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# Resistance heating of the gasket in a gem-anvil high pressure cell

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Resistance heating of the gasket strip in a gem-anvil high pressure cell was successful in obtaining sample temperatures up to 1100 °C, under pressures up to 4.0 GPa. The heating capabilities, as well as the mechanical and chemical stability, of several different gasket strips (two Ni-based alloys, Ta, Pt/Rh, and a Re/Mo alloy) with different design shapes, and two different single-crystal anvil materials (diamond and cubic zirconia) were investigated. Two gasket-strip designs were found to provide optimum uniform heating conditions while decreasing the required current needed to achieve 1100 °C. Two anvil systems were investigated to reduce the temperature increase of the pressure cell body. Cubic zirconia anvils reduced the cell-body temperature to 100 °C at sample temperatures up to 1100 °C. However, zirconia anvils often failed during heating and almost always failed during cooling. Diamond anvils with cubic zirconia mounting plates also permitted temperatures up to 1100 °C to be reached without anvil failure. However, the cell-body temperature increased to 300 °C. A sealed vacuum-type chamber was employed to eliminate the problem with gasket and anvil oxidation. The optimized operating parameters reported here provide a routine method for high temperature-high pressure studies. The method was used to densify and sinter nanosize amorphous silicon nitride and $\gamma$-alumina powders at high temperatures and high pressures. [S0034-6748(99)03610-2]

## I. INTRODUCTION

The diamond anvil cell (DAC) and the ruby fluorescence pressure measurement technique enable high pressure studies in several areas of scientific and technological interests. One main advantage of this apparatus, besides the ability to generate extremely high pressures, is the optical window that permits *in situ* analyses of samples by ultraviolet visible spectroscopy, infrared spectroscopy, Raman spectroscopy, x-ray diffraction, etc. An up-to-date list of references pertaining to these applications of the DAC can be found in Ref. 1. Parallel to the development of new materials and methods for DAC application, high temperature-high pressure (HT–HP) experiments have been carried out.[1] However, the relatively low thermal stability of diamond in air and its high thermal conductivity, make it difficult to sustain static high temperature conditions in the 1000 °C regime.

Thus, any method for simultaneous HT–HP conditions in the DAC must take into account limitations caused by material selection (anvils and high pressure cell components). Diamonds heated in air will begin to oxidize at approximately 800 °C, and at 1500 °C will begin to graphitize.[2] Also, under high load conditions and high temperatures, anvil-gasket welding can occur. Significant reductions in the strength and hardness of all metal components including the gasket will occur when the cell temperature exceeds the annealing points. This could lead to sample-pressure loss and possible anvil failure for two reasons: (1) the anvils could nonuniformly indent their softened metal supporting surfaces causing misalignment of the anvil faces; and (2) the softened gasket could flow radially unsymmetrically leading to a possible rupture of the gasket. Finally, the pressure cell metal components as well as the gasket are subject to surface corrosion at high temperatures, and, in the case of metal components with close tolerances, this corrosion often results in the binding of moving parts.

There are three primary heating methods for anvil-type pressure cells found in the literature. Each method takes into account the earlier mentioned limitations. The most common method uses a cylindrical resistance coil heating furnace positioned either internally around the anvils or around the entire anvil-piston assembly.[3–5] This heating method was the first developed for high pressure cells and is limited to routine temperatures around 500 °C with occasional excursions to 600 °C.[1] The method is practical for the study of materials at temperatures below 500 °C. However, it is not recommended for applications at higher temperatures, since the anvils and several metallic cell components, including the piston-anvil assembly, are directly subjected to the high temperatures.

The second method consists of heating the entire pressure cell in a high temperature furnace under a controlled inert environment to protect against oxidation of the diamond anvils and the metal cell components. This method has been applied to many different anvil cell designs with a routine temperature of 500–600 °C.[1] The maximum temperature

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achieved by this method has been accomplished with a DAC fabricated from a rhenium-molybdenum alloy. Such diamond cells permit heating excursions up to 900 °C.[6] However, the prohibitively high cost of the rare alloy required for the cell fabrication has discouraged its wide use.

The third method utilizes laser heating techniques to generate high sample temperatures. A variety of different laser systems have been used with this technique. The most common laser systems used for heating pressure cells are a $CO_2$ laser and Nd:yttrium–aluminum–garnet laser. Two sample heating methods are used. The sample is either heated through a single anvil or the laser beam is split and the sample is heated through both anvils. Both methods are employed to produce reported temperatures above 2000 °C.[7] The main advantage of this technique is that the cell body itself remains at room temperature. The heat is localized at the sample and is dissipated by rapid thermal diffusion through the diamond anvils into the large cell body mass. However, a large thermal gradient across the sample occurs due to the small laser spot size and high thermal conductivity of the diamond anvils, and maintaining a uniform high temperature is virtually impossible. One final difficulty encountered with this technique is that transparent samples must be coated or doped in order to increase the absorption of the laser energy to achieve high temperatures.

