



## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



**Experiment title:** In situ formation of silver and gold nanoparticles in silicate glass under excimer laser irradiation.

**Experiment number:**  
CH- 5046

<b>Beamline:</b> BM26A	<b>Date of experiment:</b> from: 07.07.2017 to: 11.07.2017	<b>Date of report:</b> 28.02.2018  <i>Received at ESRF:</i>
<b>Shifts:</b> 12	<b>Local contact(s):</b> Alessandro Longo (email: brunelli@esrf.fr)	

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## Report:

Due to the technical difficulties the experiment with laser was not possible. To solve this issue, we have prepared an extensive list of gold, silver, and silver-gold samples irradiated by different number of laser pulses. Each sample represented a single slide of soda-lime glass after Ag-Na ion exchange procedure and/or coating by 70 nm gold layer. The samples were then exposed to the laser radiation with spot size  $\sim 5 \times 5$  nm. This procedure allowed to have several different spots at one slide (figure 1). The upper part of each slide has been cleaned with acetone to remove the non-implant part of the nanoparticles from the surface of the glass. The fluence of the laser radiation was set to  $140 \text{ mJ/cm}^2$ . However, the fluctuation of the actual fluence may occur, that is why we also measured directly the fluence of the first laser pulse (which is of critical importance for the subsequent particle formation process) to be exactly  $140 \text{ mJ/cm}^2$ .

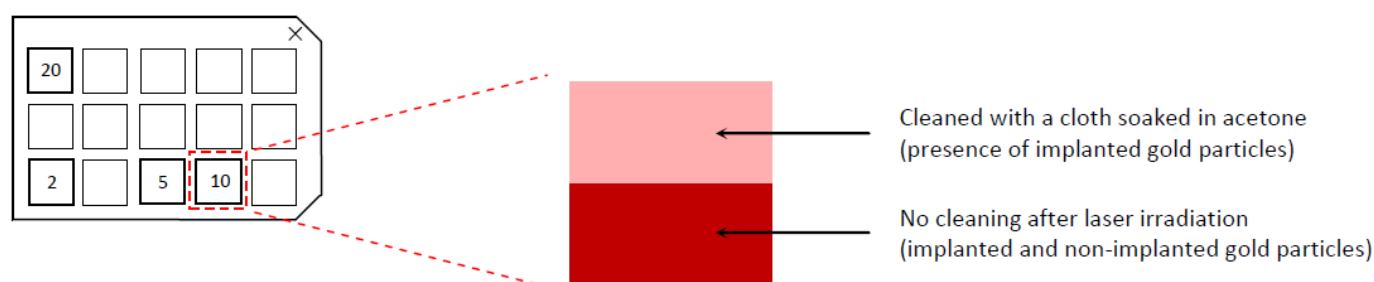


Figure 1. Schematic picture of the sample, contacting several spots irradiated by laser pulses (numbers in the picture correspond to the number of laser pulses).

The samples were fixed with plasticine on the metal holder. SAXS data were collected in the geometry when the sample was oriented perpendicularly to the incoming beam. The photon wavelength for SAXS data was set to 0.486098, 0.486499, 0.487417, 0.490056, and 0.493961 Å. Due to the low fraction of Ag in the samples, each sample was then rotated by 45° to collect EXAFS spectra in the fluorescence mode.

The integrated SAXS patterns are shown in the figures 1 – 4.

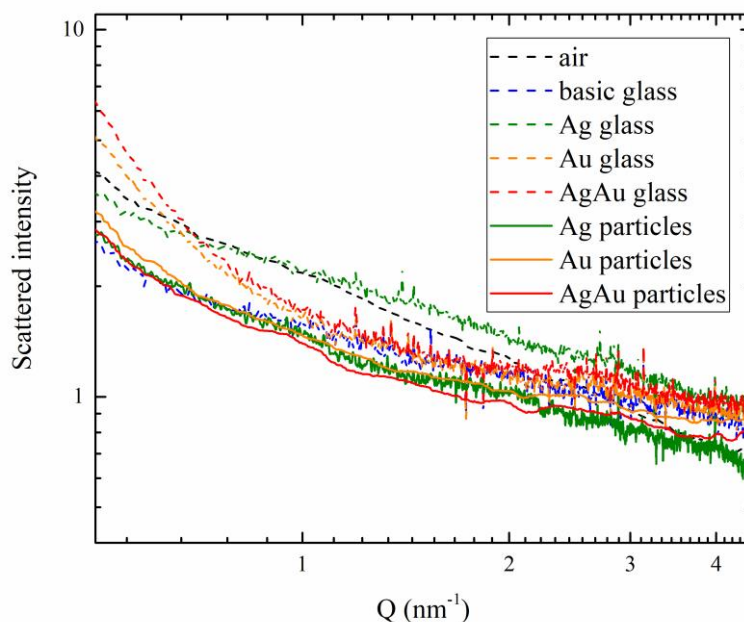


Figure 1. An overview of the obtained SAXS patterns for initial (non radiated) and final (after 1000 pulses) states of Ag, Au and mixed AgAu samples.

