

Beamstop-free small-angle X-ray scattering for multiscale structure measurement of high-energy-density plasma

Corresponding Author: Professor Masato Ota

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

Attachments originally included by the reviewers as part of their assessment can be found at the end of this file.

Version 0:

Reviewer comments:

Reviewer #1

(Remarks to the Author)

The manuscript of M. Ota et al. reports on the use of a layered system of metallic filters to attenuate the direct X-ray beam of an X-FEL source in order to perform SAXS without a beamstop. The system was successfully employed for the study of a 2D array of columnar plasma obtained by irradiating an assembly of silicon rods with a high intensity laser pulse. A wide angular range, including the area which is usually masked by the beamstop, allowed a better study of the plasma scale length and of its initial evolution with respect to the SAXS measurement in the presence of the beamstop.

The presented research is innovative and of great interest both for the SAXS and for the plasma communities. The results are fully supported by the experiments and by the data analysis. The theoretical background is well described and fitting to the experimental results.

The manuscript could benefit from some changes to further improve its readability.

In the following, details are provided.

General considerations

- Formatting (introduction, results, discussion) could improve readability
- The authors could comment in the introduction part, whether similar approaches for beamstop-free SAXS exist.
- More details on the filter system could be given (in the main text or in the supplementary information). Why was a triangular shape used? Why two sets of filters with three triangles? Why the two set of triangles have opposite directions? Which was the dimension of the triangles? Why are L1 and L2 combined?
- Last paragraph here the results that motivate the selected approach to study plasma using SAXS without beamstop are highlighted. A sentence about the research outcome in the abstract and in the introduction would greatly help in connecting the parts directly from the beginning.

Specific comments in the text

Main text

Line 49: with wide-range scattering signal is it intended a wide q-range/angular scale?

Line 71: a connection sentence could be added to introduce early on that the Si-rods were used in the later experiment. Or the sentence at line 93 could be moved here.

Line 84: a more specific one-sentence explanation of what specifically the beamstop was limiting could be useful.

Otherwise, the sentences from line 100 to 104 could be moved here to really strengthen the force of the present research compared to the previous one.

Line 97: it is stated that Utilizing layered triangle-shaped metal filters to optimally attenuate SAXS signals. Is the aim of the filters to attenuate SAXS signal or to attenuate the direct beam while keeping the best SAXS signal or both?

Line 110 and 115. it is written that the plates are attached to a linear motorized positioning stage X-Z. Are these directions coherent with the directions shown in Figure 1 a? The reference system of Figure 2b could be also added to Figure 1a.

Line 114. what is the lateral dimension of the triangles? Why is it written that the thicknesses are different when afterwards it

is stated that they all are 100 μm thick?

Line 136: Having access to the intensity at $q = 0$ has many advantages and would be extremely beneficial for many reasons, beyond HI plasma. Could the authors briefly comment on how the analysis of SAXS would be performed at infinitesimal small q , since there is still the deconvolution required of the direct, non-scattered beam and the scattered beam at extremely small q ?

Line 164 – 165: $q = 165.59 \times 10^3 \text{ (m}^{-1}\text{)}$, and $d = 1039 \text{ nm}$. Which peak/feature was taken to evaluate it? And how does the result compare to the micrograph shown of the Si-rods?

Line 180: the plasma scale length is described in supplementary section 3, not in supplementary section 4.

Line 476 of methods: the sentence about the Kapton window is not clear, as it is not clear for the reader neither the scheme of FEL setup or of the chambers. Is the detector outside the interaction chamber? Anyway, as it is not fundamental for the proposed experiment, if the description of the system becomes too lengthy, the sentence about Kapton could also be removed.

Caption of figure 3. The integration refers to supplementary section 3 and not to section 2

Supplementary Information.

Section 1. Line 70-71: if the dimension of the target is 22mm x 1.4mm x 0.5mm, it is not cubic.

From supplementary Fig 1 (b) and from the fabrication process description it seems that Cr is still staying on the top of the rods. Is it true, or was Cr removed?

Section 2

Line 98: it is Figure 1b instead of Figure 1c .

Line 101: it is Figure 1c instead of Figure 2b.

Section 3

Supplementary Figure 3. Just a suggestion: It would be useful to put the delay times just on the figure itself (maybe beside the graphs cd, gh and kl respectively).

Section 4

As the reference system and the direction of the X-ray beam with respect to the sample is different from the one of Figure 2, it would be nice to put it in supplementary Figure 4 together with the direction of the X-ray beam.

