy-edit your manuscript and any errors in your submitted revision may be present in the published version.

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The "Re-print with permission from Ref. []" has been added in the figure caption of proper figures. The re-print permission will be obtained from the published journal, and we will provide them when this manuscript is finally accepted.

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DOIs are included.

Responses to Reviewers' Comments

[Reviewers' comments are in black, and authors' responses are in blue.]

Reviewer #1:
The present manuscript describes the fabrication of single and coated SiC nano-sphere, and discusses about the measurement of the spectral emissivity of the SiC nano-spheres by FTIR. In overall, the structure of this manuscript is not very well organized, the figures can be further improved, and the discussion can be more in depth. Therefore, the reviewer would suggest a major revision for this manuscript.

1. The figures of this manuscript could be improved. For example, in line 288, the author says "Theoretical calculations give representative results of the spectral emissivity of a SiC half-space, single SiC spheres and a SiC sphere coated with a Boron Nitride (BN), as shown in Figure 13a." ,which is not found in Figure 13a. Other figures like Figure 8-12 might not be that necessary.

For this, Figure 13a and 13b accidentally have been switched. This problem has been corrected. The other figures are screenshots, which were requested by editor in the previous review. They are necessary to film the video.

2. The reviewer has something not sure about the direct measurement of spectral emissivity by FTIR. As the measured sample is a very small nano-sphere attached to a cantilever, how could the optics be focused onto such a small spot so the FTIR is only measuring the emitted energy from the nano-sphere? Another concern is that whether the thermal emission from the cantilever and the thermal cell could act as noise and affect the accuracy of the measurement, is there any noise correction methods? Also, will the signal be too weak from a single nano-sphere, how is the signal to noise ratio for the emittance measurement?

Now, a method of correcting the noise is presented in the manuscript. The reviewer's concern is right. A noise-measurement has to be made first, with only the thermal cell and the cantilever without attaching the micro/nano-sphere. It will be subtracted from the emissivity measurement in the end.

Since nobody measured the emissivity of micro/nano-spheres before, neither did the authors, the signal to noise ratios or information about the strength of the signal for micro/nano-spheres cannot be found. Based on the authors' published work in Journal of Applied Physics this February, the authors received

an invitation from JoVE's editor. In this manuscript, the authors explained the theoretical results from the published work, and then described an experimental method and the apparatus for the measurement of the emissivity of micro/nano-spheres. But the experiment was not done.

Reviewer #2:
This paper describes a procedure for fabricating micro/nano-spheres of SiC and coated SiC nano-spheres. Additionally, a technique for measuring the spectral emissivity of an individual sphere via a FTIR is described. The topic of this paper is very interesting, as micro/nano-spheres of SiC can potentially be used for controlling thermal emission, both in the near and far field. However, the paper is quite confusing, mainly due to the Introduction. In the first four paragraphs of the Introduction, the authors discuss various disjointed topics that are not always relevant to the main topic of the paper.

If my understanding of the manuscript is correct, the authors are interested by far-field thermal emission control with resonant micro/nanoparticles. The authors already established a theoretical framework for computing the spectral emissivity of micro/nano-spheres. They are now interested in fabricating these spheres. They also want to measure the spectral emissivity of individual sphere, which is a very interesting topic. Using this knowledge, it would be possible to design metamaterials / photonic crystals made of particles for controlling thermal radiation emission. If this is an OK summary of the paper, what is the point of talking about momentum transfer, entropy transfer, cross-spectral densities, vector Helmholtz equations, etc. in the Introduction? Except confusing the reader with unnecessary details, what is the purpose of talking about this? I suggest the authors to re-write the first four paragraphs of the Introduction in such a way that the reader will understand the knowledge gap in the literature and the objective / contribution of the paper.

The authors are indebted to reviewer's time to evaluate this manuscript. This manuscript explained the theoretical results from the published work in Ref. [1], and then described an experimental method and the apparatus for the measurement of the emissivity of micro/nano-spheres. The introduction section introduces the background of the classical and near-field thermal radiation, history, and the numerical method of calculating it. According to JoVE's requirement, this manuscript focuses mainly on the experimental method and process, so the authors skipped the tedious derivation and calculations, which have been explained well in Ref. [1].

Because the theoretical method used for calculation of emissivity is based on fluctuation-dissipation theorem, cross-spectral densities, and dyadic Green's functions, which can be used to determine not only energy transfer, but also entropy and momentum transfer. Though this manuscript focuses on experimental measurement mostly, in the introduction section, the authors still would like to talk about how to apply the knowledge, such as cross-spectral densities, vector Helmholtz equations. The introduction section is to introduce the background and broad impact of this work, but not a summary. The conclusion section is.

