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DESIGN OF LIQUID INJECTION DEVICE FOR THE HARD X-RAY ULTRAFAST SPECTROSCOPY EXPERIMENT STATION*

Linghao Li, Bing Li, Xing Liu, Jianwei Meng, Ruixue Zhu, Kaiyu Zhang, Tsu-Chien Weng†
Center for Transformative Science, ShanghaiTech University, Shanghai, China

Abstract

The Hard X-ray Ultrafast Spectroscopy Experiment Station (HXS) of the Shanghai high repetition rate XFEL and extreme light facility (SHINE) requires the design and manufacture of a specialized liquid sample injection device when studying the liquid phase state of matter. Due to the damage caused by high-repetition-rate XFEL pulses on the sample, it is necessary to ensure that the liquid sample is refreshed before the next pulse arrives. In order to reduce the impact of liquid film thickness on pump-probe ultrafast spectroscopy experiments, it is required that the liquid film thickness be less than 20 μm. This article describes the use of oblique collision of two jets, from simulation calculation to the construction of experimental device, and the use of absorption spectroscopy principle to construct a thickness characterization system. This system can stably produce ultrathin liquid films with thickness ranging from 3-20 μm. The article proposes views on the limitations and future improvements of this device.

INTRODUCTION

The Shanghai high repetition rate XFEL and Extreme light facility (SHINE) is equipped with a high-quality electron beam continuous wave superconducting linear accelerator with an energy of 8 GeV. The energy wavelength coverage of this device is 0.4-25 keV, and the pulse repetition rate can reach up to 1 MHz. The device has the characteristics of high brightness, short pulse, high repetition rate, and high coherence [1]. The main experimental platform of the Hard X-ray Ultrafast Spectroscopy Experiment Station (HXS) located in FEL-III is the high-energy resolution X-ray photo-in-photo-out (PIPO) spectrometer, which can achieve femtosecond time resolution by combining pump-probe technology. The reactions involved in the liquid phase state of matter are currently an important research area in the fields of chemistry and biology [2], and are also an important research direction of HXS. Therefore, it is necessary to build a liquid sample injection device that meets the requirements of the experiment station.

After in-depth analysis of the characteristics of X-ray free-electron lasers and samples, we propose the following requirements for the in-situ environment of liquid samples: Firstly, due to the high repetition rate and radiation damage characteristics of X-ray free-electron lasers, sample replacement is necessary. Therefore, we need to establish a system that can continuously deliver samples to ensure that the pulse of the X-ray free-electron laser is not wasted. Secondly, in order to control the impact of liquid film thickness on the pump-probe time resolution within 66 fs, the liquid film thickness must be less than 20 μm. At the same time, the outline of the liquid sample should be much larger than the light spot of the X-ray beam to ensure that the detector receives the signal after passing through the liquid sample.

This article designs and implements a super-thin liquid film generation device, and verifies the stability and thickness of the generated liquid film through the construction of a test optical path, which meets the experimental requirements. This research provides an important experimental foundation for subsequent research in related fields.

EXPERIMENTAL METHODS

Liquid Film Generation Device

In recent years, the principles of generating flowing liquid films suitable for X-ray spectroscopy research mainly include the following three types:

Slit jetting [3]. This method involves spraying liquid through a slit to overcome the surface tension of the liquid and form a liquid film. However, this method is limited by the size of the tube wall, and the production of microfluidic tubes with dimensions of a few microns can easily encounter problems such as clogging during use.

Liquid flow collision [4-6]. This method utilizes two liquid flows that collide with each other to form a liquid film through interaction, and has high stability. This method has broad application prospects in pump-probe ultrafast spectroscopy experiments.

Gas focusing [7-9]. This approach is similar to gas-dynamic focusing virtual nozzle (GDVN), which requires gas pressure to change the cross-sectional shape or size of the liquid flow, typically serving the needs of lower dimensions. This approach is not further discussed in this article.

Based on the principle of liquid flow collision, this study built an experimental platform as shown in Figure 1. Using an HPLC pump to provide power for liquid transport and control the liquid flow rate, a liquid pipeline was constructed at the output end of the pump, using PEEK tubes, liquid-phase connectors, T-shaped tees, stainless-steel tubes, and other parts.

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† wengzq@shanghaitech.edu.cn.

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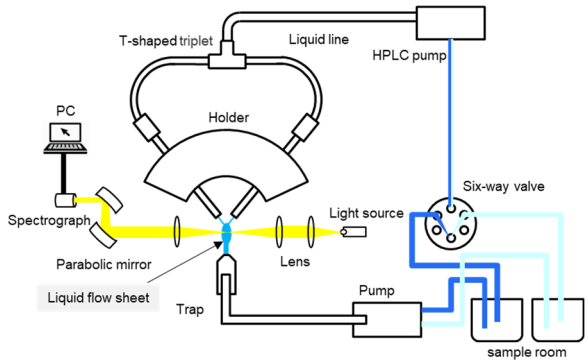

Figure 1: Liquid film generation device and characterization system.