As described earlier, the temperatures below 500 °C and above 2000 °C are routinely achieved in a DAC. However, the temperature regime between 600 and 2000 °C is much more difficult to achieve routinely and still presents a major challenge for DAC design.[2] The pressure and temperature regime up to 10.0 GPa and $<$2000 °C is important for industrial applications, i.e., where studies concerning phase diagrams, crystallographic structures, compressibility, sintering, etc., need to be carried out. Materials processing represents a recent and exciting area of high pressure research and also requires the application of simultaneous HT–HP conditions.[8–10]

This article describes the development and implementation of a modified gem-anvil high pressure cell capable of statically heating gasketed samples to 1100 °C at pressures up to 4.0 GPa. The temperature is achieved through the resistance heating of a metallic strip simultaneously used as the gasket in a DAC-type pressure cell. This method of heating a diamond pressure cell was first reported in 1970 by Moore, Sorensen, and DeVries.[11] However, in that report only Pt metal was used as the gasket material; no other gasket metals were investigated. The authors also stated that it may be difficult to reach temperatures in the 300 °C range by their technique owing to the high thermal conductivity of the diamonds. The method described in the present report represents a significant extension of this earlier work by Moore, Sorensen, and DeVries.[11]

Other internal resistance heating techniques have been reported. For example, to study the phase diagram of iron at elevated temperatures and pressures, L. Lui and W. A. Bassett[12] heated a fine iron wire in a mixture of alumina, periclase and Forsterite pressed between two opposed diamond anvils by passing a direct current through it. Building on that original idea, Boehler, Nicol, and Johnson,[13] described a resistance heating technique for the diamond cell which utilized a sandwiched stainless steel gasket arrangement (insulated from each other by an alumina cement), in conjunction with an iron wire as the heating element and also as the sample. Alumina was used as the pressure transmitting medium. The technique was used to determine the phase diagram and P–V–T data of iron. Mao, Bell, and Hadidiacos[14] described a similar, but not identical technique, to study polymorphism in iron at high pressures and elevated temperatures. Dubrovinsky, Saxena, and Lazor[15] obtained x-ray diffraction data on iron at elevated pressures and temperatures utilizing a DAC internal heating device. Adopting the basic ideas presented in Ref. 13, these authors sandwiched a thin iron foil between two gaskets using argon or corundum as a pressure transmitting medium. Variations of the original technique, with added refinements and improvements, have been used successfully in x-ray diffraction studies of the melting point and phase transitions of iron.[15,16] Although these methods are specific to the study of iron, other metals could be substituted and studied in place of iron provided they exhibited appropriately high resistivities. A recent publication by LeToullec *et al.*,[17] describes a heating technique which uses two miniature resistance coil (Kantal) furnaces in series with a Re metal gasket strip. The two miniature furnaces supply heat to the Re gasket and sample mainly by thermal conduction because of the very low resistivity and high thermal conductivity of the Re. Our heating technique described in this report is based on purely ohmic resistive heating of the gasket strip and no miniature furnaces are required. Also, our method can be used routinely without the need for sophisticated ancillary equipment. The application of this modified gem-anvil cell is demonstrated with the sintering of nanosize particles (silicon nitride and $\gamma$ alumina).

## II. DEVELOPMENT OF THE EXPERIMENTAL EQUIPMENT

### A. Gasket strip: material and design

The gasket strip is employed both as the sample confining material to maintain a uniform pressure, and as the ohmic heating element. The strip dimensions are important to both purposes. For optimum sample heating, the width and thickness of the central part of the strip containing the sample, pressed between the opposed anvil faces, should be minimized to maximize electrical resistance (ohmic heating). However, to minimize gasket distortion and possible gasket-wall rupture, the central part of the strip should be dimensionally maximized to support the necessary loads and to provide a practical sample thickness. Thus, an optimized compromise between these two extremes had to be found.

