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The laser micro-machining system for diamond anvil cell experiments and general precision machining applications at the High Pressure Collaborative Access Team

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We have designed and constructed a new system for micro-machining parts and sample assemblies used for diamond anvil cells and general user operations at the High Pressure Collaborative Access Team, sector 16 of the Advanced Photon Source. The new micro-machining system uses a pulsed laser of 400 ps pulse duration, ablating various materials without thermal melting, thus leaving a clean edge. With optics designed for a tight focus, the system can machine holes any size larger than 3 μm in diameter. Unlike a standard electrical discharge machining drill, the new laser system allows micro-machining of non-conductive materials such as: amorphous boron and silicon carbide gaskets, diamond, oxides, and other materials including organic materials such as polyimide films (i.e., *Kapton*). An important feature of the new system is the use of gas-tight or gas-flow environmental chambers which allow the laser micro-machining to be done in a controlled (e.g., inert gas) atmosphere to prevent oxidation and other chemical reactions in air sensitive materials. The gas-tight workpiece enclosure is also useful for machining materials with known health risks (e.g., beryllium). Specialized control software with a graphical interface enables micro-machining of custom 2D and 3D shapes. The laser-machining system was designed in a Class 1 laser enclosure, i.e., it includes laser safety interlocks and computer controls and allows for routine operation. Though initially designed mainly for machining of the diamond anvil cell gaskets, the laser-machining system has since found many other micro-machining applications, several of which are presented here. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4926889>]

I. INTRODUCTION

The diamond anvil cell (DAC) is the most common device for generating ultra-high pressures in a laboratory setting.¹ The technique of using a metal foil with a drilled hole placed between two diamond anvils to study the materials at high pressures under hydrostatic conditions was first developed in 1960s by Van Valkenburg and has since become a standard technique for high pressure research.^{2,3} Thus, the process of preparing a DAC high pressure experiment typically requires a robust capability for accurate machining of a round hole or several holes in the center of a gasket pre-indented to 10-100 μm . Typical dimensions for the gasket holes range from five to several hundreds of micrometers in diameter. The capability for precise machining of the smallest holes in gaskets is important for ultrahigh pressure experiments. Electrical discharge machining (EDM) systems have been routinely used for machining metal gaskets for the DAC experiments. However, with an EDM, machining a hole smaller than 50 μm is extremely challenging and machining a hole smaller than 25 μm is virtually impossible, which restricts the highest attainable pressure. Moreover, an EDM cannot be used for machining gaskets which are not electrically conductive (e.g., amorphous boron, silicon carbide, diamond, and oxides).

Aside from machining DAC gaskets, the capability to routinely machine sample assemblies with micrometer precision is required for carrying out special (e.g., high temperature)

experiments in a DAC. For example, laser heating experiments in the DAC require the preparation of precisely machined micrometer-scale thermal insulation layers (e.g., oxide or salt) which are challenging and time consuming to prepare manually due to their small sizes.

The DAC experiments often rely on X-rays for *in situ* study of materials under high pressure conditions and are routinely performed at synchrotron facilities; therefore, it is desirable to have a micro-machining system dedicated to the DAC experiments at a synchrotron beamline. We have designed and built the new laser micro-machining system at the High Pressure Collaborative Access Team (HPCAT), Sector 16 at the Advanced Photon Source (APS). The system is currently available for routine user operation.

II. SYSTEM DESIGN

A. Design goals

HPCAT facility at the APS hosts hundreds of visiting users each year from a broad range of scientific backgrounds. Given the large number of individual users, a system should be easily and safely operated with minimal training and at the same time robust and flexible enough to accommodate a large number of machining requirements. To meet these considerations, the system was designed to meet the following set of requirements:

- Class 1 laser system—no laser training or personal protective equipment required; it should be operational in crowded areas such as sample preparation laboratories.
- User friendly—easy to operate and the training takes only a few minutes.
- Unlike EDM micro-machining—it should be very easy to align the workpiece; also, the part being machined should have a location accuracy around $1\ \mu\text{m}$.
- Easy to use workpiece mount.
- 3D machining capabilities.
- Fast—machining of an average gasket hole typically takes about a minute or less.
- Sturdy and low maintenance—it is virtually impossible for users to misalign or accidentally damage the system.
- Environment control chambers—allow machining hazardous materials (e.g., beryllium) and materials sensitive to oxidation (with Ar-H₂ flow).
- Short pulse—ablates material without thermal melting.
- Very large dynamic range of laser power and laser frequency—can machine features ranging from $5\ \mu\text{m}$ holes in gaskets for ultrahigh pressure experiments to micro-machining mm sized pieces.

B. System components

The laser cutting/machining is accomplished by locally ablating small volume of material from the workpiece by a finely focused ($<3\ \mu\text{m}$ FWHM) pulsed IR laser⁴ while using motorized translational stages to move the position of the workpiece to trace out the required profile. For example, machining a $100\ \mu\text{m}$ diameter hole entails moving the

workpiece to trace out a $100\ \mu\text{m}$ outside diameter circle using two motorized translational stages with sub-micron resolution. In order to accomplish this, the laser machining system includes several key component sub-systems: the optical sub-system, the electrical sub-system, the motion control sub-system, the workpiece environmental containment sub-system, and the electronic interlocks and control sub-system.