Liquid Film Characterization System

For the generated liquid film, its size and thickness need to be tested to ensure compliance with the requirements of the experimental station. A 3×10^{-5} mol/L methylene blue solution [10] was selected as the sample liquid for the experiment. A colorimetric cuvette was used for the molar absorptivity test. It was found that the visible light absorption peak of the methylene blue solution at this concentration was at 664 nm, with a molar absorptivity of 6.78×10^{4} L/(mol·cm).

The thickness of the liquid film is characterized by reference to the Lambert-Beer law, which calculates the film thickness based on the intensity absorption of light by the liquid film. Using HL-2000 as the light source, the light beam is emitted from a fiber with an inner diameter of 600 μm, and a monochromatic light source with a desired wavelength band is obtained through an optical filter. Since the light source is a point light source, it needs to be focused and aligned, and an experimental optical path as shown in the figure is constructed. After the converged light is received by the fiber probe, a PG-2000 fiber spectrometer and its supporting spectral testing software Morpho are used for spectral analysis.

RESULTS

Flow Rate Testing

In the experiment, due to the influence of factors such as the sealing performance of the liquid pipeline and the viscous resistance of the pipe wall, there is a difference between the actual flow rate of the liquid emitted from the stainless-steel pipe and the flow rate set by the pump [11]. Therefore, before measuring the thickness of the liquid film, it is necessary to test the actual flow rate of the jet. The test results are shown in Figure 2. The higher flow rate set by the pump is denoted as A, and the lower flow rate is denoted as B. The liquid flow rate for subsequent thickness characterization experiments is also tested and calculated according to these two gears.

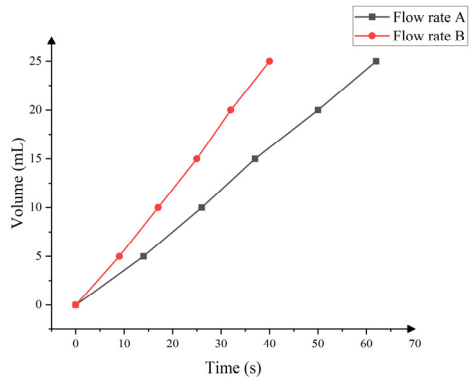

Figure 2: Flow rate test.

It can be seen that the actual volumetric flow rate of the liquid at flow rate A is 0.406 mL/s, and the volumetric flow rate at flow rate B is 0.629 mL/s. The inner diameter of the pipeline is 125 μm, which is converted to a length flow rate of 33.084 mm/s and 51.256 mm/s, respectively.

Calculation of Liquid Film Thickness

In order to have a estimate of the size and thickness of the liquid film, the liquid film generated by the collision of two jets can be simulated and calculated based on the Hasson-peck formula [12, 13]:

$$\frac{hr}{R^2} = \frac{sin^3\theta}{(1-\cos\phi\cos\theta)^2} \quad (1)$$

$$\frac{r_e}{R\,We} = \frac{sin^3\theta sin^2\psi}{4(1-\cos\phi\cos\theta)^2} \quad (2)$$

Equation (1) is the thickness distribution formula, and equation (2) is the liquid film shape formula. According to these two equations, the thickness of the liquid film is mainly affected by the collision angle and the pipe diameter, while the jet velocity will affect the size of the liquid film. Figures 3 and 4 show the radial variation of the thickness of the liquid film formed under different collision angles and different pipe diameters under limited conditions. By summarizing the results of the simulation calculations, this experiment finally chose to use 125 μm stainless-steel pipe for the collision experiment. At the same time, in order to simplify the calculation and statistics, the collision angle of the two jets was designed to be 90° and 120°.

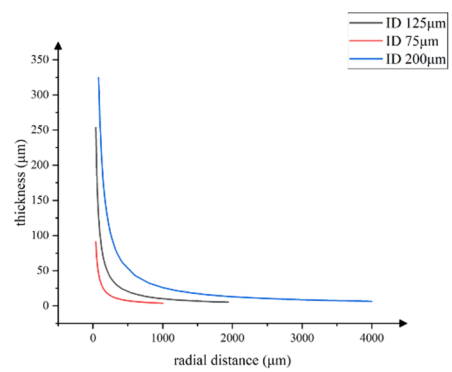

Figure 3: Simulation curve of liquid film thickness: jet diameter factor.