Several gasket heating element designs were investigated. Initially, a gasket strip of uniform width was used. Although this design localized heating at the center of the gasket strip, it required very high electrical currents and produced significant temperature rises in the gasket mounting interfaces. Therefore, modifications, of the gasket strip shape were attempted to focus the resistance increase near the center of the strip where the gasket hole is located. From several

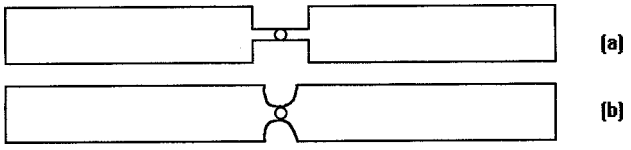


FIG. 1. Schematic diagrams of two gasket strip heating elements that were developed for heating the sample in the pressure cell.

shapes investigated, two that performed well are shown in Fig. 1. Both designs maximized the localized heating of the gasket strip center in the vicinity of the gasket hole while reducing the temperature increase at the mounting interface. The two designs differ mainly in the length of the narrow portion of the strip. A gasket hole is drilled in the center of the narrow portion of the strip to permit sample confinement. The two designs shown in Fig. 1 provided the most uniform and controllable heating of all strip shapes investigated.

Besides the strip shape, the material's mechanical stability under HT–HP conditions is critical to the gasket performance. Mechanical properties, such as high yield strength and cold working hardenability as well as low creep rates at high temperatures, are important. Resistance to oxidation at high temperatures and to anvil-gasket welding are also critical. Therefore, a number of metals and alloys were tested for optimized ohmic heating, with reduced oxidation properties. Rhenium and rhenium/molybdenum alloys are excellent metals for use as gaskets at HT–HP.[6,18] These materials are reported to eliminate anvil-gasket welding, while they retain their mechanical properties. However, rhenium and its molybdenum alloys are not readily available and, therefore, other gasket materials were also considered. The different refractory metals and alloys tested were Inconel 600, Inconel X750, platinum/rhodium 60/40, rhenium/molybdenum alloy 47.5/52.5, and tantalum. The gasket thickness for all materials was $254\pm3$ $\mu$m prior to indentation. For Inconel X750, gaskets $381\pm3$-$\mu$m-thick were also investigated.

Initially, the heating tests were made outside the pressure cell. The gasket strips were mounted at opposite ends by screws attached to two ceramic towers on an insulator plate. The electrical terminals of the power supply were connected to the gasket by the same screws. The current and temperature were monitored, while the voltage was manually increased in steps of 0.1 V.

The power supply used to heat the strips was a Power Ten Inc. 2500 W direct current (dc) power supply, designed specifically for systems requiring a variable dc source with good ripple and regulation characteristics. It features constant current/constant voltage operations with automatic crossover and provides a wide range of voltage and current levels, i.e., (0–7.5) V and (0–300) A dc with a 10 mV ripple. The current and voltage were measured using the meters provided on the power supply.

The temperature was measured by a type K thermocouple (wire diam 0.254 mm) in physical contact with the high resistance region of the gasket. Figure 2 shows the temperature increase as a function of the electrical current for all materials tested and for both designs shown in Fig. 1. The much higher electrical resistivity of the Inconel alloys compared with tantalum and platinum/rhodium translated into a

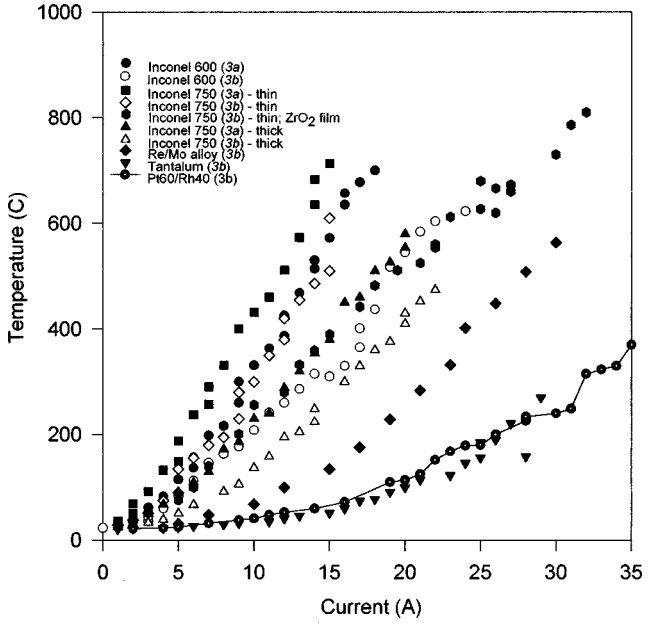


FIG. 2. Temperature increase at the center of the strip as a function of the electrical current for different metallic strips and different designs, tested outside the pressure cell. In the legends, (a) and (b) are the designs of Fig. 1. In the case of Inconel X750, the designations *thin* and *thick* correspond to thickness of 254 and 381 $\mu$m, respectively.

significantly higher temperature for a given electrical current. The rhenium/molybdenum alloy results were intermediate for all gasket materials studied. The highest temperatures were attained with the gasket design shown in Fig. 1(a), with the maximum temperature for any given current achieved using Inconel X750. However, temperature measurements on gasket strips of the design shown in Fig. 1(b) typically gave a more uniform and stable high temperature zone (as evidenced by uniform color) because its shape provided a larger mass of alloy metal in the region of the hot zone.