1. The laser and the optical components

The layout of the laser and the main components of the optical sub-system are presented in a schematic view in Figure 1. The main component on the optical system is Teem Photonics (model PNP-M08010) Q-switched pulsed infrared (1064 nm) laser with a pulse width of 400 ps, $90\ \mu\text{J}/\text{pulse}$, and variable frequency modulated from 0 to 1000 Hz. Other components of the optical system include: a Class 1 green (532 nm) alignment laser, a laser beam expander to expand and adjust the focus of the IR laser beam, laser steering mirrors, a motorized rotational stage with a $\lambda/2$ waveplate (Figure 2(K)) and a Glan-Foucault prism polarizer (Figure 2(L)) for attenuating the laser power, a $20\times$ Mitutoyo NIR imaging objective (Figure 2(M)), and a short pass filter and two TV cameras (Figure 2(J)) with a specially chosen lens systems to provide two different magnification views of the workpiece simultaneously. The low magnification view is used for quickly locating and choosing the region of interest on the workpiece before machining, and the high magnification view is used for fine position alignment. A light-emitting diode (LED) illumination system (Figure 2(N)), which works in transmission mode, allows for imaging the laser-machined features and for viewing transparent samples.

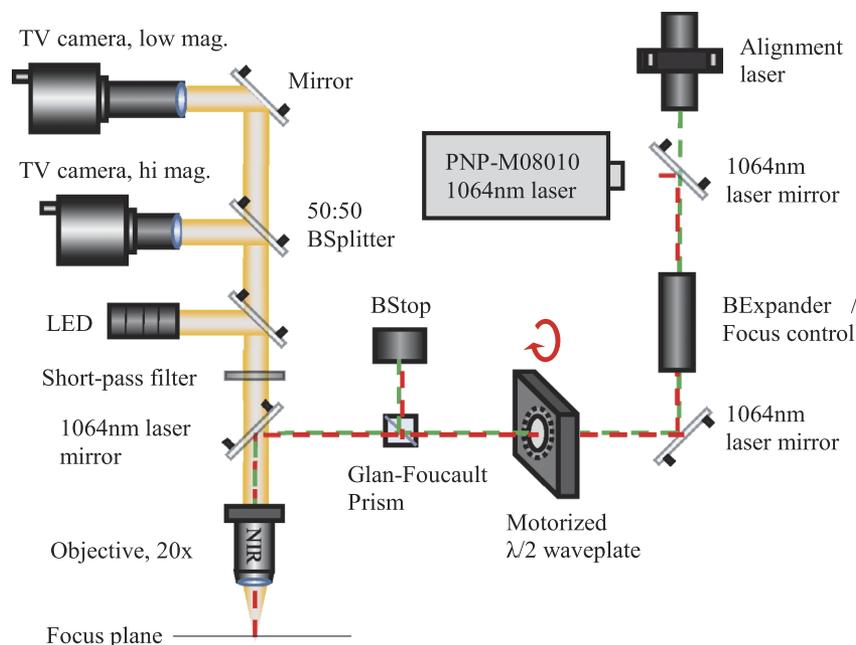


FIG. 1. Schematic layout of the optical sub-system. The IR laser beam (red dashed line) is combined with 532 nm alignment laser and then expanded and adjusted for divergence using a beam expander, attenuated as required using a $\lambda/2$ waveplate and a polarizer, and focused onto the workpiece surface through the $20\times$ objective. Visible image of the workpiece surface is magnified with the $20\times$ objective and transmitted to two separate TV camera imaging systems. An LED light source is used to illuminate the workpiece surface.

