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Electric discharge machine for preparation of diamond anvil cell sample chambers

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We have designed and constructed a novel electric discharge machine designed primarily for the preparation of sample chambers in rhenium and stainless steel gaskets for diamond anvil cell experiments. Our design combines automatic stage movement with relatively low voltage (100 V) operation and routinely achieves a drilling / erosion speed of approximately 0.4 µm s⁻¹. The machine is used for preparing 100 µm diameter sample chambers for diamond anvil cell experiments with 250 µm culets, and has also been used to prepare 50 µm diameter sample chambers for diamond anvil cell experiments with 100 µm culets to access pressure of 165 GPa.

I. Introduction

Since its invention in the late 1950s the diamond anvil cell (DAC) has allowed the generation of static high pressures in the laboratory far in excess of those possible with any other device. Due to the insulating and transparent nature of the diamonds the DAC also allows a large variety of experimental measurements to be conducted on samples in-situ at high pressure: Many optical spectroscopy techniques, electrical resistivity measurements, X-ray and neutron diffraction, X-ray spectroscopy and (last but not least) direct visual observation of samples using an optical microscope.

The DAC (figure 1) works by compressing the sample between the culets of 2 diamonds, with a metal plate (the gasket) constraining the sample in the lateral direction. The diamond culet diameter varies depending on the required pressure, with a smaller culet allowing the generation of higher pressure (figure 1). Typically, for experiments using culets of 100 µm diameter or smaller a bevelled diamond culet is utilized (diameters quoted are those for the inside of the bevel rather than the outside). The maximum pressure achievable, assuming good alignment etc., is limited by breakage of the diamonds (all but one of the data points in figure 1 correspond to diamonds the authors have personally broken). Using the conventional DAC with bevelled diamonds it is possible to reach pressure of just over 400 GPa [1]) but higher pressure have been reported using novel double stage DACs[2].
Figure 1. (Left) Graph showing maximum pressure achievable in the DAC as a function of diamond culet diameter. (Top right) Schematic diagram of typical DAC experiment [3]. PTM refers to the pressure transmitting medium, and the gasket is shaded in grey. (Bottom right) Photograph of typical DAC sample chamber. The schematic diagram and photograph are adapted with permission from D. Smith, R.T. Howie, I.F. Crowe, C.L. Simionescu, C. Muryn, V. Vishnyakov, K.S. Novoselov, Y.-J. Kim, M.P. Halsall, E. Gregoryanz and J.E. Proctor, ACS Nano, 2015, 9 (8), pp. 8279-8283. Copyright (2015) American Chemical Society.

Prior to the high pressure experiment the gasket is prepared by indenting in the DAC until it has a thickness of between 1/10th and 1/6th of the culet diameter, then the sample chamber (with diameter about 50% of the culet diameter) is created in the centre of the indentation using a mechanical drill, electrical discharge machining (EDM) or laser cutting. The error in positioning of the sample chamber must be small compared to the culet diameter in order to achieve the highest pressures possible with the culet diameter in use. Typically, mechanical drilling is used for the very largest sample chambers (ca. 300 μm sample chambers for 600 μm culets), laser cutting for the smallest (up to 50 μm sample chambers for 100 μm culets). Electrical discharge machining is used for most sample chambers.

In a typical DAC experiment, diamonds with 250 μm diameter culets may be utilized, and a 100 μm diameter sample chamber drilled in an indented rhenium gasket to achieve pressure of up to 70 GPa.

II. Principles of EDM

The use of EDM in machining both small and large workpieces predates the invention of the DAC, let alone the use of EDM to create DAC sample chambers. In the EDM process a conducting electrode is gradually brought closer to an (also conducting) workpiece until sparks flow between them, at which point erosion of the workpiece takes place and the workpiece and sample can then be brought closer together to allow erosion to continue. A mark in the workpiece the shape of the electrode is created.
The process is conventionally referred to as “drilling”, even though it is not drilling in the normal sense of the word; The process takes place when the workpiece and electrode are not touching. Instead, the workpiece and electrode are gradually brought closer together with a constant high voltage applied between them. At some point, the electric field strength in the region between the workpiece and the electrode exceeds the dielectric breakdown strength of whatever medium is present in this region. This is when sparks begin to flow and erosion due to localized melting of the workpiece takes place. For this reason, EDM is usually not performed in air, but with a fluid such as paraffin oil between the workpiece and sample that has a significantly lower dielectric breakdown strength than air. The lower breakdown strength ensures that erosion takes place when the electrode and workpiece are further apart, reducing the risk of the electrode and workpiece touching. In addition, the presence of a dielectric fluid allows debris produced in the erosion process to be dispersed [4].