### B. Anvils

The behavior of alternative gem anvils for HT–HP studies has been investigated. J. Xu and E. Huang[19] previously reported a similar resistance heating technique using yttria stabilized cubic zirconia anvils in a Mao–Bell-type pressure cell. During that work a sample of graphite was pressurized to 6.0 GPa and heated. Raman spectroscopy of the recovered sample showed that the graphite transformed to diamond, indicating that the temperature achieved was in excess of 1000 °C, the temperature necessary to observe the graphite-diamond phase transition at 6.0 GPa.[20] No *in situ* temperature measurements were reported and the actual temperature is only inferred by the observed phase transition. Furthermore, despite the experimental success of achieving 1000 °C at 6.0 GPa, there was no description of the experimental details of the method used, and no subsequent published work was found concerning the applicability of their method to routinely generate HT–HP conditions in the cubic zirconia anvil cell.

In the present work, the HT–HP studies using a cubic zirconia anvil cell were done with samples of amorphous nanosize silicon nitride powder, processed using $254\pm3$-$\mu$m-

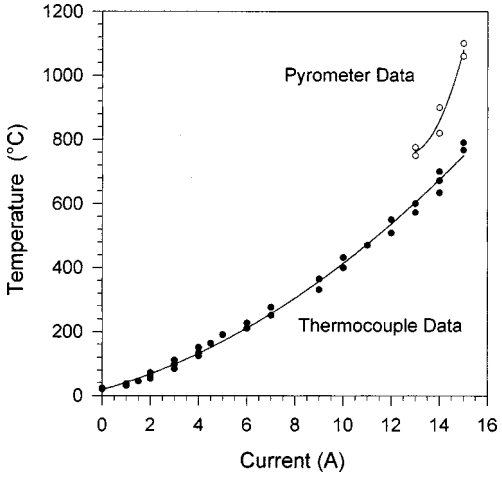


FIG. 3. Temperature of silicon nitride sample as a function of the electric current applied to the strip in a cubic zirconia anvil pressure cell.

thick Inconel X750 gasket strip with the design shown in Fig. 1(a). The samples were loaded to pressures between 0.5–2.0 GPa and heating and cooling rates as low as 5 °C/min and as high as 20 °C/min were used. The advantages of using cubic zirconia anvils were observed in the low power required to heat the gasket strip to high temperatures and in the low temperature rise in the cell components. Figure 3 shows the obtained thermocouple and pyrometer measurements of a silicon nitride sample pressed to 0.5 GPa and heated using the gasket strip heater. A maximum sample temperature of 1100 °C was achieved while the cell temperature never exceeded 100 °C, indicating that the low thermal conductivity of the cubic zirconia anvils relative to diamond anvils is effective in isolating sample heating from the cell body. However, besides this clear benefit of zirconia anvils, a number of experimental difficulties were encountered during HT–HP runs. For example, slow heating rates were required to achieve maximum temperature without anvil failure. Furthermore, the anvils were never recovered from a high temperature experiment because they always cracked during cooling. Finally, when attempts to retrieve the sample were made at the end of the experiment, one or often both of the anvils separated near their culet tip with some zirconia material adhering both to the sample and gasket indent wall. Despite the minimal success accomplished using cubic zirconia anvils for HT–HP studies, their use as a thermal barrier was the basis of the development of the current HT–HP design.

The second anvil design involved a sandwich of diamond anvil and cubic zirconia plates (Fig. 4). In this design the diamond anvils are mounted on cubic zirconia plates that act as thermal insulators to reduce the heat loss to the metallic pressure cell components. To further reduce heat loss, the cubic zirconia plates and the diamond anvils were fastened in place utilizing a ceramic cement (AREMCO 552). Using this configuration, typical cell component temperatures were maintained at approximately 300 °C when the sample temperature was higher than 1000 °C. These results indicate that this configuration provides adequate thermal isolation of the diamond anvils from the metal cell components, eliminating the oxidation and metal softening problems often associated with high temperature studies.