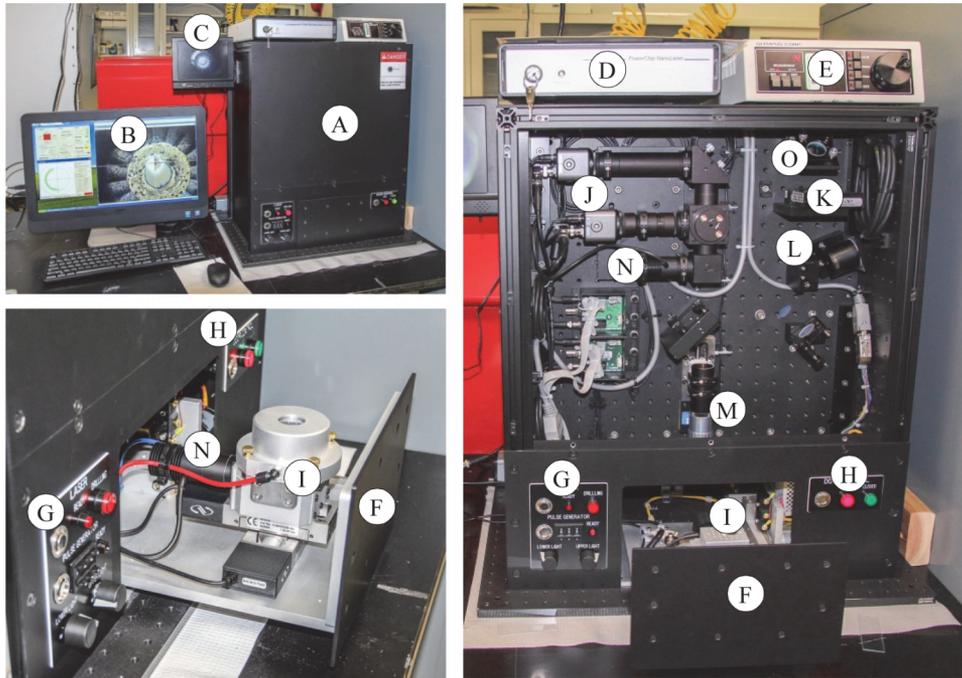


FIG. 2. Annotated photographs of several views of the laser-machining system. Overview of the laser-machining system during operation (upper left), laser-machining system with front cover removed for demonstration of internal component systems (right), slide out door and workpiece holder assembly (bottom left); (A) laser machining system enclosure; (B) all-in-one control computer; (C) low magnification view monitor; (D) laser key interlock; (E) TV cross-hair/reticle generator; (F) slide-out door cover plate; (G) laser and illumination controls and indicators; (H) door open/close control and gas flow connections; (I) workpiece holder assembly mounted on two horizontal motorized translational stages, shown with optional upper cover and gas flow connections; (J) TV cameras and imaging lens system; (K) $\lambda/2$ wave plate assembly; (L) prism polarizer and beam stop assembly; (M) imaging objective mounted on a vertical motorized translational stage; (N) LED illumination source. Compartment on the reverse side (not shown) houses the lasers, the laser expander optics, and the electronics control unit. The laser beam is steered from the rear side to the front optical system through a cut-out opening (O).

2. X-Y motion and focus control

The 2D motion of the workpiece stage (Figure 2(I)) and the motion of the imaging objective normal to the workpiece surface (Figure 2(M)) are enabled by use of motorized linear stages (linear motion resolution: $0.1 \mu\text{m}$; stage manufacturer: Newport; model: MFA-PP).

3. Laser power control

Laser power control is important for efficient micro-machining of different types of materials. Due to the differences in the laser interaction with the target materials, different materials should be treated with different laser powers. Specific laser machining mechanics related to different materials have been studied extensively and can be found elsewhere in literature.⁴⁻⁸ Moreover, the lowest laser power setting is useful for machining the smallest features (e.g., $5 \mu\text{m}$ holes in gaskets for ultrahigh pressure experiments), while the highest laser power settings can be used for rapid micro-machining millimeter sized features. In order to achieve a very high dynamic range of effective laser power, we allow controlling both the pulse frequency and/or the energy of individual laser pulses. The frequency modulation is enabled using a timing circuit which triggers individual pulses of the laser. The pulse energy control is achieved by using a $\lambda/2$ plate to rotate the laser beam polarization relative to a Glan-Foucault polarizer prism. The $\lambda/2$ plate rotation angle is adjusted with a motorized rotational stage (Newport, model SR50PP).

4. Electrical integration and the motion/logic controller

An electrical system has been designed to integrate all of the powered components of the laser micro-machining system in order to provide unified control and operation of the system.

The 4-axis controller/driver (manufacturer: All Motion; model: EZ4AXIS) is used to drive all of the motorized stages in the system (described in Sections II B 2 and II B 3). The EZ4AXIS controller features a number of digital and analog inputs/outputs which are used as interfaces for programmable logic that monitors the safety interlocks (described in Section II C) and enables the operation of the laser. A schematic block diagram of electronic inputs and outputs is shown in Figure 3. The EZ4AXIS controller interfaces with a Windows based control PC via a USB connection. The control PC and the software are described in Section II D.

5. Workpiece holder, gas flow system, and gas-tight enclosure

The environment control chamber allows laser-machining hazardous materials (e.g., beryllium) and materials sensitive to oxidation (with Ar-H_2 flow). The chamber includes a lower base with a quartz window for bottom illumination, a magnetic-release base with a central aperture to allow for the through-illumination of a piece being machined and a micro-breadboard (a custom made plate, 1.75 in. in diameter, includes

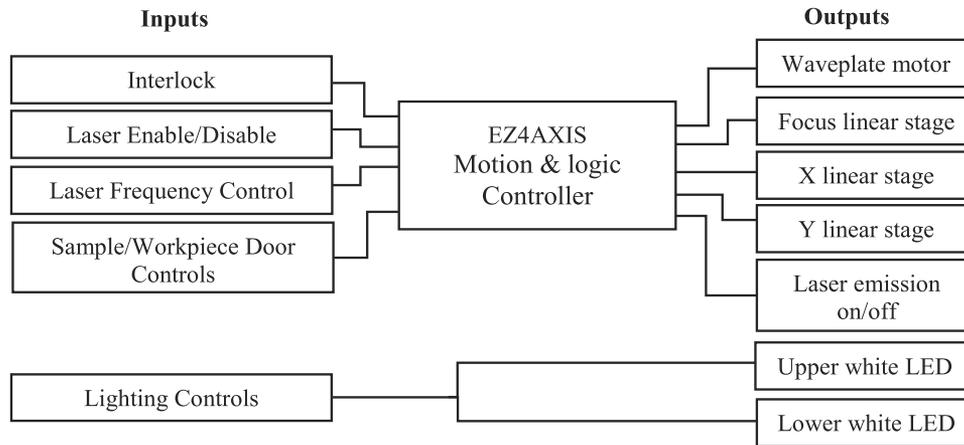


FIG. 3. Block diagram of the electric control sub-system of the laser-machining system. This sub-system manages all of the powered components of the laser-machining system. The motion and logic controller constantly monitors the state of interlocks, front panel push-buttons, laser pulse frequency generator settings, and the laser enable state.

4-40 UNC tapped holes arranged in a square grid spaced 0.25 in.) for holding the workpiece. Optionally, an upper cover which includes gas flow connectors and a transparent fused quartz window can be placed over the lower base plate and fastened with four screws to create a protective environment for laser machining a workpiece. A cross-sectional computer-aided design (CAD) drawing of the workpiece holder with the top cover attached is shown in Figure 4.

