Producing diamond anvil cell gaskets for ultrahigh-pressure applications using an inexpensive electric discharge machine

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Small holes are drilled in diamond anvil cell gaskets to contain and pressurize samples. As high-pressure technology pushes the multimegabar regime, smaller-tipped diamond anvils are being increasingly utilized. Consequently, well-centered holes with diameters smaller than 100 μm need to be routinely produced in these gaskets made of exceedingly hard metals. We describe the construction of an inexpensive electric discharge machine that can drill metals with holes as small as 25 μm in diameter. This method of drilling is easy to use, far less expensive than other commonly used techniques, and has the advantage of being effective on extremely difficult to machine metals such as rhenium.

I. INTRODUCTION

In recent years there has been a steady increase in the maximum static pressures attainable, from ~35 GPa in the 1970’s up to a maximum reported pressure of 550 GPa in 1986.1-3 These incredible records have been achieved through refinements of diamond-anvil-cell (DAC) technology.2,4 All DACs generate pressures by compressing samples between the faces of two diamond anvils. Generally, a gasket formed out of a hard metal is used to contain the samples on the diamond tip (culet), the region of maximum pressure. A typical gasket is produced by first preindenting a sheet of rhenium to a thickness of 20–50 μm.5 A small hole is then drilled that is centered on the culet imprint in the deformed sheet. A general trend leading to higher pressures has been the use of smaller culet faces in the diamond anvils. For instance, diamonds with culet flats of 50 μm are routinely used in multimegabar experiments, requiring gasket hole diameters as small as 25 μm.5 Complicating the gasket making process are sample stability considerations, specially crucial for soft systems such as H2.6,7 Unstable samples will migrate under load from the center to the edge of the culet, limiting the highest pressures of the experiment and often causing premature failure of the anvils. One key factor to enhancing stability of soft samples is the precise centering of the hole relative to the culet face. We note that making a small (~25 μm) hole within 3 μm of the culet center is extremely difficult and can require several attempts even under ideal circumstances.

Commonly used techniques for drilling small holes have significant drawbacks. The most common method suited for large holes (greater than ~100 μm) is mechanical drilling, but this process becomes increasingly difficult to implement with decreasing hole diameter. A zoom microscope (80× magnification), drill press, and small-diameter tungsten carbide bits are the required equipment. The microscope and the drill press currently cost about $3200 (U.S.). A reasonable supply of bits can also be very costly; for example, 25-μm-diam tungsten carbide bits cost ~$110 each.7 Because of their fragility, one may expect to break about 4 bits in machining one small-diameter (25 μm) hole. Thin shafts also flex markedly during drilling, severely complicating the centering procedure.

Laser drilling is another method for preparing holes but, of course, is dependent on the availability of a high-power laser with appropriate focusing optics. A typical high-power Nd:YAG or CO2 laser used in these applications costs ~$20 000 and a typical optical microscope setup is ~$10 000. Despite its ease of use, this technique is not a viable alternative for many laboratories because of its high initial cost. Also, there are important safety issues related to the use of invisible radiation at extremely high powers.

The third alternative that we discuss here is the electric discharge machine (EDM). The EDM is well suited for cutting arbitrary shapes in metals that are difficult to machine due to hardness and/or brittleness.8,9 The EDM process was described in the 1940’s by the Lazarenko’s,10 and since then numerous refinements have been discussed in the literature with regard to particular applications.11-14 In this process, a tool and a sample of different voltage polarity are brought into close proximity initiating a spark across the gap. With enough available energy, this spark can melt a small portion of the sample. By iterating this erosion step, intricate shapes can be machined into metals. The process is in general slower than either mechanical or laser drilling. Since the process can be applied to either soft or hard metals, the EDM is ideally suited for drilling high-pressure gasket materials. Unfortunately, commercial machines having the required high tolerances needed for diamond anvil cell applications can cost upwards of $10 000. Motivated by a desire to drill out small holes efficiently and inexpensively, we developed an EDM that is well suited for this task and can be easily built in any laboratory.