The diamond anvil mounted over cubic zirconia plates was tested under pressure to investigate anvil alignment and stress distribution. Distilled water was compressed and routine pressures of 4.0 GPa were obtained with the diamond/cubic zirconia configuration, without failure of the plates and anvils. HT–HP compaction experiments were also conducted using nanosize silicon nitride and $\gamma$ alumina to determine the viability of the diamond/cubic zirconia configuration. These samples were also compacted routinely under HT–HP conditions (1100 °C and 4.0 GPa) without degradation of the cell components and anvil failure.

### C. High temperature pressure cell

#### *1. Gasket heating*

The National Institute of Standards and Technology (NIST)-type high pressure cell needed to be modified to permit the insertion of the gasket heating system. In addition, the cell needed to be electrically isolated from the metal gasket and all electrical components of the circuit. This was accomplished by modifying the basic diamond anvil cell design that utilizes a resistance coil heater. The main modifi-

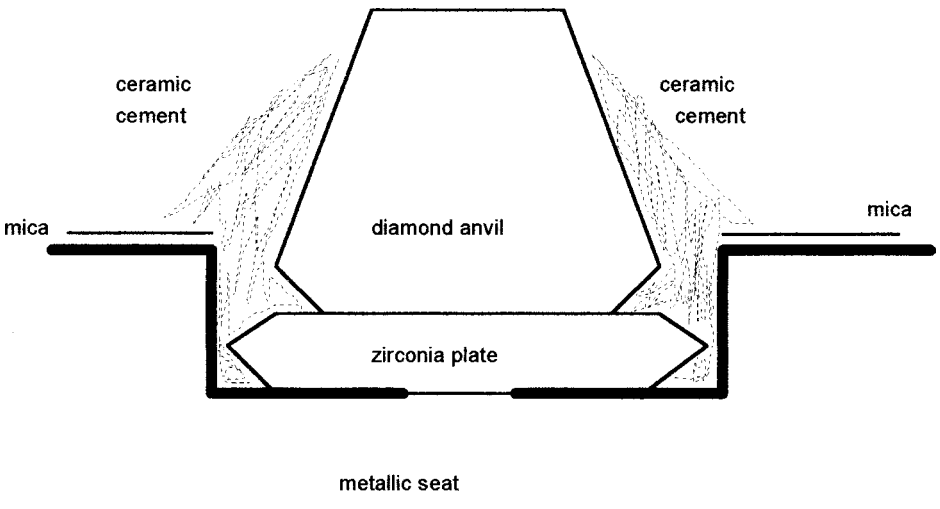


FIG. 4. Schematic view of the mount configuration with the cubic zirconia plate at the bottom of the diamond anvil.

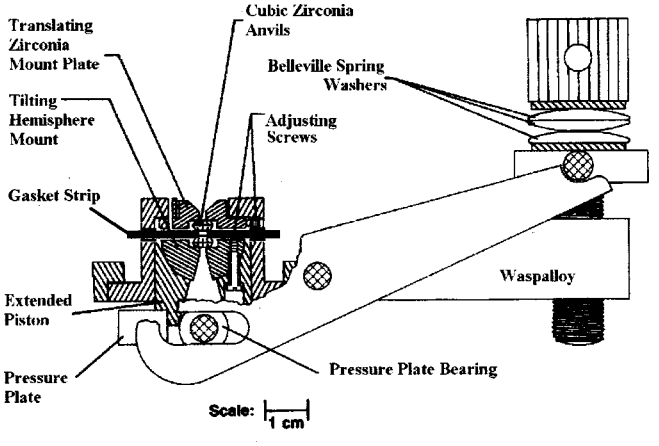


FIG. 5. A schematic diagram of the pressure cell used for heating the sample. The cell is less massive than the original design and there is a cylindrical cavity around the piston/anvil assembly to accommodate an insert that contains the electrical terminals for the gasket.

cations are depicted in Fig. 5. Two small orifices, 180° apart through the cylinder wall, provide exit/entrance slots for the gasket.

The cylindrical cavity designed to accommodate a cylindrical resistance coil heater, is used in the present design to accept an electrical and thermal insulator insert. Figure 6 shows a schematic of the press-fit cylindrical insert. Three mounting screws are used to simultaneously mount the gasket and the power supply terminal posts. The insert is made of a high temperature machinable ceramic (MACOR). MACOR is an excellent thermal insulator which significantly reduces the amount of thermal energy transferred from the gasket strip to the surroundings. Furthermore, MACOR is also a good electrical insulator. Thus, the insert effectively isolated the gasket from the pressure cell cylinder and body. The rectangular grooves in the ceramic insert are positioned on axis with the two orifices. The interior wall surfaces of the two orifices were coated with a durable ceramic adhesive (AREMCO Cerama-Dip 538) to electrically insulate the gasket from the metallic cylinder wall. Furthermore, the gasket was electrically isolated from the metal surfaces of the translating plate and rocking hemisphere, by using circular cut outs of high quality mica sheet fastened in place with ceramic cement (AREMCO 552). The gasket was fastened in position by two copper blocks, mounted in the ceramic insert

