

# FOILED AGAIN: SOLID-STATE SAMPLE DELIVERY FOR HIGH REPETITION RATE XFELs

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## Abstract

XFELs today are capable of delivering high intensity pulse trains of x-rays with up-to MHz to sub-GHz frequency. These x-rays, when focused, can ablate a sample in a single shot, requiring the sample material to be replaced in time for the next shot. For some applications, especially serial crystallography, the sample may be renewed as a dilute solution in a high speed jet. Here, we describe the development and characterization of a system to deliver solid state sample material to an XFEL nanofocus. The first application of this system will be an x-ray laser oscillator operating at the copper  $K\alpha$  line with a  $\sim 30$  ns cavity.

## INTRODUCTION

Early x-ray free electron lasers, e.g. LCLS, permitted relatively slow replenishment of sample material between shots. But, as noted by [1], “supplying pristine material has not been an issue at first-generation XFELs running at up to 120 Hz, it becomes a question of the utmost importance at megahertz repetition-rate XFELs, where the jet speed needs to be much higher and extremely carefully controlled to ensure that pristine sample is supplied within time delays as short as 0.22–1  $\mu$ s between X-ray pulses”. For samples which can be delivered as a liquid solution, this problem may be addressed by high speed jets, but this approach is not without potential problems [2], e.g. limited viscosity. However, for solid-state samples, it was previously adequate to raster scan the samples using motion stages [3], but at MHz rep rates, this is not a practical approach. In this work, we discuss the requirements and design of a high speed, solid-state sample delivery system for use with high rep rate XFELs based on a spinning disk.

The first version of this sample delivery system is designed to enable an x-ray laser oscillator (XLO). An XLO, as envisioned by [4], is a means of using an x-ray free electron laser (XFEL) to pump gain medium in a Bragg crystal optical cavity to generate high power, fully coherent, transform-limited hard x-ray pulses with improved stability when compared to XFEL pulses. Presently, an effort to demonstrate the first XLO is underway [5]. This proof-of-concept XLO will operate at the copper  $K\alpha_1$  line, 8.05 keV, pumped by 9 keV XFEL pulses from LCLS. In this configuration, the LCLS will deliver a train of pump pulses, spaced at  $\sim 30$  ns, a timing equivalent to the round trip of the cavity. In each subsequent pass, the LCLS pulse will excite the copper gain medium

to population inversion, permitting amplification of the circulating ASE. A schematic overview of the experiment is provided in [4].

A crucial hurdle in this experiment is to ensure that fresh gain medium is present for each pulse since the ultra-high intensity x-ray pulses will transform any target material to a hot and rapidly expanding plasma. Early efforts focused on the development of high speed water jets, doped with copper nitrate [6]. At 200 m/s, the jet moves by 6  $\mu$ m every 30 ns, enough to replace the gain medium in time for the next pulse. This approach was originally preferred due to the option to achieve very high speeds and the intrinsically self-healing nature of the medium. However, solid targets are appealing since they offer a much higher number density of the target atomic species while having effectively no other elements present to absorb the pump pulse. In particular, it was experimentally determined that the manageable concentration of the copper nitrate is limited to 4 molar, which corresponds to a number density of about 2 copper atoms per cubic nanometer, while solid copper has a number density of 80 atoms per cubic nanometer. The latter fact reduces the pump pulse intensity requirements, therefore decreasing the heat load on the in-coupling crystal, while maintaining the same photon yield. In addition to the high speeds required, there are many additional constraints which influence the design of a solid-state target for an x-ray laser oscillator.