We have designed and built our system with specific abilities in mind: (1) to drill holes, small or large, which have precisely defined geometries, (2) to center holes with 2–3 μm tolerances reproducibly, and (3) to handle any metal, specifically hard-to-machine metals such as rhenium and inconel. We will describe an apparatus that not only meets these goals but that can also be built for at most $1700. Since many of the needed parts for the EDM are common items in
most laboratories, construction can be as low as $\sim$-$10-20$. Just as important is the EDM’s low cost of use and maintenance, since there is no need for costly drill bits.

II. EDM PROCESS

Spark erosion occurs when a tool discharges current to the sample through a dielectric medium. This process, while easy to visualize, is a complicated process that has been analyzed and modeled in great detail.\textsuperscript{15-21} We now provide the general ideas.

The spark is the essential step in the erosion process. The discharge locally heats both the electrode and the sample, thus melting small portions of material. The choice of dielectric medium plays a key role here. As the discharge passes through the dielectric fluid, a plasma is created that can cause local pressures of several kilobars and temperatures of several $10^9\text{K}$.\textsuperscript{16,17} As the discharge ceases, the collapsing plasma mixes the molten metal and the cool dielectric, driving the metal particulates away from the surface of the sample. Without the proper dielectric, the molten metal welds back onto the surface resulting in inefficient spark erosion.

While realistic simulations of the EDM process require significant computational power, some essential insight can be obtained from simplified, special-case models. We would like to briefly discuss a simple 2D model examined by Carslaw and Jaeger\textsuperscript{22} and extended by DiBitonto et al.\textsuperscript{16} Similar models with varying boundary conditions have also been discussed by Van Dijck and Dutte\textsuperscript{18} and Barrufet et al.\textsuperscript{19} We begin with a half-space of dielectric and a half-space of metal at a temperature $T_0$. A point heat source constant for time $t$ is applied at the interface as shown in Fig. 1. With increasing time, the volume of molten metal increases. This heat conduction problem is governed by the following:

$$\frac{1}{\alpha} \frac{\partial T}{\partial t} = \frac{\partial^2 T}{\partial r^2} + \frac{2}{r} \frac{\partial T}{\partial r},$$

where $\alpha=\kappa_T/(\rho C_P)$, $\kappa_T$, $\rho$, and $C_P$ are the thermal diffusivity, the thermal conductivity, the density, and the heat capacity, respectively. A constant power, $F_c V I$, is assumed during the pulse, where $V$, $I$, and $F_c$ are the voltage, the current and the fraction of the total power ($VI$) which is dissipated at $r=0$.\textsuperscript{16,27} The constraints that we impose on our system vary from those previously reported.\textsuperscript{16} The initial conditions are

$$T=T_0 \quad \forall r, \quad t=0.$$  \hspace{1cm} (2a)

The dielectric is assumed to be a perfect thermal insulator. Therefore, given a heat source at the origin, the following boundary conditions hold:

$$T=T_0 \quad \text{for} \quad r\rightarrow \infty$$

$$-\kappa_T \vec{V} \cdot \hat{n}_z = \frac{\partial}{\partial z} \delta(x) \delta(y) ; \quad t>0$$

at the metal-dielectric interface. \hspace{1cm} (2c)

where $\delta=F_c V I$ and $\hat{n}_z$ is the normal to the metal-dielectric surface. The metal-dielectric interface is defined by the $z=0$ plane. The solution to Eq. (1) is therefore\textsuperscript{22}

$$T=T_0 + \left[ \frac{F_c V I}{2 \pi \kappa_T \tau} \right] \text{erfc} \left( \frac{r}{2 \sqrt{\alpha t}} \right).$$

At the melt radius, the following condition holds:

$$T_M = T_0 + \left[ \frac{F_c V I}{2 \pi \kappa_T \tau_M} \right] \text{erfc} \left( \frac{r_M}{2 \sqrt{\alpha t}} \right).$$

where $T_M$ and $r_M$ are the melting temperature of the metal and the radius of the hemisphere of molten metal, respectively. Equation (4) determines the relation between the volume of molten metal, $V_M=\frac{4}{3}\pi (r_M)^3$, and the input power $VI$. Without solving in detail, one can readily see that as $VI\rightarrow \infty$ then $T_M\rightarrow \infty$, and conversely, as $VI\rightarrow 0$ then $r_M\rightarrow 0$. Clearly, more power equates to a faster cutting rate.