The environmental control chamber of the laser-machining system meets the safety standard at the APS for beryllium machining. The chambers can be loaded/unloaded only in special designated areas, while the machining itself can be performed in a general user area, where the laser micro-machining system is located. A total of 4 identical environment modules have been manufactured and are available for use at HPCAT. Having separate modules ensures that there is no contamination of general users' materials from the modules which have been used in the laser processing of hazardous materials. Moreover, the environmental control modules can be loaded and sealed externally and placed into the laser-machining system. The top part of magnetic-release base (Figure 4) (Thorlabs, model SB1) is interchangeable to potentially allow users to easily fit custom-mounted workpiece assemblies into the laser-machining system.

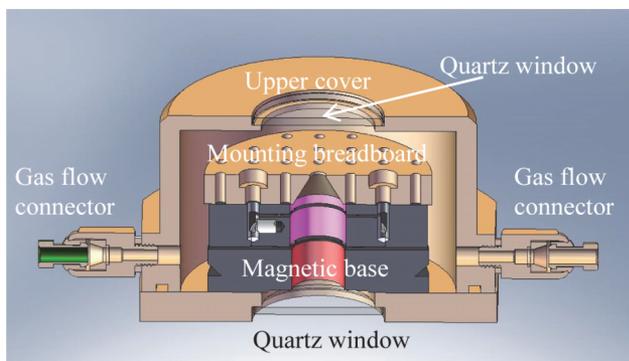


FIG. 4. CAD drawing of the workpiece holder on a kinematic mount and an upper cover equipped with gas flow connectors.

6. Class 1 laser enclosure

Although the pulsed laser is classified as Class 3B, the laser machining system was designed to meet the safety category of Class 1 laser, i.e., to be compatible with use in general areas by users without any specific laser safety training. Therefore, the IR laser and the beam path are contained inside of a rigid black anodized aluminum enclosure (Figure 2(A)). The interlock system disables the laser emission if the enclosure is open. The front panel of the laser machining system enclosure contains several push-button controls and indicators (Figures 2(G) and 2(H)) as well as the door cover of the motorized slide-out drawer which houses the workpiece stage (Figures 2(F) and 2(I)). Motorized opening and closing of the workpiece stage door helps to minimize mechanical shocks and maintain long term system stability. The push button on the right-hand side controls the opening and closing of the slide-out workpiece door/drawer (Figure 2(F)) and the adjacent indicators (Figure 2(H)) display the state of the door interlock and the overall readiness of the system. The controls on the left hand side of the front panel are used to enable and adjust the laser settings (i.e., laser pulse frequency) and operate the white-light LED illumination lamps (Figure 2(G)). Internally, the enclosure is divided into three distinct compartments. The bottom compartment houses slide-out drawer with the workpiece stage and interlock sensors and relays. The top-rear compartment houses the IR laser, an alignment 532 nm laser, the laser beam expander, and the logic controller electronics. The top-front compartment (Figure 2, photograph on the right) houses the optical components described in Section II B 1.

C. Safety and operational interlocks

1. Hardware safety interlocks

Hardware safety interlocks ensure that a user cannot be exposed to the powerful IR laser beam during normal operation. The primary safety interlock is a key based system (Figure 2(D)) which ensures that the laser will turn on only when the key is inserted and turned towards the Power On position. The secondary hardware interlock ensures that the

IR laser may not power on whenever the workpiece drawer is open. Whenever any of the hardware interlocks have been opened and closed, the IR laser must be manually re-enabled by the user by pressing a button on the front panel.

2. Software interlocks

Several additional interlocks, which are not essential to safety but largely for reliable operation, are handled in the client software (described in Section II D). These interlocks help to ensure predictable behavior of the laser system during the actual process of machining a workpiece. The software system of interlocks is programmed to follow several logical rules in order to ascertain the readiness of the system and decide whether to allow the machining routine to commence.

- (1) The system is *ready* when all of the following are true: door interlock is closed, the laser pulse generator is turned on, and the laser enabled signal is on.
- (2) The laser *emission-on* signal may only be issued when the system is in the *ready* state.
- (3) The laser *emission-on* signal must be turned off whenever the system is not in the *ready* state.
- (4) The machining routine may be allowed to start only when the system is *ready*.
- (5) The machining routine must automatically pause if, at any time during the machining, the system is no longer in the *ready* state.
- (6) If the software program is unexpectedly terminated by the user, the laser *emission-on* signal must be turned off.

D. Control PC and software

The computer program for controlling the laser-machining system is based on LabVIEW and can run on any stand-alone MS-Windows (XP, 7 or 8) computer. The main user interface window of the control program is shown on Figure 5. From the

main window, a user can align the workpiece position and focus, adjust the laser power setting, view the status of safety interlocks, and open windows for various machining sub-routines, each of which comes with its own interface window. Each of the dedicated machining interfaces handles the calculation of the 3D shape drive-path and executes the drilling. The most commonly used micro-machining interfaces are as follows:

- *Circle*. The circle interface window can be used to machine any size circular or elliptical shapes. This is the most commonly used window (Figure 6) and it is normally used to machine round holes in pre-indented gaskets for the DAC experiments. Aside from making holes, this window can also be used to machine round samples from sheets of starting material. Several examples are given in Section IV A 1.
- *Circles*. This interface allows the user to machine 1D or 2D arrays of circles from sheets or foils of starting materials used for unattended machining of many samples.
- *Recess*. This window can be used to machine 3D holes and pits.
- *Rectangle*. The rectangle is commonly used to machine square or rectangular samples out of sheets or foils of starting material.
- *Custom*. The custom window can be used to load user-supplied geometry coordinate files to cut along custom 2D or 3D profiles used for special micro-machining, etching, and engraving applications. Several applications are given in Sections IV C and IV D.
- *Manual*. The manual window allows the user to control the machining path in real-time with the motion of the computer mouse, and can be used to fine tune already machined parts or quickly create custom shape μm -size parts without the prior need to generate geometry coordinate files.