## SYSTEM REQUIREMENTS

The physics needs of the experiment will impose a number of requirements on the design of the sample and delivery system including:

- Sample speed – The target must deliver a fresh sample for each x-ray pulse. The required sample speed is therefore dictated by the XFEL repetition rate and the crater size left by the x-rays. For the XLO, the crater diameter left by the nanofocus in the copper foil is approximately 10  $\mu$ m, with pulses arriving every 30 ns. If the nanofocus spot is small compared to the crater, the target must advance at least (crater radius)/(pulse period) = 170 m/s. We note that the exact copper foil damage mechanism is a topic of active research in various groups around the world, so the final velocity requirements will be established during the experiment [7].
- Sample thickness and tolerance – The target’s thickness must be selected to optimally interact with the incoming x-ray pulse while not overly attenuating the signal. For

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the XLO, the optimum thickness is approximately 25  $\mu\text{m}$ . Variations in thickness of up to 10% result in acceptably small degradation of performance. Off-the-shelf copper foils had their thickness profiles measured using SLAC synchrotron facility – SSRL – and found to have thickness variations below the 5% level.

- Longitudinal offset – The target must not deviate excessively from the focal point of the x-rays. For the XLO, the nonlinear nature of the interaction puts stringent requirements on the displacement, limiting the allowable offset to approximately  $\pm 30 \mu\text{m}$ , comparable to the Rayleigh range of the x-rays at nanofocus (See Figure 1).

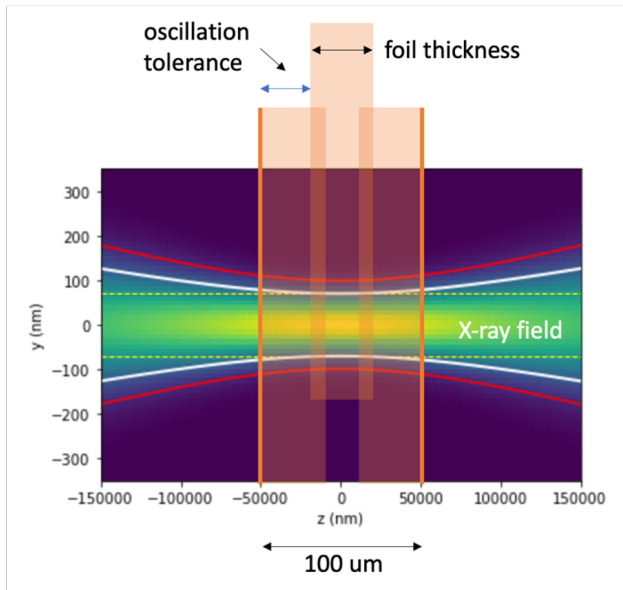


Figure 1: X-ray nanofocus and tolerable longitudinal oscillations.

- Sample lifetime – The target should supply sufficient material without requiring the sample disk to be replaced too frequently. It is assumed that the x-ray will hit a random angular position on the target so the probability of hitting unused sample material decreases with increased run time at a given radius. Depending how frequently it is tolerable for the x-ray pulse to miss the target, the target will be translated transversely by a crater radius (moving the x-ray focus to a new radius) to replenish the sample material. For the XLO, a 152 mm outer diameter foil disk with 13 mm of “rim” exposed, can provide a range of lifetimes, shown in Figure 2, depending on the acceptable frequency of bad shots.
- Helium tolerance – The XFEL x-rays are appreciably scattered by air so the sample should be in a helium environment. It is necessary that the sample delivery system can tolerate this. Helium has the added benefit of a much higher speed of sound (927 m/s) than air (344 m/s) which allows the sample to move faster without being transonic or supersonic, avoiding instabilities associated with these regimes.

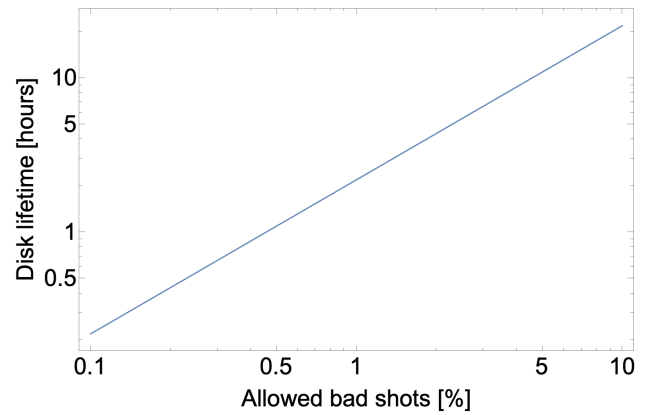


Figure 2: XLO sample lifetime as a function of allowable frequency of bad shots.