In equation (14) N should be defined.

In line 201 and 202 the description of the X-ray beam position could be still misunderstood. It could be added that the beam is perpendicular to the rod height and parallel to the substrate. Or a scheme could be added to supplementary Figure 7 with the reference system and the direction of the X-ray beam with respect to the rods assembly.

Caption of Supplementary Figure 9 a: it is the density profile of an ionized rod in real space, not of a grating.

Reviewer #2

(Remarks to the Author)

I co-reviewed this manuscript with one of the reviewers who provided the listed reports. This is part of the Communications Physics initiative to facilitate training in peer review and to provide appropriate recognition for Early Career Researchers who co-review manuscripts.

Reviewer #3

(Remarks to the Author)

This paper uses a new technique based on Free-electron lasers that make it possible to use in-situ Small-Angle X-ray Scattering (SAXS) to study laser-produced high-energy-density plasmas at solid density with nanometer and femtosecond resolution. However conventional SAXS misses important small-angle signals near the direct beam because of the beamstop. This work demonstrates a beamstop-free SAXS system using layered metallic filters, allowing continuous measurement of both small- and wide-angle scattering with a large dynamic range, including the critical region around the direct X-ray beam. Applied to submicron silicon-rod plasma arrays, the method revealed multiscale rarefaction dynamics and showed that the initial plasma scale length strongly influences laser absorption, opening new possibilities for studying diffuse plasma phenomena such as filamentation and turbulence.

The work of Ota et al. is thorough and complete, the figures and text are well connected. The beamstop free technique is interesting and yields valuable information. The paper is worthy of publication in this Nature journal without change.

Version 1:

Reviewer comments:

Reviewer #1

(Remarks to the Author)

The authors have optimally addressed the issues raised in the first review report. The manuscript is sound, and explanations and data are clearly expressed. The publication on Communication Physics is therefore strongly recommended.

Reviewer #2

(Remarks to the Author)

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Dear Dr. Saleem Denholme,

We sincerely appreciate the reviewers' valuable comments and suggestions, which have helped us improve the manuscript. We have revised the manuscript accordingly. Our responses to all reviewers' comments are provided below. The corresponding changes in the revised manuscript are highlighted in yellow in accordance with the revision guidelines of *Nature Portfolio*, and the corresponding changes in the revised manuscript are highlighted in yellow.

Reply to Reviewer #1 (from here R1):

R1 (1-1)

The manuscript of M. Ota et al. reports on the use of a layered system of metallic filters to attenuate the direct X-ray beam of an X-FEL source in order to perform SAXS without a beamstop. The system was successfully employed for the study of a 2D array of columnar plasma obtained by irradiating an assembly of silicon rods with a high intensity laser pulse. A wide angular range, including the area which is usually masked by the beamstop, allowed a better study of the plasma scale length and of its initial evolution with respect to the SAXS measurement in the presence of the beamstop.

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Authors (1-1)

We appreciate R1's positive comment. We will reply to the recommendations you mentioned above in the following text.

R1 (1-2)

- *Formatting (introduction, results, discussion) could improve readability*

Authors (1-2)

We have revised the Introduction to improve the logical flow. The readability of the Results and Discussion sections has also been improved by addressing R1's comments, as described below.

(Revised) Current main text, line 92:

In this Article, we develop a beamstop-free SAXS technique that provides continuous angular coverage from the direct beam (0th order) to higher-order scattering in a single exposure. The technique employs layered triangle-shaped metal filters to spatially tailor the attenuation of the scattering intensity, substantially extending the effective dynamic range of a conventional X-ray CCD camera while

preserving SAXS signals near the direct beam. This capability allows multiscale spatial dynamics to be investigated in a single shot. We apply the beamstop-free SAXS technique to investigate the ultrafast evolution of a two-dimensional array of submicron columnar plasmas generated by irradiating a high-intensity (HI) laser pulse onto a silicon (Si) rod assembly. To ensure a well-defined initial plasma state, we used a rigorously designed quasi-two-dimensional assembly of submicron silicon rods with a lattice symmetry, sharp edges, and constant parameters (e.g., diameters, spacings, and heights). Notably, high-aspect-ratio rods are utilized to study the penetration of the laser field inside the gap between them. The beamstop-free SAXS technique enabled continuous measurements of the temporal evolution of the nanometer-scale plasma dynamics on the surface of rods in the radial direction over several tens of picoseconds by varying the timing between the HI laser (pump) and the XFEL (probe). The beamstop-free SAXS measurements reveal that the microscale initial state of the preplasma generated by the HI laser pre-pulse strongly influences the absorption efficiency of the main pulse, thereby governing the subsequent macroscale plasma evolution.