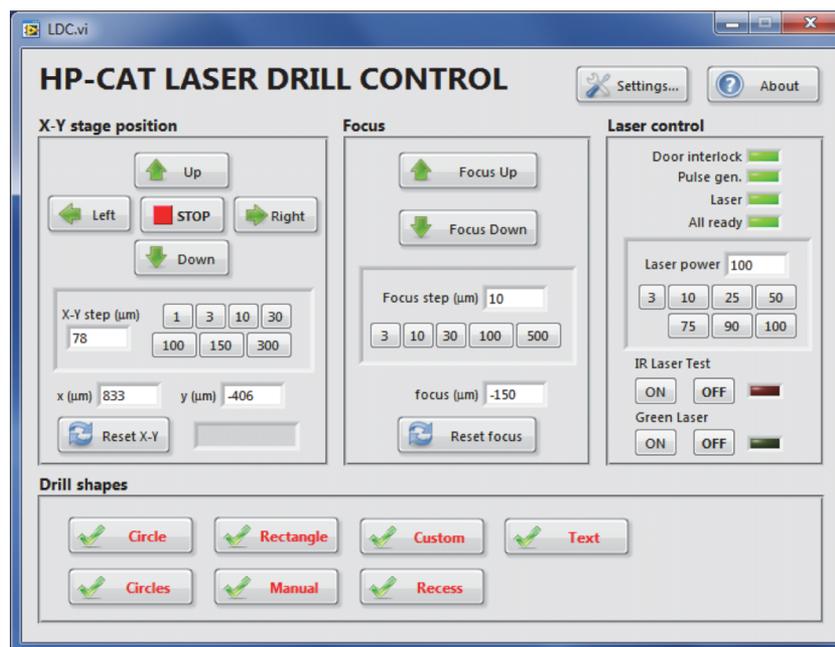


FIG. 5. Main window of the graphical user interface of the HPCAT laser-machining system. Separate machining functions are opened in separate windows.

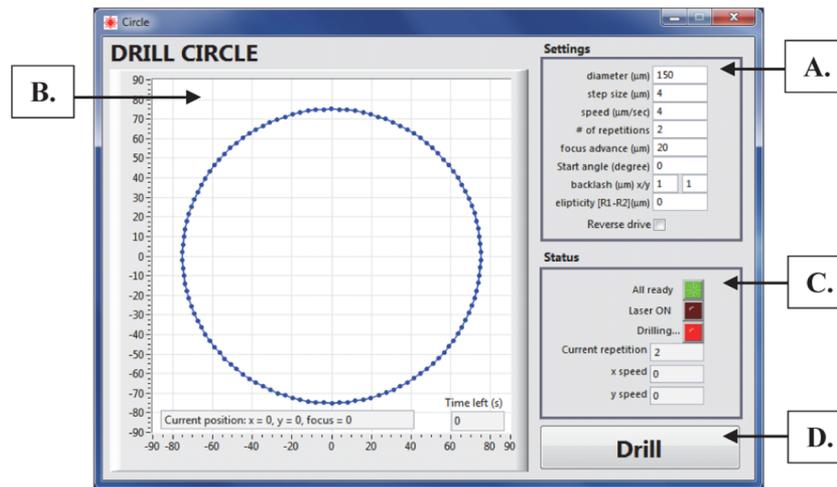


FIG. 6. Annotated screen capture of the circle machining graphical user interface (A—circle machining settings, i.e., circle dimensions, machining velocity, etc.; B—calculated machining path, discrete points interpolate the circular motion of the workpiece stage during the machining process; C—machining process monitor; D—button to start the machining process).

III. SYSTEM OPERATION

The laser-machining system was commissioned and made available for user operation in June 2014. During the first 6 months of operation, approximately 3000 gaskets were micro-machined by various users for DAC experiments at the APS. Such an unexpectedly high throughput has demonstrated the ease of use and robustness of the new laser micro-machining system. The success of the new system comes from the fact that it was designed from the beginning as an integrated hardware system complete with complimentary dedicated software.

The basic list of steps for machining a hole in a DAC gasket, or any other part/sample, using the new laser-machining system is as follows:

- The gasket is placed flat and attached to the workpiece stage.
- The 2D position of the gasket and focus are adjusted using the graphical user interface (GUI) in the main program window to center the gasket. Rough alignment is done by watching the low magnification TV image. Fine position adjustment is done by viewing the high-magnification video on the PC and aligning the center of the indented gasket with the cross-hair overlay.
- Laser power is adjusted to suit the particular gasket material and the machining path thickness.
- Each of the interlocks is checked by looking at the indicators on the main GUI window to make sure of the readiness of the laser-machining system.
- *Circle* GUI window is opened by clicking the *Circle* button on the main window and the circle dimension settings are input.
- Clicking the *Drill* button starts the machining process.
- After the machining is complete, the door can be opened and the gasket safely removed.