I also have a series of comments:

1. The English is generally OK, but it is not great. Some sentences are grammatically incorrect. Please take some time reviewing the English. This will help improving the clarity of the manuscript.

The grammar and spelling in the manuscript has been reviewed.

2. The authors should look at the work of Wheeler at the University of Toronto. Wheeler studied

theoretically and experimentally Mie resonance-based metamaterials made of resonant micro/nanoparticles such as SiC.

The authors thank the reviewer bringing this useful paper, which can help us better understand the validity and application of the Mie theory.

3. Lines 64-65: What are the size and length scale? This is not clear. Do you mean size of the objects and their separation distance?

Yes, it is the size of the objects. Here, it is micro/nano-spheres.

4. Line 70: "size and scale dependence are typical characteristics of near-field phenomena" is not clear.

Near-field thermal radiation depends on the size, geometry of the objects and the separation distance, unlike classical thermal radiation.

5. Line 80: Ref. 38 is not about cylindrical objects. Ref. 38 discusses spheres and cubes.

Yes, Ref. [38] has been cited for spherical objects, instead of cylindrical ones.

6. Lines 83-85: "these phenomena for a single object requires a further study and a good understanding when the size of the object is reduced to be comparable or less than the thermal wavelength." This sentence seems important in describing the motivation underlying the paper. However, it is everything but clear. Basically, you are interested in analyzing the size effect on the emissivity of resonant micro/nano-spheres, right?

Yes, the authors are interested in the size effects on the emissivity of small-scale spheres. Please see the theoretical results as shown in Figure 15b. It shows the emissivity of micro/nano sized spheres for various radii.

7. Line 122: Change "mirco" by "micro".

It has been changed.

8. Line 129: Define the abbreviation BN in the text.

It has been defined in the introduction.

9. Do you have any experimental spectral emissivity that you could compare against theoretical predictions? The results shown in Fig. 13 are only predictions, correct?

These results shown in Figure 15 are the theoretical predictions. The authors received an invitation from JoVE's editor based on the authors' published work in Journal of Applied Physics this February. That was a theoretical but not an experimental work. In this manuscript, the authors explained the theoretical results from the published work, and then described an experimental method and the apparatus for the measurement of the emissivity of micro/nano-spheres. But we have not conducted any experimental

emissivity measurements.

10. Lines 288-314: The discussion of the results shown in Fig. 13 is quite unclear. After reading this paragraph, it is still difficult to interpret the spectra plotted in Fig. 13.

These are based on the theoretical calculations, which are described in more details in Ref. [1]. Please refer Fig. 2 on page 5 of Ref. [1].

Reviewer #3:
The authors have measured the spectral emissivity of SiC micro/nano-spheres and coated SiC nano-spheres. This is the first time that I have seen the study about the spectral emissivity of micro-nano-spheres. This is a very interesting work. The paper is well written and has very insightful data relating to the spectral emissivity of SiC micro/nano-spheres. The conclusions are sound and the prior art is cited adequately. I believe it can be published in Journal of Visualized Experiments.

I want to ask the authors some questions about the experiment.
1. The experiment procedures have been descried in the manuscript, but I haven't found the introduction about the measurement uncertainty of the measurement apparatus. I think it is very important for a measurement apparatus.

Since nobody measured the spectral emissivity of micro/nano-spheres before, neither did we, there is no information about the experimental results. Regarding the measurement uncertainty, it mainly arises from the fabrication of the micro/nano-spheres, for example, the surface roughness that can be comparable with the size of sphere. That would affect the measurement.

2. When the sample radiation source was measured by FTIR, the emission spectrum should include the emission of the sphere and micro-cantilever. The authors is how to deal with the problem. It should be described in the manuscript clearly.

As mentioned before, our idea is to make a noise measurement with only the thermal chamber and the cantilever, so that the noise (signal/response) from the background and cantilever alone can be subtracted from the total spectral emissivity in the end. This has been described in the manuscript.

3. The optical box shown in Figure7 is not clear, I couldn't find the clear light path. I consider that Figure 7 is problematic from the Animation1. Is the rotating mirror a parabolic mirror?

The figure is from the company Bruker Optics, which can be found via the link below.
https://www.bruker.com/fileadmin/user_upload/8-PDF-Docs/OpticalSpectrospcopy/FT-IR/VERTEX/AN/AN99_SolarThermalMaterials_EN.pdf

Best regards,

Yi Zheng, Ph.D.