III. Challenges in the application of EDM to machining of DAC sample chambers

The application of EDM to the machining of DAC sample chambers is challenging for several reasons. Firstly, extremely accurate positioning of the sample chamber in the centre of the indent is required, although the alternate approach of eroding the hole before indentation has been attempted [5]. Secondly, the workpiece and electrode need to be extremely close (< 10 \( \mu \)m) together for erosion to take place. This leads to the ever present risk of them becoming too close or even touching, which can lead to (1) Short-circuiting of the power supply, (2) The workpiece becoming welded to the electrode. (3) The electrode being knocked out of position - since the electrode is a wire with ca. 100 \( \mu \)m diameter only the slightest force is required for this to happen. In addition, application of EDM to eroding very large sample chambers (300 \( \mu \)m + diameter) is hampered by the fact that it can be a very slow process – the speed with which a sample chamber can be eroded is expected to increase as the cube of the chamber diameter.

However, in order to obtain the highest possible pressure in a DAC experiment, it is important that the sample chamber is as circular as possible – an irregularly shaped sample chamber will be less stable. EDM achieves the neatest (no burrs or offcuts) and most circular sample chamber possible with any technique – it is therefore worthwhile to seek better solutions to the challenges described above to enable the use of EDM to drill sample chambers for which laser cutting and mechanical drilling are currently used.

IV. Design of our EDM

In the design of the EDM presented here, we began from four basic principles:

1) The voltage used for erosion was set at 100 V. In principle, a higher voltage gives better results. It ensures that the critical breakdown field is reached when the electrode and workpiece are further apart so reduces the risk of them touching. However, using a higher voltage leads to potential safety issues and also to the sample chamber eroded in the workpiece potentially being much larger than the electrode and hard to control. Our design differs in this way from the principal existing EDM design for DAC gasket preparation in the literature, which operates at 1.2 kV [6].
2) The mechanical alignment was to be performed using commercially available parts designed for alignment of optical components – these allow accurate and reproducible alignment to an accuracy of ca. 1 µm.

3) A microscope examining the workpiece vertically was to be utilized to enable viewing the best possible image of the workpiece during alignment, and prevent possible errors due to oblique viewing.

4) Automatic stage movement in the z-direction was to be implemented as an essential labour-saving feature.

The mechanical design of our EDM is presented in figure 2. The workpiece is held in place on the stage by a metal plate with a circular aperture screwed down over the workpiece, which also provides electrical connectivity. The stage is mounted on an xyz micrometer mount which can be moved with a precision of ca. 1 µm. In the xy plane the stage is moved using manually controlled micrometers and in the z axis the stage is moved using a motorized micrometer (Thorlabs Z825). The electrode is a short (ca. 2 mm protruding) tungsten wire crimped into an aluminium tube (outside diameter 2.0 mm, inside diameter 1.0 mm) which is in turn held in place and electrically connected using a screw terminal. This assembly is held in place using two Thorlabs kinematic bases (KB25). The positioning of the electrode is therefore not adjustable but is reproducible to a precision of ca. 1 µm.

The electrode can be removed by prising apart the kinematic bases and a microscope can be placed on the same kinematic bases with the same precision and reproducibility. The microscope eyepiece is fixed and the objective lens can be moved in the xy plane by a small but adequate amount using the adjustment on the lens mount (Thorlabs CXY1). A reticle consisting of cross hairs and concentric circles is placed at the focal point of the eyepiece lens, between it and the objective lens. In figure 3 photographs are shown of the instrument with the electrode mounted ready to drill, and with the microscope mounted for alignment.