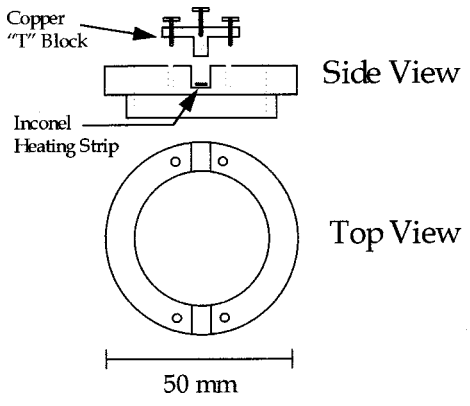


FIG. 6. Circular insert for terminal mounts and power supply hook up.

(Fig. 6). The copper block are ''*T*'' shaped and were fastened with two brass screws which thread into helicoil inserts in the ceramic piece that pressed the block against the heating strip. The copper block has two primary functions: (1) to make electrical contact with the gasket, and (2) to fix the gasket position to maintain its alignment with the anvils. A central brass screw provided the fastening post for the power supply input wires in the copper block. For even better electrical contact, the gasket ends were folded over and fastened to the copper block by the terminal screws. The insert ring, the AREMCO Cerama-Dip 538 adhesive and the mica sheets completely isolate the gasket from all metallic cell components, while providing a highly efficient circuit so that most of the thermal energy is localized to the central portion of the gasket. This permitted the application of large currents (40–50 A) for ohmic gasket heating.

### 2. Temperature measurements

The article by LeToullec *et al.*[17] utilized a novel fluorescence method for internal measurement of temperature while simultaneously measuring pressure utilizing two sources of fluorescence, ruby and $SrB_4O_7:Sm^{2+}$. The pressure is determined from the $SrB_4O_7:Sm^{2+}$ shift, which, to a first approximation, is independent of temperature. The temperature is extracted from the ruby $R_1$ line shift that is composed of a combination of both pressure and temperature shifts. In the original article describing the particular $SrB_4O_7:Sm^{2+}$ fluorescence method of concern here, Datchi, Letoullec, and Laubeyre[21] point out that the technique is useful for measuring **pressures** only below 900 K (627 °C) because of a drastic fluorescence intensity decrease with increasing temperature. The ruby R lines have a similar problem with intensity decrease, in addition to a coalescence of the two initially resolved $R_1$ and $R_2$ peaks, but these problems initiate at somewhat lower temperatures. They start to become significant at around 300 °C, possibly 350 °C. Certainly at 500 °C, the two peaks are completely unresolved and the temperature component to the shift as well as the pressure component are only very rough approximations. In any case, use of this novel fluorescence technique, which permits extracting the temperature component from the ruby R-line shifts is limited, at most to 450–500 °C and, therefore, is of no value for the temperature range of concern in our experiments.

From our own research experience with anvil-type pressure cells, we are aware of the importance of internal sensors, not only for temperature measurement, but also for pressure measurement, where the problem was first identified with the introduction of the ruby gauge (an internal sensor). In our gasket-heating method, we chose to measure temperature by utilizing an optical micropyrometer, specifically an instrument enhanced for temperature measurement in the 1000 °C range and also calibrated for that range, in conjunction with a standard thermocouple technique. Utilizing these techniques we have obtained both a temperature measurement for the gasket (external to the sample) and a temperature measurement for the sample (internal-spectral emission from mainly the sample) and have attempted to evaluate the observed differences between them. The temperature differ-

ence increases with increasing temperature as illustrated in Fig. 3, an observation we anticipated based on our earlier work with thermocouples.

In our experiments, the bead of a type K thermocouple entering through one cylinder wall orifice was positioned in contact with one lateral facet of the diamond anvil. It is assumed that the measured diamond anvil temperature provided an accurate measure of the average sample temperature, due to the high thermal conductivity of diamond. Inside the cell, the thermocouple wires were covered with adhesive cement (AREMCO 552) to hold them in place over the mica sheet and to isolate them from the heating strip. Inside the cylinder wall orifice, a small mica sheet was used to isolate the thermocouple wires from the gasket strip. The uncertainty in the temperature measurement is estimated to be $\pm 50$ at 1000 °C. A second type K thermocouple was placed outside the cell, inside an external hole in the cylinder, to measure the temperature of the anvil mounting assembly. This provided a measurement of how much heat was conducted away from the hot zone.