- Positional control – The target must be able to be positioned with a resolution that is small compared to the allowable longitudinal displacement from the focus and compared to the crater size, which dictates the transverse step. For the XLO, the required positioning resolution is on the order of single microns, which is readily achievable with off the shelf gear-driven and piezo-driven positioning systems. The requirements on stage speed are very low, as it only needs to translate by one crater radius whenever the current radius has been ablated to the bad shot limit ( $\ll 1 \text{ mm/s}$ ).

## EXPERIMENTAL RESULTS

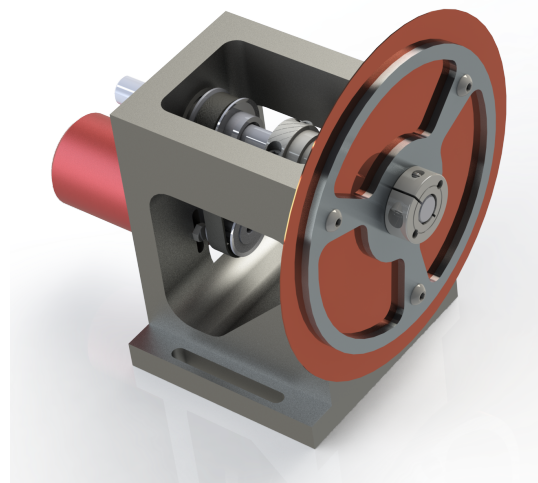


Figure 3: Prototype sample delivery system with unsupported foil rim.

The first version of the sample delivery system (See Figure 3) sandwiched the 25  $\mu\text{m}$  copper foil target between two retaining disks, which connected to a rotating shaft, supported by two ball bearings. The retaining disks were 127 mm in diameter, leaving a 13 mm rim of foil exposed.

This was spun by an off the shelf 2.3 kW brushless motor and speed controller, controlled by a PID feedback system reading the motor's integrated Hall effect sensors. The system demonstrated rotational speeds of more than 32,000 RPM.

The foil's longitudinal oscillations were measured using a microscope lens on a high speed camera, pointed azimuthally at the rim, Figure 4, and analyzed using image processing (A different illumination angle and tighter zoom than shown in that figure were used to improve spatial resolution). Although the oscillations generally were below the 30  $\mu\text{m}$  limit, the disks sometimes suffered from aeroelastic flutter, especially at higher speeds. No method was found to reliably prevent this instability, so the exposed rim approach was abandoned.

The original design used an unsupported rim to avoid occluding the sample. A new retaining disk design was created that extended beyond the foil, using spokes that block 8% of the foil. This was deemed an acceptable exchange for the improved reliability. The oscillations of the retaining disk, as measured by high speed camera, were again below the required 30  $\mu\text{m}$  limit but with the new geometry it was not possible to measure the foil itself. Future work will include the measurement of the foil oscillation using laser Doppler vibrometry. This design met all the requirements detailed in the previous section.

The prior implementations were low cost prototypes. The final target delivery system (Figure 5), currently under construction, has been redesigned to use air bearings (fed with helium) instead of ball bearings to improve precision, increase longevity, and reduce friction/wear.