![Figure 2](image)

Figure 2. (left) Schematic diagram of EDM in erosion position. (right) Schematic diagram of EDM in viewing position. W – workpiece, P – paraffin oil.
The electrical circuit diagram of the EDM with principal components labelled is shown in figure 4, and the complete circuit diagram including component values and manufacturer part numbers is shown in the supplementary information. The instrument operates using two power supplies, V1 (24 volt r.m.s. a.c.) and V2 (15 volt d.c.). A 3 volt d.c. supply (V3) is produced using the 15 volt d.c. supply and the adjustable voltage regulator TS317CM. R2 is adjusted until the output is exactly 3 V when the instrument is constructed. The three volt supply operates an adjustable light to illuminate the stage and a light which is illuminated when both power supplies are turned on and the unit is ready to drill (i.e. both S1 and S2 are closed).

The high voltage applied between the electrode and the workpiece is generated by connecting V1 to a cascade voltage multiplier circuit of the Greinacher / Cockcroft – Walton design (1N4934 silicon diodes D1 – D6 and ceramic capacitors C1 – C6). In theory this multiplies V1 by a factor of 6 to 144 V, in practice due to losses in the components the output is 106 V. This method is used in preference to utilizing a conventional 100 volt d.c. power supply because it is more robust against the effects of short-circuiting (e.g. if the workpiece and electrode touch).
The d.c. supply generated by the voltage multiplier is then fed through a capacitor discharge unit (CDU) to allow accurate control of how much charge flows in each spark. The total capacitance of the CDU, and therefore the, charge flowing in each spark is determined by which of the capacitors C9 – C12 are switched into the circuit using switches S3 – S6. The capacitors charge through the transistor Q1 as this leads to faster charging than a pure RC circuit. The capacitors discharge through the 1N4934 silicon diode D7. The 0.6 volt voltage drop across the diode ensures that Q1 is turned completely off whilst the capacitors are discharging by placing the base terminal at a lower voltage than the emitter terminal. The variable resistor R1 is adjusted so that when the electrode and workpiece are not close (i.e. open circuit) the voltage between the electrode and workpiece is 100 V. This is monitored using the voltmeter U1. The switch S7 reverses the polarity of the voltage applied between the electrode and workpiece. For the gasket materials we drill (rhenum and stainless steel) and the electrode we use (tungsten) applying the positive voltage to the electrode gives the best results, but the voltage can be reversed if it is desired instead to erode the end of the electrode to make it flatter.

The Z825 motorized actuator that moves the stage up and down is also connected into the circuit shown in figure 4. The actuator is connected up using the supplied 9 pin DIN connector and a voltage of 3 V applied between pins 5 and 7 to slowly move the actuator. The direction of movement depends on the polarity of the applied voltage. The stage can be moved up or down manually (by closing S9 or S10) or, if S7 is switched into position 1 it will be moved automatically whilst eroding.

To move the stage automatically during erosion the voltage between the electrode and workpiece is monitored. Under open circuit conditions the voltage remains at 100 V and the stage is therefore moved up to come closer to the electrode. When the dielectric breakdown strength of the paraffin oil is reached and sparks fly, the voltage between the electrode and workpiece drops and the stage is then moved down.

This automatic movement is achieved as follows. The voltage between the electrode and workpiece cannot be connected directly to the inputs of U2 as it would be much higher than the supply voltage for U2 when the electrode and workpiece are not close. The voltage is therefore reduced to ca. 15 V using potential divider R6. It is then fed into operational amplifiers U2A and U2B both connected as comparators (no feedback). U2A and U2B compare the voltage to a reference voltage produced by appropriate adjustment of R5. Under open circuit conditions (i.e. the circuit needs to move the stage up) U2B produces an output close to 3 V and U2A produces an output close to 0 V. Conversely, when the electrode and workpiece are close and the stage needs to move down U2A output is close to 3 V and U2B output is close to 0 V. This reversible polarity is then fed into the Z825 actuator via the h-bridge circuit consisting of Q2 – Q7.

The EDM can be left unattended whilst erosion takes place though it is only for the larger sample chambers that this is necessary or wise, due to the high erosion speed.