A micro-optical pyrometer with enhanced sensitivity in the 700–1200 °C range was also used to measure the sample temperature in the higher end of the temperature range studied. The sample and the gasket strip surrounding the sample were imaged through an objective lens with a 147 mm focal length adapted to the pyrometer. The uncertainty in the measured pyrometer temperature is estimated to be $\pm 50$ °C at 1000 °C. In the low temperature range of the pyrometer (700 °C), the temperature measurements were less accurate when diamond anvils were used because the transmission efficiency of diamond is relatively low and reduces the intensity of the radiation detected by the pyrometer. This reduction in intensity is compounded by the low emissivity of the gasket strip in this temperature range. Thus, the uncertainty in the temperature measurement is estimated to be $\pm 75$ °C at 700 °C.

#### 3. Pressure measurements

The ruby fluorescence technique was used to measure the pressure inside the cell using a microscope system described earlier.[22] The pressure measurements have a standard uncertainty of $\pm 0.05$ GPa when made in a hydrostatic environment at room temperature. Line broadening and reduced intensity of the R lines at elevated temperatures contribute to an increase in the pressure measurement uncertainty which is approximately $\pm 0.1$ GPa at 300 °C. The standard uncertainty in the pressure measurement in the absence of strictly hydrostatic conditions is estimated to be $\pm 0.15$ GPa. Pressures were measured before and after sample heating. No pressure measurements were made at elevated temperatures because of significant loss in fluorescence intensity and the coalescence of the two R-line peaks. Sample pressures after heat treatment were typically higher by about 1.0 GPa.

### D. Isolation chamber

A special isolation chamber was designed and constructed to run the experiments in a controlled inert atmosphere to minimize oxidation of the diamond anvils, metallic gasket, and the other metal cell components at high temperatures. The isolation chamber consists of three main parts, as shown in a simplified schematic in Fig. 7: a stainless steel alloy circular table, a glass cylinder body, and a transparent Plexiglas circular cover. O-ring seals are used at the table/cylinder and at the cover/cylinder body interfaces to seal the chamber. Swagelok-type connectors are used in the stainless steel table for feedthrough lines for gas input and exhaust. Conax connectors were used for electrical feed throughs and special thermocouple feedthrough were also used (for details, see Fig. 7 caption).

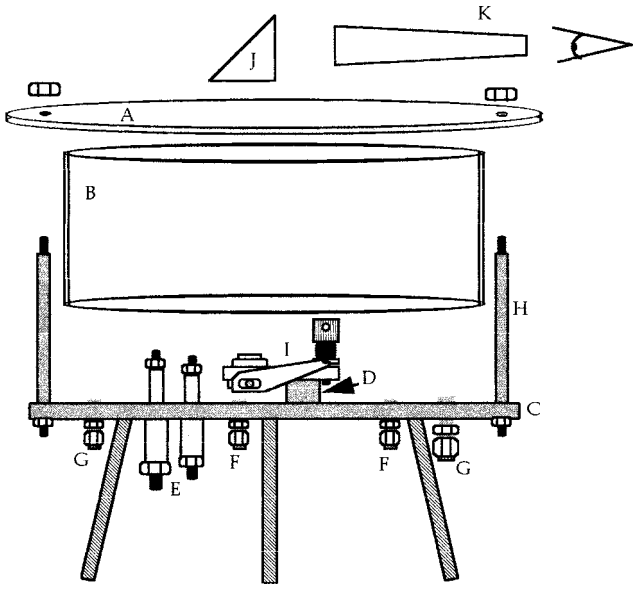


FIG. 7. Schematic view of the heating chamber. (a) Plexiglas cover; (b) glass cylinder, (c) stainless steel table, (d) stainless steel block welded to the table to hold the pressure cell using two threaded holes, (e) Conax feedthrough fittings used to make the electrical contact between the power supply, from outside, and the gasket strip in the pressure cell, (f) two Conax feedthrough fittings for the thermocouple wires, (g) two Swagelok-type feedthrough for input and exhaust gas flow, (h) three threaded rods used for sealing the chamber, (i) high pressure diamond cell, (j) 45° viewing mirror, and (k) optical pyrometer

The temperature measurement made with the pyrometer through the Plexiglas top slightly underestimates the temperature because the Plexiglas window can absorb some of the visible radiation emitted by the hot sample. A similar effect was observed when a glass window was used. However, based on calibration measurements using both window materials, the relative standard uncertainty in the temperature measurement is increased by less than 5% in the range of 800–1200 °C, and is considered negligible for the experiments of concern here.