Machining other shapes can be done by following a similar set of steps. Each of the available shape routines as well as the custom and manual machining options is routinely used.

IV. APPLICATIONS

A. Machining circular features

1. Machining circular holes in diamond anvil cell gaskets

By far, the most common application of the new laser machining system is the cutting of circular holes in pre-indented DAC gaskets. The dimensions of the required gasket holes vary greatly from 5 μm for ultra-high pressure experiments up to about 1000 μm for relatively low pressure experiments which, for various experimental reasons, require a larger volume of material. Most commonly, users arrive with diamond anvils of 150–300 μm in diameter, which requires them to cut gasket holes of 80–150 μm in diameter. A microscope photograph of a typical size hole laser-cut in rhenium metal gasket, pre-indented between double-bevel diamonds to 25 GPa, is shown in Figure 7(a). The gasket shown in the microscope photograph in Figure 7(a) is purposely tilted with respect to the microscope imaging plane in order to demonstrate the typical quality of the sharp cut edge achieved by the laser micro-machining system.

Typical ultra-high pressure (>1 Mbar) experiments require gaskets to be made with a hole of 50 μm in diameter and smaller, and centered with ~ 1 μm accuracy. Numerous ultra-high pressure experiments have been conducted with gaskets prepared with the help of the new laser-machining system.

In addition to metallic gaskets, some users' experiments require the use of non-conductive gasket material, such as pressed boron-epoxy (Figure 7(b)), cubic boron nitride, or other ceramic composites. Such non-metallic gaskets have also been routinely machined using the new system.

2. Machining specialized sample assemblies for the laser heated DAC

Some DAC experiments require the generation of high temperature in addition to high pressure in order to study materials under a broad range simultaneous high-temperature and

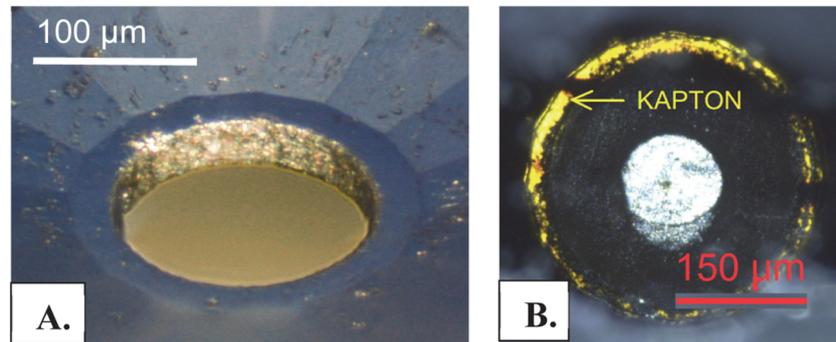


FIG. 7. Microscope photographs of circular holes machined in pre-indented gaskets for DAC experiments. (a) Gasket material: rhenium, indented thickness $\approx 30 \mu\text{m}$, hole diameter = $110 \mu\text{m}$; (b) laser-machined boron-epoxy gasket (grey ring) held in a laser-machined polyamide (i.e., Kapton) outer gasket (yellow ring), viewed through the diamond (image credit: Ligang Bai).

high-pressure. The laser heating technique is now commonly used in order to produce temperatures of thousands of degrees in samples pressurized in a DAC.^{9–11} Unlike a general room-temperature DAC experiment, the laser heated DAC technique comes with an additional set of sample preparation and loading requirements. For a proper sample loading, i.e., in order to attain a desired temperature response during laser heating, one must include optically transparent and thermally insulating layers between the sample and the diamonds. Incorrectly loaded samples will likely show temperature instabilities during laser heating which can lead to unsuccessful experiments. At very high temperatures, laser heated metal samples may form carbides due to carbon diffusing from the diamond anvils¹² and therefore have to be insulated from the diamonds as well as possible.

We find that full encapsulation of a metal sample within single-crystal layers of a material with a melting temperature above that of the metal sample helps to avoid laser heating temperature instabilities associated with other sample loading

techniques. That is, for many metals, Al_2O_3 encapsulation works well, whereas for metals with extremely high melting temperature (e.g., molybdenum), the only insulation medium that should be used is MgO , which has an even higher melting temperature. Thus, the new laser-machining system has provided the technical capability for micro-machining special geometry sample and insulation layers' assemblies (Figure 8) to achieve repeatable and reliable temperature response in laser heated samples.

Our recent observations have shown that using laser-machined single crystal MgO assemblies instead of previously used compressed MgO powder for the encapsulation of molybdenum samples helps to completely avoid the metal's surface modification during laser heating. Avoiding surface modifications of the samples during laser heating helps to achieve a very stable temperature response in the laser heated samples up to and above the melting temperatures. A predictable temperature response in laser heated molybdenum samples under high pressure has enabled us to conduct reliable experiments with