Much more interesting is an analysis determining the optimum parameters for cutting speed.\textsuperscript{16} Because in a real EDM, there is a finite time associated with the discharging and charging of capacitors, times $t$ and $t_{\text{off}}$ exist, when the power pulse is turned on and off, respectively. The cutting rate during one complete cycle $\Delta t_{\text{cycle}}=t+t_{\text{off}}$ is

$$\frac{V_M}{\Delta t_{\text{cycle}}} = \frac{\frac{4}{3}\pi (r_M)^3}{t+t_{\text{off}}}.$$  \hspace{1cm} (5)

The cutting speed can be easily maximized,

$$\frac{\partial(V_M/\Delta t_{\text{cycle}})}{\partial t} = 0$$

yielding the following relation for the maximum cutting rate conditions:

$$\frac{3}{\Delta t_{\text{cycle}}} \frac{dr_M}{dt} \bigg|_{\text{optimum}} = \frac{r_M}{t_{\text{off}}+t_{\text{optimum}}}.$$

Equations (4) and (7) together yield $t_{\text{optimum}}$. The conclusion that we can draw from this simple analysis is that given a current $I$, a voltage $V$, and an off time $t_{\text{off}}$ there exists an optimum time $t_{\text{optimum}}$ for power pulse duration that will provide the maximum cutting rate. Higher end commercial EDMs have the electronic circuitries to take full advantage of this property. In the system described here, $R_2$ (the 5 $\Omega$ resistor) and the capacitor vary the discharge time and have been chosen appropriately.
not only the sample but also the tool, degrading the precision currently, and current pulse frequency. High energies, system can be set up to preferentially erode the sample. Parameters cannot be controlled independently in the device of the machining. Depending on the EDM configuration, the current, and the polarity of the tool. Some of these described here. Since high-pressure applications require surface finishes to be no more than a few µm rms, appropriate parameter ranges have to be selected empirically.

The EDM action involves other complicated processes making realistic analysis difficult. For example, the following need to be taken into account: a mixed system of plasma, liquid and solid components, phase transitions, chemical reactions, frequency-dependent conductivity effects, and others.

Lastly, the precision of the machining is extremely important and also depends on the configuration and parameters of the EDM. Among them are electrode-sample gap, voltage, current, and current pulse frequency. High energies, for example, will melt relatively large amounts of material leaving the surface finish rough. The discharge tends to erode not only the sample but also the tool, degrading the precision of the machining. Depending on the EDM configuration, the system can be set up to preferentially erode the sample. Parameters that control the preferential erosion of the sample include the composition of the electrode, the pulse rate of the current, and the polarity of the tool. Some of these parameters cannot be controlled independently in the device described here. Since high-pressure applications require surface finishes to be no more than a few µm rms, appropriate parameter ranges have to be selected empirically.

III. APPARATUS

We now describe a system that (1) has a range of parameters that permits reasonable spark erosion and (2) allows precise manipulation of the tool and sample. A brief description of the circuit is as follows and is drawn in Fig. 2(a). A variable low-voltage alternating current power supply (e.g., a variac or a commercial low-voltage ac power supply) is connected to a high current diode resulting in a quasidirect current which charges an electrolytic capacitor. The capacitor is the charge reservoir that supplies the high power during the discharge process, and the $R_2$ (5 Ω) resistor controls the discharge rate of the capacitor. The $R_1$ (160 Ω) resistor simply acts as a high-wattage current limiter in case the tool and the sample short circuit. An elegant alternative to $R_1$ is a common 40 W light bulb. An optional voltmeter alerts the user when the tool and sample have touched.