## III. RESULTS AND DISCUSSION

### A. Performance of the cell design

The cell configuration used here permitted temperatures up to 1100 °C and pressures up to 4.0 GPa to be reached. In earlier experiments conducted in air at this temperature, the diamond anvils were slightly etched and the gaskets uniformly oxidized on that part of the strip located inside the pressure cell up to the region of the copper blocks. In some experiments, the Inconel gaskets welded to the diamond an-

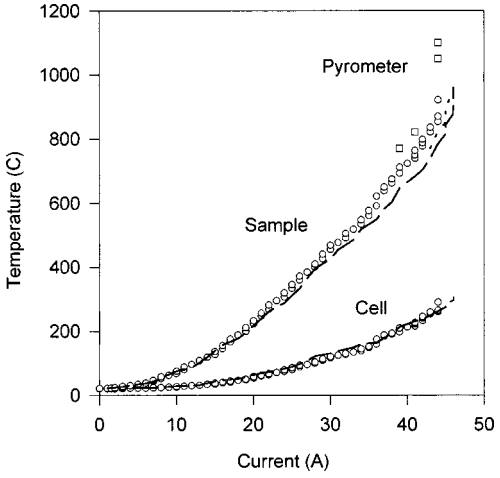


FIG. 8. Temperature of $\gamma$-alumina sample and cell body as a function of the electric current applied to the strip. The open circles correspond to the case where a thin layer of sample covered both anvil surfaces, the dashed lines correspond to the case where the strip was coated with a thin film of yttria-stabilized cubic zirconia, about 19 $\mu$m thick. Open squares are the pyrometer measurements with the dotted lines referring to the case where the strip was coated with the thin film of zirconia.

vils surfaces. To minimize this welding process, a thin layer of precompacted sample was pressed between the gasket and the diamonds. This layer also inhibited the etching of the diamond anvil, minimized sample contamination from the gasket and acted as a thermal barrier between the gasket and the diamond. A test with an Inconel gasket coated on both sides with a 19-$\mu$m-thick cubic zirconia film was not successful in minimizing the gasket oxidation because the film delaminated from the strip during pressing and exposed the metal surface to the atmosphere.

The cubic zirconia plates cracked after the first run, probably due to thermal expansion stresses. Since the plates were fastened between the diamond anvil and the cell surface with ceramic cement, the cracks did not prevent their subsequent use. The cracked zirconia plates were used repeatedly for about ten experiments without any degradation of the pressure performance of the cell. If necessary the cubic zirconia plates could be replaced on a routine basis.

The high temperature limit was imposed by the gaskets, which exhibited creep (time-dependent plasticity characteristic) and oxidized in the central region or hot zone. When the temperature was $\geqslant$1100 °C, the loss in strength of the Inconel X750 gasket resulted in severe gasket thinning and tearing in the region between the diamond surfaces, breaking up electrical continuity. This upper limit on the useful temperature range was consistently observed in all of the high temperature experiments.

Diamond and gasket oxidation were minimized when the cell was heated inside a controlled inert atmosphere of argon, using the isolation chamber depicted in Fig. 7. The temperature limit was also slightly increased under such conditions. The increase in temperature is most likely due to the reduced gasket oxidation during sample heat up. Gasket oxidation resulted in the cracking of the metal strip and the creation of a large amount of porosity, both having the effect of reducing the ohmic heating property of the gasket. Therefore, the gasket is still the limiting parameter of the maximum temperature achieved, even when the oxidation problem is minimized.

### B. Processing experiments

The newly developed technique was used to hot press nanosize particles of $\gamma$ alumina and amorphous silicon nitride nanosize powders at temperatures up to 1000–1100 °C and pressures of 1.5–3.0 GPa. The gasket strips used for these experiments were made of Inconel X750 (254 $\mu$m thick) and shaped as in Fig. 1(b). Figure 8 shows the sample and cell temperature rise for a precompacted $\gamma$-alumina sample where the voltage was increased in steps of 0.05 V at intervals of about 30 s. The pyrometer reading indicated a higher temperature than the internal thermocouple. The final sample temperature at 4 V and 44 A was between 1000 and 1100 °C according to the pyrometer, while the cell body remained at a relatively low temperature (300 °C). The recovered samples were generally very thin (30–50 $\mu$m) and had very smooth mirror-like surfaces. These results demonstrate that the technique can be used successfully to hot press ceramic powders. A discussion of these experiments will be published elsewhere.

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