R1 (1-3)

• *The authors could comment in the introduction part, whether similar approaches for beamstop-free SAXS exist.*

Authors (1-3)

Recent advances in SAXS instrumentation have followed three representative directions: high-dynamic-range detectors, beam attenuation systems used in conjunction with conventional beamstops, and semi-transparent beamstops. High-dynamic-range detectors extend the measurable intensity range of the detector, while beam attenuation systems reduce the intensity of the transmitted beam to avoid detector saturation. Semi-transparent beamstops further allow partial transmission of the direct beam, enabling simultaneous monitoring of the transmitted beam and scattering signals close to the beam center. However, these approaches still rely on a beamstop or are primarily intended for transmitted-beam diagnostics rather than eliminating the missing angular region in SAXS measurements. Consequently, a practical beamstop-free SAXS technique capable of simultaneously acquiring scattering signals over the full scattering-angle range in a single exposure has yet to be demonstrated. We have added the explanation in the main text.

(Revised) Current main text, line 84:

, with three different gain stages and 75 μm pixel pitch. It is widely used at the

R1 (1-4)

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• More details on the filter system could be given (in the main text or in the supplementary information). Why was a triangular shape used? Why two sets of filters with three triangles? Why the two set of triangles have opposite directions? Which was the dimension of the triangles? Why are $L1$ and $L2$ combined?

Authors (1-4)

A triangular shape was adopted because the width of the attenuation region for the one-dimensional SAXS profile can be optimized by vertically shifting the triangular filter along the direction in which the SAXS signal extends. In contrast, if a rectangular filter were used, its width would have to be precisely machined in advance to achieve the desired attenuation.

In principle, increasing the number of filters extends the achievable dynamic range. Therefore, the use of additional filters can further overcome the dynamic-range limitation of the detector. Ideally, each filter should be mounted on an independent motorized positioning stage so that its position can be adjusted individually. Filter System 2 has three windows: one without a filter, one containing two triangular filters, and one containing four triangular filters. In our experimental setup, including the target and the X-ray CCD camera, the filter system enables the SAXS signal to be distributed over a sufficiently large active area of the detector while remaining within the camera's dynamic range. Therefore, only three filters were required. Two of these three filters (L_1 and L_2) were combined because only two motorized linear X-Z positioning stages were available in front of the X-ray CCD camera; consequently, one stage was assigned to Filter System 1 and the other to Filter System 2. The geometry of the filters mounted together on Filter System 2 was designed based on the calculated SAXS intensity distribution.

The two set of triangles have opposite directions because the intensity gradient of the critical low- q SAXS peaks near the center is steep, resulting in a very narrow attenuation region. By utilizing the apex of a triangular filter, the attenuation width can be adjusted with high spatial resolution without requiring high machining precision.

A detailed description of the filter system has been added to the Methods section.

(Revised) Current main text, lines 123 and 335:

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$L1$ and $L2$) were mounted together on Filter System 2 because only two motorized linear X-Z positioning stages were available. The geometry of the combined filters

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• Last paragraph ◊ here the results that motivate the selected approach to study plasma using SAXS without beamstop are highlighted. A sentence about the research outcome in the abstract and in the introduction would greatly help in connecting the parts directly from the beginning.

Authors (1-5)

We have added the following sentences to the Abstract and the Introduction:

“stochastic plasma dynamics under high-energy-density conditions”

“The beamstop-free SAXS measurements reveal that the microscale initial state of the preplasma generated by the HI laser pre-pulse strongly influences the absorption efficiency of the main pulse, thereby governing the subsequent macroscale plasma evolution.”

(Revised) Current main text, lines 57, 96 and 109:

R1 (1-6)

Line 49: with wide-range scattering signal is it intended a wide q-range/angular scale?

Authors (1-6)

Yes. R1’s understanding is correct. We have revised the term as suggested by R1.

(Revised) Current main text, line 48:

R1 (1-7)

Line 71: a connection sentence could be added to introduce early on that the Si-rods were used in the later experiment. Or the sentence at line 93 could be moved here.

Authors (1-7)

We meant rod assembly by the use of “wires” in line 72. To prevent the confusion, we revised it as rod assembly.