V. Operational procedure

The electrodes are produced by inserting commercially available tungsten wire into a length of aluminium tube and crimping the end of the tube to secure the wire. The wire is then cut off ca. 2 mm beyond the end of the tube. For preparing sample chambers 100 µm diameter and above we use wire purchased from Goodfellow and for the smallest sample chamber the machine has drilled (60 µm diameter) we instead used a tungsten scanning tunnelling microscope (STM) tip purchased
Once the electrode is prepared, the procedure to operate the EDM is as follows:

1. Following indentation, the workpiece is placed on the EDM stage and secured.
2. Pre-alignment process. The EDM is set up in the erosion position and the stage is manually moved upwards until it is close to the electrode. The stage is then moved in the XY plane until visual inspection indicates that the electrode is over the indentation in the gasket (workpiece). The EDM is then set up in the viewing position and the stage is moved up / down to focus. The objective lens is then moved until the mark of the indent is clearly visible through the eyepiece. If the electrode has already been aligned for a previous erosion process and not adjusted since, this pre-alignment process can be skipped.
3. The EDM is set up in the erosion position and the stage is moved so that the electrode is above some flat area of the workpiece away from the indent. Paraffin oil is added into the stage and automatic erosion is used to drill a test hole.
4. The EDM is set up in the viewing position and the paraffin oil is removed using a pipette. Cotton buds are then used to soak up every drop of oil from the actual workpiece as if it is not completely dry the image in the microscope can be distorted. The objective lens is moved until the test hole is visible, and is in the centre of the reticle cross-hairs.
5. The stage is then moved until the indent of the diamond culet is in the centre of the reticle cross-hairs. The EDM is then set up in the erosion position, paraffin oil is placed and the sample chamber is drilled in the centre of the indent.

The position of the electrode in the erosion position is sufficiently reproducible that it is possible, if desired, to use the microscope to check if the hole has been drilled all the way through the indentation then replace the electrode in the erosion position to drill further if it has not.

VI. Results: Erosion speed and accuracy

The machine has been in regular use for a number of years, both for research and for undergraduate projects (see for instance refs. [3][7-9]). To evaluate the speed and accuracy of the erosion we recently ran a series of tests. For these we drilled 10 gaskets indented using a DAC equipped with 250 µm diameter culets. This is the smallest culet size for which the spark eroder is routinely used in gasket preparation; for smaller culets laser cutting is usually used instead. We drilled with a 100 µm diameter bit, resulting in a sample chamber diameter of ca. 125 µm; the largest that would typically be drilled for this culet diameter. These tests therefore represent a hard and rigorous assessment of the machines’ capabilities.

The stainless steel gaskets were indented, and the thickness at the indent on each gasket was measured with a micrometer prior to erosion. The erosion process was conducted using a total capacitance of 0.077 µF in the CDU set using S3 – S6. The process was run for the erosion times specified in table 1. In some cases the initially selected erosion time proved too short following inspection of the gasket using the microscope and the electrode had to be placed back in the machine for further erosion, up to the total erosion time listed in table 1. In one case (gasket B) the
initially selected erosion time was too long resulting in the only gasket in the set drilled that was not usable.

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<tr>
<td>A</td>
<td>29</td>
<td>75</td>
</tr>
<tr>
<td>B</td>
<td>20</td>
<td>60</td>
</tr>
<tr>
<td>C</td>
<td>22</td>
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<td>D</td>
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<td>75</td>
</tr>
<tr>
<td>I</td>
<td>29</td>
<td>75</td>
</tr>
<tr>
<td>J</td>
<td>25</td>
<td>75</td>
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Table 1. Thickness and erosion time (125 µm sample chamber) for 10 gaskets prepared for DACs with 250 µm culet.

Figure 5 shows the sample chambers drilled in these gaskets using the instrument. According to our experience, in 9 out of the 10 gaskets drilled in this test (all except B) the sample chamber is aligned well enough for the gasket to be used in a high pressure experiment. In 6 (gaskets A, C, E, G, H, I) it is well enough aligned to conduct an experiment at pressures up to ca. 60 GPa, the highest pressure that would be routinely attained using 250 µm culets. Erosion speed obtained is 0.33 – 0.49 µms⁻¹. In all cases alignment was performed using a test hole drilled for 60 seconds (stage 3 described in section V), and the photograph shown was taken on the side of the gasket on which alignment and erosion had been performed.