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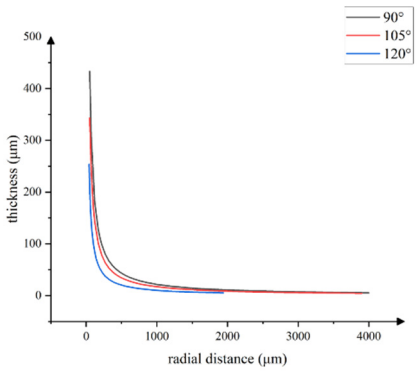

Figure 4: Simulation curve of liquid film thickness: collision angle factor.

Characterization of Liquid Film Thickness

The ultra-thin liquid film generation device can eventually form a liquid film as shown in Figure 5. We conducted quantitative measurements of the liquid film size, and the results showed that the width of the liquid film was between 1-2 mm, with a maximum length of 3-6 mm, which was much larger than the size of the focused spot, meeting the requirements for conducting the experiment. In addition, within this range, as the flow rate increases, the size of the liquid film also increases, while as the collision angle increases, the size of the liquid film decreases.

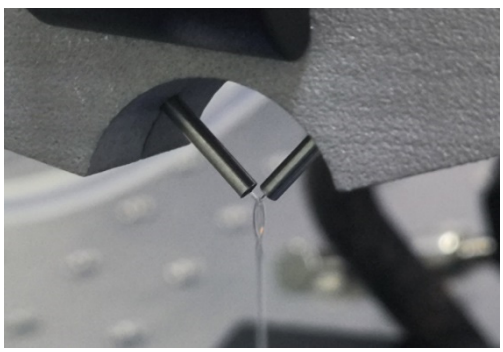

Figure 5: Schematic diagram of liquid film.

The focused light passes through the liquid film and is received by the spectrometer's fiber probe. By comparing the intensity changes at the 664 nm absorption peak of the methylene blue solution before and after passing through the liquid film, the absorption data is converted into thickness data according to the Lambert-Beer law, and the final result is shown in Figure 6. From the figure, it can be seen that although the size of the liquid film decreases after increasing the angle, the thickness of the liquid film can be reduced to a certain extent. The reason is speculated to be that the increase in collision angle increases the radial momentum, making it easier for the liquid to overcome surface tension and become a flat liquid film. The increase in liquid flow rate increases the thickness of the liquid film, and the range of measured data also increases. The reason is speculated to be that after the collision of higher flow rate liquid, the stability of the liquid film itself is impacted to some extent, resulting in large changes in measured data. However, in the experiment with a collision angle of 120°, the liquid film thickness meets the requirements of the experimental station.

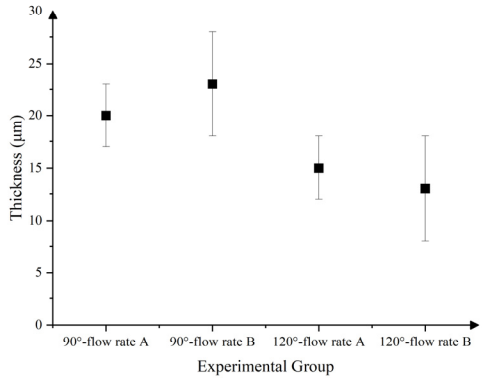

Figure 6: Liquid film thickness data chart.

SUMMARY

The article studies a liquid sample injection device applied to the hard X-ray ultrafast spectroscopy experiment station of the Shanghai high repetition rate XFEL and extreme light facility. The device is based on the principle of liquid flow collision, which can form a liquid film that meets the experimental requirements, and is equipped with a thickness measurement device for the liquid film. By designing a specific optical path, the precise measurement of the liquid film is achieved. In the experiment, methylene blue solution is used as the sample solution to test the device. The results show that the size and thickness range of the liquid film are consistent with the theoretical calculation results, meeting the needs of the hard X-ray ultrafast spectroscopy experiment station for liquid sample testing.

At the same time, there are some areas for improvement in this device: Firstly, the mechanical fixing method used for all liquid pipelines and clamping devices has the problem that if the angle needs to be changed or the stainless-steel tube needs to be replaced, it needs to be disassembled and replaced with the corresponding components, and then reinstalled. The whole process is very cumbersome. The follow-up improvement plan is to automatically adjust the collision angle through motor control, and change the installation method of the stainless-steel tube to make it easier to disassemble and replace. Secondly, for the thickness detection device, currently the spectrometer can only measure the thickness of liquid samples with absorption peaks from visible light to near-infrared bands. If the testing range needs to be expanded, it is necessary to use a light source with a wider wavelength range and use equipments such as FTIR.

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