(Revised) Current main text, line 71:

.},{“family”:“Barberio”,“given”:“M.”},{“family”:“Scisciò”,“given”:“M.”},{“family”:“Humières”,“given”:“E.”,“non-dropping-particle”:“d”},{“family”:“Antici”,“given”:“P.”}],“issued”:{“date-ev. Lett.”,“language”:“en”,“license”:“http://link.aps.org/licenses/aps-default-license”,“page”:“145001”,“source”:“DOI.org (Crossref)”,“title”:“Near-Complete

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d's largest lasers. It also generates gigantic quasi-static electromagnetic fields that

R1 (1-8)

Line 84: a more specific one-sentence explanation of what specifically the beamstop was limiting could be useful. Otherwise, the sentences from line 100 to 104 could be moved here to really strengthen the force of the present research compared to the previous one.

Authors (1-8)

We have added a few sentences at the end of the paragraph to stress the importance of the beamstop-free SAXS technique.

(Revised) Current main text, line 89:

R1 (1-9)

Line 97: it is stated that Utilizing layered triangle-shaped metal filters to optimally attenuate SAXS signals. Is the aim of the filters to attenuate SAXS signal or to attenuate the direct beam while keeping the best SAXS signal or both?

Authors (1-9)

We meant that the attenuation should be optimized for SAXS peaks, especially continuously connect the signals from 0th order to higher order. However, SAXS peaks around the direct X-ray beam, such as the first- and second-order peaks, have almost the same intensity as the direct beam; therefore, both of them cannot be separated. Therefore, optimizing SAXS peaks also optimize the original beam profile. We have added an explanation to clarify the purpose of using the layered triangular-shaped filters, as described in Author Response (1-8), and have also included this explanation at the beginning of the next paragraph.

(Revised) Current main text, line 92:

R1 (1-10)

Line 110 and 115. it is written that the plates are attached to a linear motorized positioning stage X-Z. Are these directions coherent with the directions shown in Figure 1 a? The reference system of Figure 2b could be also added to Figure 1a.

Authors (1-10)

We have revised Fig. 1a and added the reference system.

(Revised) Fig.1a in the main text.

R1 (1-11)

Line 114. what is the lateral dimension of the triangles?

Why is it written that the thicknesses are different when afterwards it is stated that they all are 100 μm thick?

Authors (1-11)

We have added a scale bar indicating the spatial dimensions on the X-ray CCD camera in Fig. 1b. Therefore, the lateral dimensions of the triangular filters can now be inferred.

We prepared two filter frames, each containing several windows with different aluminum filter thicknesses. For example, Filter System 1 consists of five windows: one without a filter and four with 100, 200, 300, and 400 μm thick Al filters, as shown in Fig. 1a. The optimal filter thickness depends on the SAXS intensity distribution, which varies according to the sample morphology and scattering profile. For samples exhibiting a steep SAXS intensity decay, a thicker Al filter is required to sufficiently attenuate the low- q scattering signals. Therefore, the optimum combination of triangle-shaped filters with different thicknesses should be selected depending on the experiment. In the present experiment, however, the selected triangle-shaped filters happened to have the same thickness (i.e., 100 μm). We have added the detailed explanation in Methods (line 335).

R1 (1-12)

Line 136: Having access to the intensity at $q = 0$ has many advantages and would be extremely beneficial for many reasons, beyond HI plasma. Could the authors briefly comment on how the analysis of SAXS would be performed at infinitesimal small q , since there is still the deconvolution required of the direct, non-scattered beam and the scattered beam at extremely small q ?

Authors (1-12)

We have added the explanation.

(Revised) Current main text, line 150:

R1 (1-13)

Line 164 – 165: $q = 165.9 \times 10^6 \text{ m}^{-1}$, and $d = 1039 \text{ nm}$. Which peak/feature was taken to evaluate it? And how does the result compare to the micrograph shown of the Si-rods?

Authors (1-13)

$q_0 = 5.9 \times 10^6$ was obtained by positions of the SAXS peaks in the preshot of Fig. 3c.

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(Revised) Current main text, lines 179:

R1 (1-14)

Line 180: the plasma scale length is described in supplementary section 3, not in supplementary section 4.

Authors (1-14)

We meant that readers should refer to Supplementary Fig. 5 in supplementary note 4 for the definition of the scale length. To clarify this, we added the figure number.