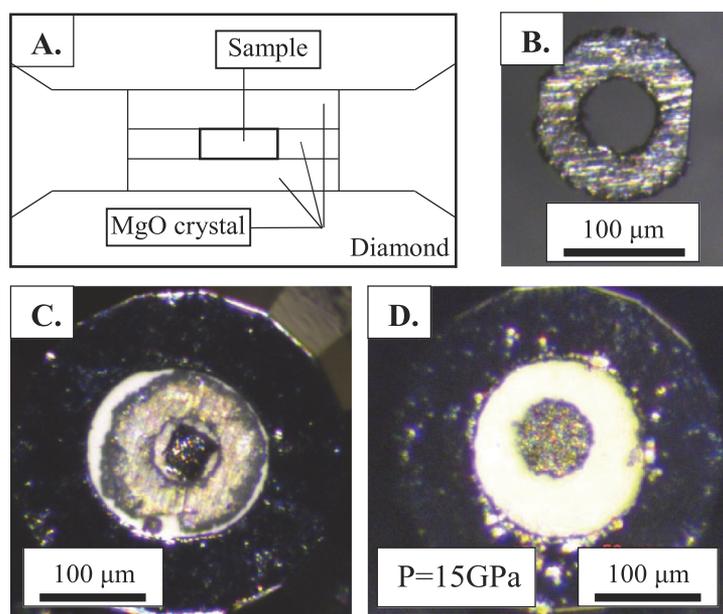


FIG. 8. A bismuth metal sample is confined between two MgO single crystal layers and a MgO single crystal micro-gasket layer. (a) Schematic drawing of sample encapsulation. The middle layer secondary gasket, laser-cut from a $15 \mu\text{m}$ thick sheet of single crystal MgO (also shown in (b)), prevents the sample from spreading out radially during axial compression. (b) Microscope photograph of a MgO crystal secondary micro-gasket, typical thickness $\approx 15 \mu\text{m}$. (c) Microscope photograph of the sample assembly before closing the DAC. (d) MgO encapsulated metal sample in a DAC at a pressure of 15 GPa.

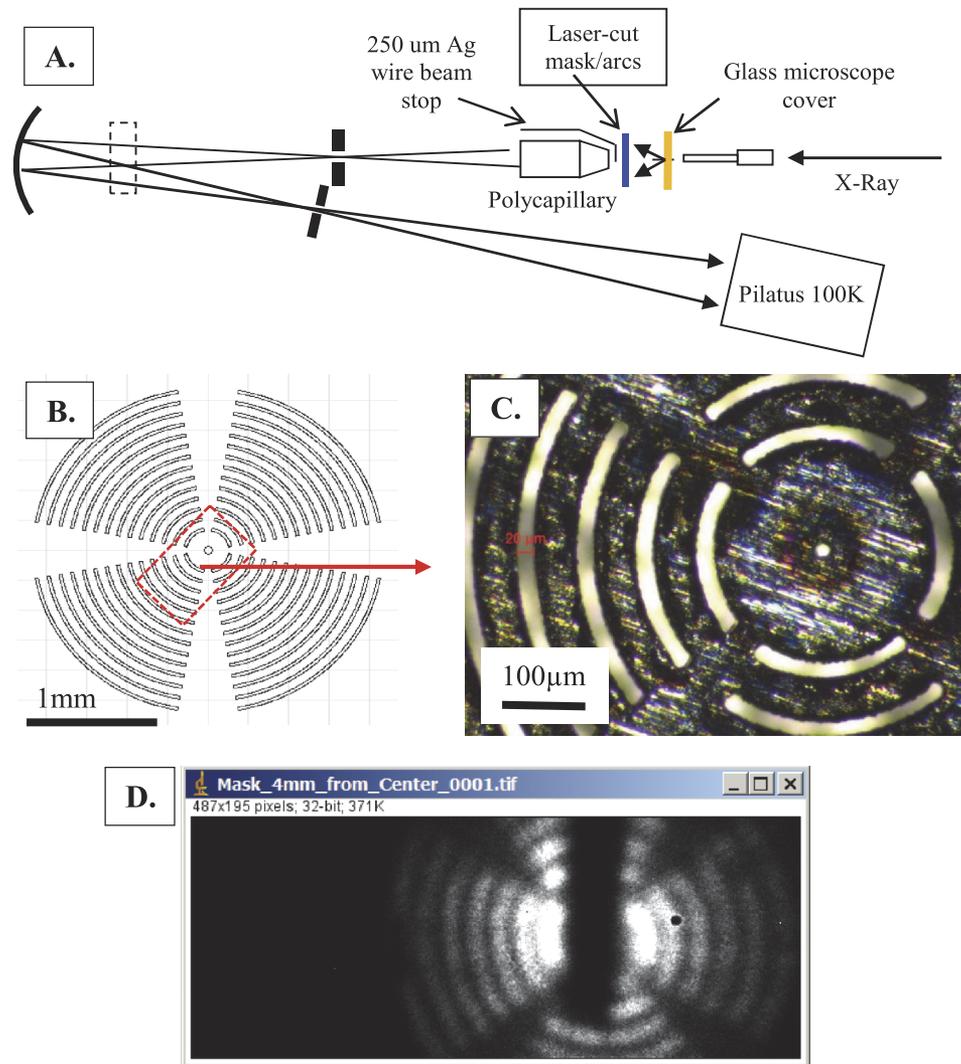


FIG. 9. (a) Schematic layout of the inelastic x-ray scattering (IXS) experimental setup at the x-ray beamline 16 ID-D at APS, the location of the laser-cut IXS calibration mask is shown (image credit: Paul Chow); (b) IXS mask machining path schematic; (c) a microscope photograph of a magnified region on the IXS calibration mask. The outline of the region shown in the photograph with respect to the entire IXS mask is shown by the dashed rectangle in (b); (d) an IXS angle calibration image collected with the Pilatus 1K detector.

in situ x-ray diffraction and establish melting temperatures of molybdenum at high pressures. New data on the melting study of molybdenum melting under high pressure using the laser-cut insulation layers will be reported elsewhere.