We would like to emphasize that there was no selection procedure for gaskets to be included in the results shown in table 1 and figure 5, on the basis of successful alignment of the sample chamber or any other criterion. 10 gaskets were drilled in succession and all have been included, regardless of the success of the erosion procedure.

Figure 5. Sample chambers drilled using the instrument in gaskets A – J (see table 1). In all cases the total culet diameter is 250 µm, and the sample chamber diameter is ca. 125 µm.

An older version of the instrument (before the automatic stage movement was installed) was used to drill a ca. 60 µm diameter sample chamber in a gasket indented with 100 µm diameter bevelled culets. For this, an STM tip was used as the electrode as described above. Due to the tapered nature of the tip it was necessary to drill some distance from one side of the gasket, then turn the
gasket over, re-align and drill some distance from the other side several times, to ensure the sample chamber drilled had a diameter as constant as possible. A pressure of 165 GPa was reached in the experiment performed using this gasket [7]. Since installing the automatic stage movement, the smallest sample chamber we have attempted to drill is 100 µm diameter, for an experiment using a DAC equipped with 250 µm diameter culet.

The instrument is also regularly used for drilling larger holes, even up to 500 µm diameter (for DACs equipped with 1000 µm culets). In this case the alignment requirements are less rigorous but the erosion time increases significantly due to the increased thickness of gasket to erode as well as the increased diameter. Typically a gasket indented for use with 600 µm diameter culets is 100 µm thick. For these larger gaskets we would switch to a higher capacitance using S3 – 6.

VI. Discussion and Conclusions

We have presented the design of an EDM that allows fast and accurate erosion of the sample chambers for diamond anvil cell experiments. The erosion voltage is 100 V, a relatively low voltage desirable for safety reasons. Our instrument utilizes mechanical components purchased off-the-shelf instead of custom-constructed components for the alignment. The components used are designed for alignment of laser spectroscopy experiments where alignment with ± 1 µm precision is crucial. The optical alignment is performed using a microscope that views the workpiece at 90° to the surface, avoiding any potential errors caused by oblique viewing of the sample.

A custom-designed electronic feedback mechanism is used to automatically move the stage up and down during erosion to continue erosion whilst not crashing the electrode into the workpiece and a capacitor discharge circuit is employed to control the current flow between the electrode and workpiece.

The instrument is regularly used to drill stainless steel and rhenium, for which we use the same drilling parameters. We have not tested it with a tungsten workpiece, but expect that adjustment in parameter such as the drilling polarity may be necessary in this case since the electrode is also made from tungsten.

Our machine differs from existing EDM designs in the literature in several design parameters, the most important of which is the choice of voltage. Other designs have opted for a much lower erosion voltage of 25 kV (Lorenzana et al. [4]), due to the welding problem discussed earlier, or a much higher voltage of 1.2 kV (Pugh et al. [6]). An industrial electric discharge machine designed for broken screw and tap removal has been modified to erode DAC sample chambers at ca. 80 volts [7]. The higher voltage has the advantage described earlier; erosion can take place when the electrode and workpiece are further apart, reducing the risk of them coming into contact. On the other hand, operation at 1.2 kV necessitates stringent safety precautions. We therefore, in this contribution, demonstrate an EDM operating at 100 V as a reasonable compromise. We obtain good erosion results without the potential safety problems resulting from the use of high voltage. Similarly to ref. [6] we implement automatic stage movement in the z direction during erosion, but we use a purely electronic method rather than an optoelectronic method. Our circuit has around half the number of components compared to the instrument described in ref. [6].

The result of our work is a highly accurate instrument which has proved reliable over several years of use both in research and teaching. To quantitatively evaluate the performance of the instrument against existing designs [4][6] it would be necessary to construct machines to all three designs and...
run the exact same tests on all three machines; this is not feasible. However, our machine has
produced reliable results over several years of operation. It has been used extensively for both
teaching and research, including for preparation of some, or all, of the sample chambers for
experiments in a number of publications at pressures up to 165 GPa (for instance refs. [3][8-10]).

VII. Supplementary Material
A complete circuit diagram including manufacturer’s part numbers and component values is
available.

VIII. Acknowledgements
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figures.

IX. References