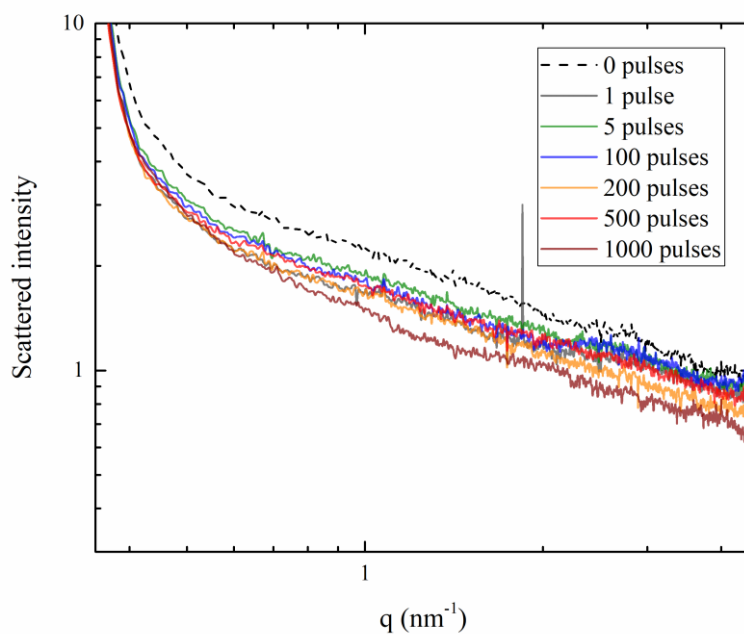


Figure 2. Evolution of SAXS pattern for pure Ag samples depending on the number of laser pulses.

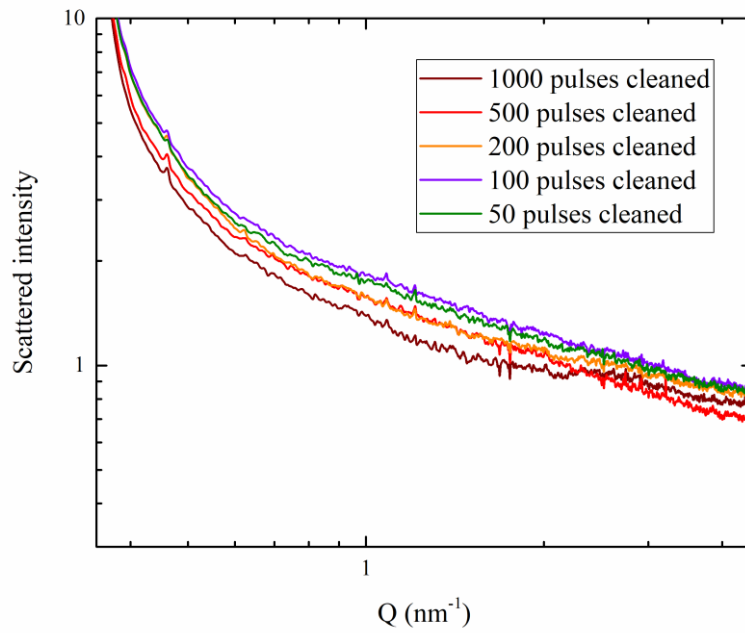


Figure 3. Evolution of SAXS pattern for mixed AgAu samples depending on the number of laser pulses.

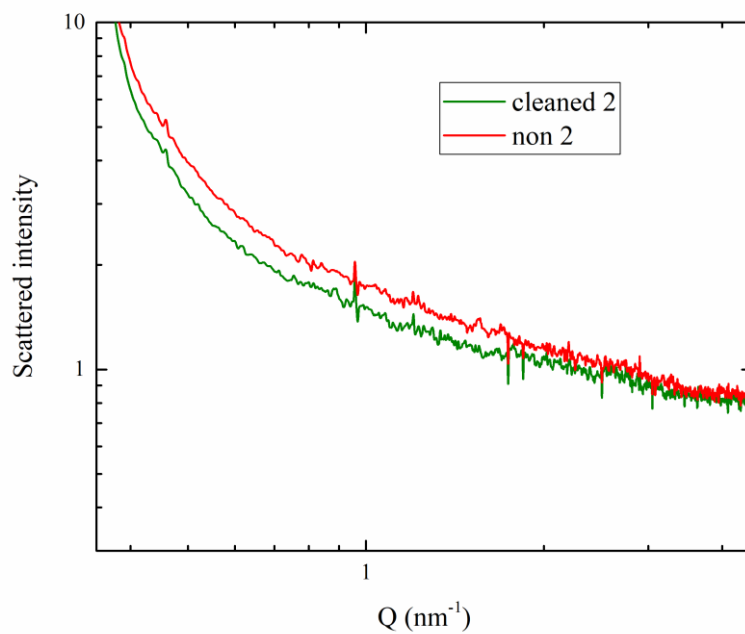


Figure 4. The effect of the non-implanted pure Au particles on SAXS patterns.

At the same time EXAFS analysis indicated a small decrease of Ag-O contribution which is explained by the reduction of Au to form pure Ag and mixed AgAu particles (Figure 5). Further analysis is in progress together with the Au  $L_3$  data obtained on BM23.

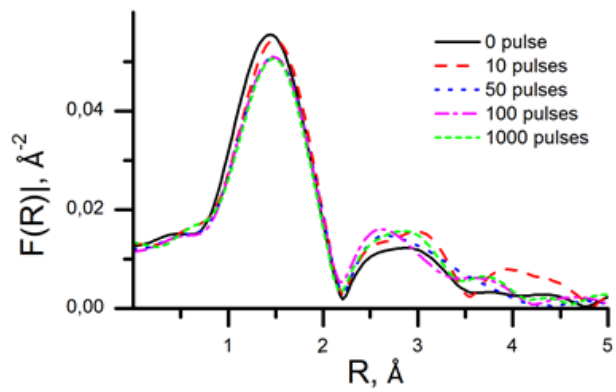


Figure 5. Reduction of Ag-O contribution with formation of small Ag particles.