Figure 2(b) presents a schematic of the mechanical portion of the system. Commercially available small diameter wire is used as the cutting tool and is connected to the negative terminal of the circuit. The tool is mounted on an electrically insulated X-Y-Z translation stage (~$500). The stage allows µm-size movements of the cutting tool with respect to the gasket. The gasket is held in place by a small metal mount, which is connected to the positive side of the circuit. The entire assembly is then viewed through a stereo zoom microscope (80× magnification and ~$1200) and the cutting tool is moved into position. The translator and the zoom microscope constitute almost the entire price of the apparatus described here. Drilling a hole consists simply of placing a drop of dielectric in the gasket indentation and then bringing the bit close enough to the gasket for a spark to jump across the gap. The tool is manually moved up and down µm's at a time to allow the capacitor to recharge, to induce sparking, and to clear the gasket tool gap of interfering floating particulates. After initial centering, the gasket remains centered relative to the bit since the tool and gasket do not make physical contact. Thus, in contrast to mechanical drilling, flexing of the bit is not a source of error, an important consideration for the smaller diameter holes. This allows holes to be centered to within the precision of the optical system and translation stages.

IV. RESULTS AND DISCUSSION

Choosing the appropriate cutting tool material is important. As mentioned earlier, both the tool and the sample erode during the process. One way of forcing preferential erosion in the sample (conversely, minimizing tool erosion) is to choose a high-melting-point metal for the tool, such as tungsten. Tungsten wire can be bought in many diameters, 10 µm and greater at very modest costs. In addition, the wire is flexible and robust enough not to deform permanently in the event that the tool touches the gasket surface (which is often). The wire electrode may be used many times before replacement is necessary. It is also possible to maintain a stock of only the largest diameter wire and make tips of varying diameters from this wire. Polarity of the tool can affect the erosion rate of both the tool and sample, but we found little effect in our system.

For a given electrical configuration as in Fig. 2, the diameter of the hole drilled is related not only to the diameter of the cutting tool but also to the power $VI$, as discussed earlier. Higher voltages reduce cutting times but generate holes that are larger than the diameter of the tool and degrade the quality of the drilled surface. An additional disadvantage
of high voltages (≥50 V) is that the tool and sample tend to weld together frequently if they make contact. This rarely happens at the lower operating voltages (25–30 V). Typical settings used to drill a ~40 μm hole in a rhenium gasket preindenteted to 25 μm thickness requires a 25 μm tungsten electrode, with a 25–30 V potential difference. Under these conditions it takes ~5–10 min to mount the gasket, align the tool, and drill through the rhenium. These settings need to be modified slightly for the smaller diameter electrodes because the power discharged from the capacitor is sufficient to melt the tungsten electrode. For 20-μm-diam electrodes or smaller, we found that a 5 μF capacitor and 45 V source yielded optimum results (Fig. 3). We have successfully tested numerous gaskets with soft samples such as N₂ to as high a pressure as 136 GPa.

The EDM process is to a certain extent insensitive to the exact configuration of the system. A wide range of resistor and capacitor combinations yielded adequate results. For large holes of greater than 500 μm diameter, surface finish may not be so important. Consequently, one can significantly accelerate the process by increasing the capacitance (or voltage). The choice of dielectric has also been found to have little effect on the quality of the cuts provided the viscosity of the fluid was low enough. For high viscosity dielectrics, clear the sample-tool gap of metal and other particulates becomes extremely inefficient causing undesirable arcing and a rough surface finish. In addition to using commercially available dielectrics that are specifically sold for EDM applications, we have successfully used other kinds of fluids including vacuum pump oil, machine oil, kerosene, common organic solvents (e.g., methanol and ethanol), and vegetable cooking oil. Water and methanol were of limited success and are not recommended. Other dielectrics such as kerosene have high vapor pressures and toxicity considerations are important. Noteworthy was vegetable oil which yielded reasonable results and is nontoxic.

The EDM described here can be made from the surplus parts found in many laboratories and has been shown to be capable of drilling holes down to 25 μm. While we have not attempted to do so, we believe that with thinner diameter wires and lower-power settings, this system can certainly produce smaller holes. About 5–10 min are required to drill a well-centered hole through a standard rhenium gasket. Lastly, we emphasize that this technique consistently yields well-centered gaskets in the first attempt even for the smallest diameter holes.

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4 Other hard but ductile gasket materials are commonly used such as steel, stainless-steel T301, Inconel, and so forth. See A. Jayaraman, Rev. Mod. Phys. 55, 65 (1983).
7 The National Jet Co., LaVale, Maryland.
8 "Thermal Machining Processes" (Society of Manufacturing Engineers, Dearborn, Michigan, 1979).