(Revised) Current main text, line 200:

R1 (1-15)

Line 476 of methods: the sentence about the Kapton window is not clear, as it is not clear for the reader neither the scheme of FEL setup or of the chambers. Is the detector outside the interaction chamber? Anyway, as it is not fundamental for the proposed experiment, if the description of the system becomes too lengthy, the sentence about Kapton could also be removed.

Caption of figure 3. The integration refers to supplementary section 3 and not to section 2

Authors (1-15)

Yes. The X-ray CCD camera was outside the interaction chamber. Because the description of the system becomes too lengthy, we removed the explanation of the Kapton window to avoid confusion. The R1's comment that the integration refers to supplementary section 3 and not to section 2 is correct, and we revised it.

R1 (1-16)

Supplementary Information.

Section 1. Line 70-71: if the dimension of the target is 22mm x 1.4mm x 0.5mm, it is not cubic.

From supplementary Fig 1 (b) and from the fabrication process description it seems that Cr is still staying on the top of the rods. Is it true, or was Cr removed?

Authors (1-16)

We revised the word as cuboid.

(Revised) Current supplementary text, line 71:

-irradiation experiments, patterned regions were located near a corner of a 22,000 $\mu\text{m} \times 1,400 \mu\text{m} \times 500 \mu\text{m}$ cuboid target.

R1 (1-17)

Section 2

Line 98: it is Figure 1b instead of Figure 1c .

Line 101: it is Figure 1c instead of Figure 2b.

Authors (1-17)

To avoid the confusion in line 98 we revised the expression.

We revised “Figure 2b” in line 101 as “Figure 1c”.

(Revised) Current supplementary text, line 98:

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R1 (1-18)

Section 3

Supplementary Figure 3. Just a suggestion: It would be useful to put the delay times just on the figure itself (maybe beside the graphs cd, gh and kl respectively).

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Authors (1-18)

We added the delay time on the left side of Supplementary Fig. 3.

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(Revised) Supplementary Fig. 3.

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R1 (1-19)

Section 4

As the reference system and the direction of the X-ray beam with respect to the sample is different from the one of Figure 2, it would be nice to put it in supplementary Figure 4 together with the direction of the X-ray beam.

In equation (14) N should be defined.

In line 201 and 202 the description of the X-ray beam position could be still misunderstood. It could be added that the beam is perpendicular to the rod height and parallel to the substrate. Or a scheme could be added to supplementary Figure 7 with the reference system and the direction of the X-ray beam with respect to the rods assembly.

Caption of Supplementary Figure 9 a: it is the density profile of an ionized rod in real space, not of a grating.

Authors (1-19)

To clarify the direction the X-ray beam with respect to the grating, we put an arrow in Supplementary Fig. 4b.

N is the number of groove, and we added the explanation.

To clarify the direction of the X-ray beam with respect to the grating, we put an arrow in Supplementary Fig. 7 b and add the explanation “parallel to the substrate”. R1's comment that it should be the density profile of an ionized rod in real space rather than of a grating is correct, and we have revised the manuscript accordingly.

(Revised) Current supplementary text, lines 194, 201 and the caption of Supplementary Fig. 9:

$\sigma r = 0 - 300 \text{ nm}$).

Reply to Reviewer #2 (from here R2):

R2 (2-1)

I co-reviewed this manuscript with one of the reviewers who provided the listed reports. This is part of the Communications Physics initiative to facilitate training in peer review and to provide appropriate recognition for Early Career Researchers who co-review manuscripts.

Authors (2-1)

We thank the reviewer for this information and appreciate the time and effort devoted to the review of our manuscript.

Reply to Reviewer #3 (from here R3):

R3 (3-1)

This paper uses a new technique based on Free-electron lasers that make it possible to use in-situ Small-Angle X-ray Scattering (SAXS) to study laser-produced high-energy-density plasmas at solid density with nanometer and femtosecond resolution. However conventional SAXS misses important small-angle signals near the direct beam because of the beamstop. This work demonstrates a beamstop-free SAXS system using layered metallic filters, allowing continuous measurement of both small- and wide-angle scattering with a large dynamic range, including the critical region around the direct X-ray beam. Applied to submicron silicon-rod plasma arrays, the method revealed multiscale rarefaction dynamics and showed that the initial plasma scale length strongly influences laser absorption, opening new possibilities for studying diffuse plasma phenomena such as filamentation and turbulence.

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Authors (3-1)

We sincerely thank R3 for the positive evaluation of our work and for recommending the manuscript for publication. We greatly appreciate the reviewer's encouraging comments.

Review report

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