A Nd:YAG laser has been used to approximate some of the behavior expected when the target is ablated by an x-ray laser pulse. The optical laser cannot be focused as tightly as the x-rays and the interaction is largely at the surface, rather than interacting with the whole volume, so it represents a worst-case perturbation of the foil. To estimate the impulse from the laser, it was focused onto an unsupported strip of the foil and recorded with the high speed camera. The laser punched craters through the thickness of the foil with a diameter of approximately 10  $\mu\text{m}$ , roughly consistent with the expected x-ray laser crater size [8]. This excited flexural and torsional modes, which were extracted and characterized using machine vision, that were used to calibrate a finite element analysis (FEA) simulation of the foil strip. This yielded an estimate for the laser impulse of approximately 500 nano-

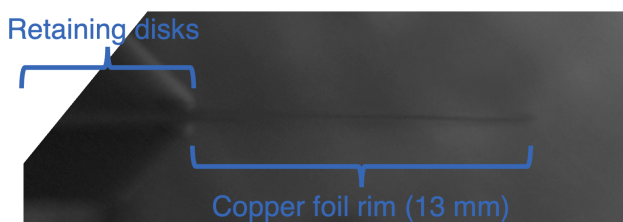


Figure 4: Microscope view from high speed camera of unsupported foil rim at 24,000 RPM in helium atmosphere.

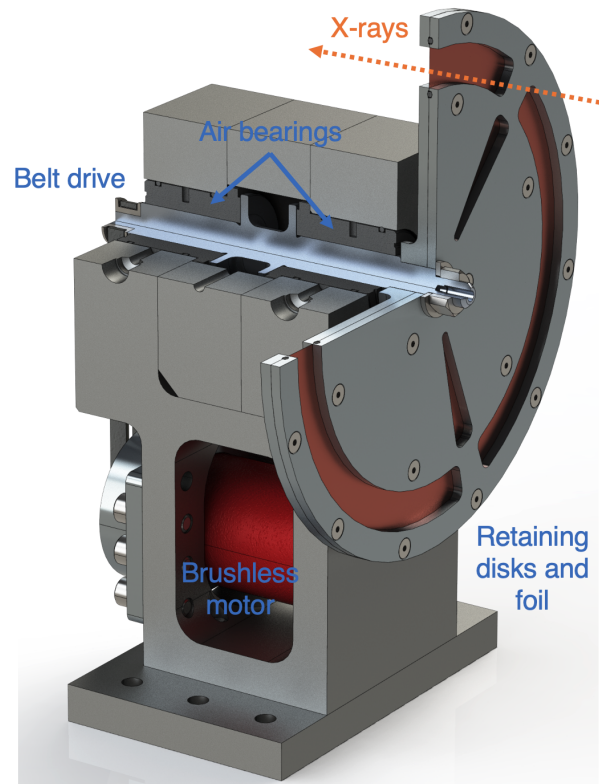


Figure 5: Final sample delivery system, designed with helium-fed air bearings.

Newton-seconds (roughly three orders of magnitude higher than the radiation pressure alone). This value was in turn used in an FEA simulation of the foil supported by the retaining disks, to understand the displacement expected due to the ablation. The simulation returned a worst case value of less than 10  $\mu\text{m}$ , which conforms to the requirements.

These efforts, including SEM imaging of the optical laser craters, are being used to benchmark magnetohydrodynamic simulations of the laser-target interaction, and are discussed in much greater detail in these proceedings in [8]. Future work will include shooting the mounted foil while spinning and measuring the oscillation using laser Doppler vibrometry. Taking many shots at the spinning target will reveal how much of the target can be ablated while remaining stable.

## DISCUSSION

Multiple iterations of a solid-state sample delivery system for a high repetition rate XFEL experiment have been developed and characterized. This system is capable of fulfilling the stringent requirements imposed by the physics needs of these experiments, including the need to very rapidly replace the sample between XFEL pulses, while maintaining excellent mechanical stability, even when subjected to perturbations arising from the laser-target interaction. This approach may be applied to future MHz XFEL experiments and could be adapted for use at synchrotron light sources, which also feature multi-MHz repetition rates.

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