3. Laser cutting of multiple parts for large volume press (LVP) assemblies

LVP technique, currently established at HPCAT,¹³ requires rapid manufacture of round platinum caps to act as endpoints of the pressure chambers. The manufacture of a large number of the platinum caps for LVP experiments is now possible using the new laser-machining system. Typical dimensions of the platinum caps are 1/16 in. (~1.6 mm) in diameter and 25 μm in thickness.

B. Machining with a gas-tight workpiece enclosure

An environment control chamber, as described in Section II B 5, allows micro-machining of hazardous materials such as

beryllium, which is a gasket material for x-ray diffraction and x-ray spectroscopy experiments in radial geometry in DAC. Beryllium has very low x-ray absorption and x-ray scattering (compared to, e.g., rhenium, steel) and helps to obtain better signal from the samples in x-ray scattering experiments.

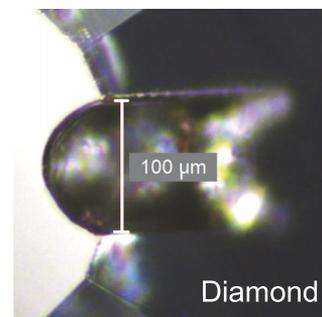


FIG. 10. Microscope photograph showing a recess cut on the side of a culet of a single crystal diamond. The depth of the recess is 150 μm . The recess surface (not in focus) is transparent and not darkened.

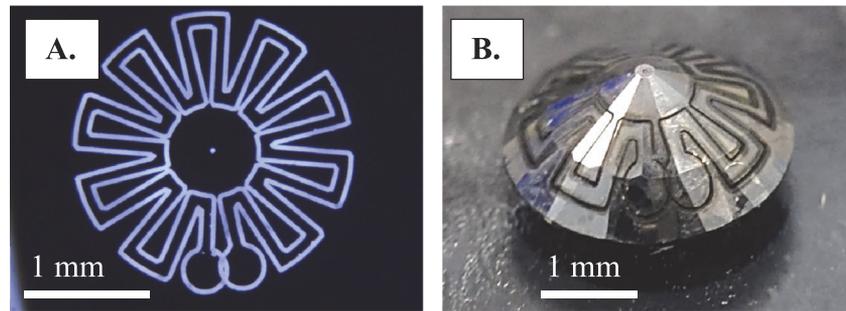


FIG. 11. 3-D resistive heating circuit patterned onto a tungsten sputter-coated diamond anvil. (a) Optical microscope photograph of the patterned circuit in transmission mode. (b) Photograph of the finished diamond anvil resistive circuit (image credit: Jeffrey Montgomery).

C. Complex 2D shapes

1. Inelastic x-ray scattering (IXS) calibration mask

The capability of cutting complex 2D shapes with the new laser micro-machining system can be demonstrated by an example of the machining of an arc-mask for the calibration of the forward IXS angles in a setup with a poly-capillary device. The use of poly-capillary in x-ray spectroscopy applications is described elsewhere in literature.^{14,15} A new x-ray spectroscopy development project at beamline 16 ID-D of HPCAT required a special mask to be machined out of high-Z metal (e.g., molybdenum) with μm -level precision. A schematic layout of the experimental setup showing the location of the IXS calibration mask is shown in Figure 9(a). The IXS mask consists of concentric arc slots made in a 6 mm (outer diameter), 25 μm thick molybdenum disk. A drawing of the required laser-machining path is shown in Figure 9(b). A microscope photograph of a magnified region of the finished IXS mask is shown in Figure 9(c). The radius of the arc slots closest to the central hole is 140 μm , and thickness of each arc slot is 20 μm . The calibration mask was successfully used in the setup shown in Figure 9(a) to calibrate the x-ray scattering angles. An IXS calibration image collected with the Pilatus 1K detector is shown in Figure 9(d).

Moreover, machining of the mask succeeded due to the fact that the machining could take place in an oxygen free environment to prevent the oxidation of molybdenum. In fact, initial attempts to machine molybdenum in air produced undesirable results due to rapid surface oxidation. The environmental chamber (described in Section II B 5) with UHP argon + 2% H_2 flow was used to reduce the surface reactions on the workpiece during machining. Some surface reaction was seen next to the cut-edges after machining of the workpiece (darkened regions adjacent to the cut edges, Figure 9(a)); however, the machining still produced sharp cut-edges, which could not be achieved without the use of the environmental chamber.

D. Complex 3D shapes

1. Cutting complex 3D shapes: Recessed diamonds

In order to minimize the background signal caused by diamond x-ray scattering in elastic x-ray scattering DAC experiments at 16 ID-D, special 3D recesses were cut out on the side

of the culets of diamonds using the *Recess* machining function. This application demonstrates the capability of the laser-machining system to successfully machine features in bulk single crystal diamond. The ability to machine and shape diamond culets with the new laser-machining system can potentially lead to many interesting applications in DAC high pressure science. A microscope photograph showing the laser cut recess feature on the side of a diamond culet is shown in Figure 10.

2. Cutting along a complex 3D path

The laser-machining system can work with user-supplied geometry coordinate files to cut along custom 3D paths. An experimental heating-element circuit was micro-machined onto a diamond anvil which was previously sputter-coated with tungsten (Figure 11). The resistive heater diamond anvil is a novel development for high temperature and high pressure material studies.

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