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New Developments in Deformation Experiments at High Pressure

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2 **New Developments in Deformation Experiments at High Pressure**

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INTRODUCTION

Although the importance of rheological properties in controlling the dynamics and evolution of the whole mantle of Earth is well-recognized, experimental studies of rheological properties and deformation-induced microstructures have mostly been limited to low-pressure conditions. This is mainly a result of technical limitations in conducting quantitative rheological experiments under high-pressure conditions. A combination of factors is changing this situation. Increased resolution of composition and configuration of Earth's interior has created a greater demand for well-resolved laboratory measurement of the effects of pressure on the behavior of materials. Higher-strength materials have become readily available for containing high-pressure research devices, and new analytical capabilities—in particular very bright synchrotron X-ray sources—are now readily available to high-pressure researchers.

One of the biggest issues in global geodynamics is the style of mantle convection and the nature of chemical differentiation associated with convectonal mass transport. Although evidence for deep mantle circulation has recently been found through seismic tomography (e.g., van der Hilst et al. (1997)), complications in convection style have also been noted. They include (1) significant modifications of flow geometry across the mantle transition zone as seen from high resolution tomographic studies (Fukao et al. 1992; Masters et al. 2000; van der Hilst et al. 1991) and (2) complicated patterns of flow in the deep lower mantle (~1500-2500 km), perhaps caused by chemical heterogeneity (Kellogg et al. 1999; van der Hilst and Karason 1999).

These studies indicate that while large-scale circulation involving the whole mantle no doubt occurs, significant deviations from simple flow geometry are also present. Two mineral properties have strong influence on convection: (1) density and (2) viscosity (rheology) contrasts. In the past, the effects of density contrast have been emphasized

(Honda et al. 1993; Kellogg et al. 1999; Tackley et al. 1993), and the influence of rheology has been demonstrated by geodynamic modeling (Davies 1995; Karato et al. 2001).

Rheological properties of the Earth's mantle can be inferred from analyses of geodynamic data such as post-glacial crustal movement and gravity anomalies (Mitrovioca and Forte 1997; Peltier 1998). While such geodynamic inference provides an important data set on rheological structures of the Earth, there are serious limitations for these approaches. First, the resolution of post-glacial rebound data to infer mantle viscosity is limited to ~ 1200 km. Below that depth, there is little constraint for viscosity from post-glacial rebound (Mitrovioca and Peltier 1991). Furthermore, strain magnitude involved in post-glacial rebound is much smaller than strain magnitude involved in convection, and this raises the issue of effects of transient rheology (Karato 1998). Gravity data (e.g., the geoid) have better sensitivity to radial variation in viscosity in the deep mantle (Hager 1984), but they suffer from non-uniqueness (King 1995; Thoraval and Richards 1997). Therefore, experimental studies on mineral rheology remain a vital component in inferring mantle rheology.

High-pressure rheology may also hold some of the keys for understanding deep earthquakes. Four classes of mechanism have been suggested over the years, including thermal runaway instabilities (Hobbs and Ord 1988; Ogawa 1987), instabilities accompanying recrystallization (Post 1977), dehydration embrittlement (Jiao et al. 2000; Raleigh and Paterson 1965), and instabilities associated with polymorphic phase transformations (see also reviews by Green and Houston (1995) and Kirby et al. (1996)). Again, the main reason uncertainty still exists is the poor knowledge of materials properties at high pressures. Experimental testing of the thermal runaway involved in plastic instability requires high rates of deformation, which are difficult to obtain in small samples; modeling would be a useful tool for this mechanism, but critical parameters for models (rheological properties of deep Earth materials) have been missing so far (Hobbs and Ord 1988; Karato et al. 2001). Well-controlled deformation experiments under deep mantle conditions are needed to provide greater understanding of the shearing instability associated with the phase transformations of olivine to wadsleyite and ringwoodite (Burnley et al. 1991; Green et al. 1990) and the possibility of faulting associated with dehydration of the dense hydrous magnesium silicates (Green 2001).

Why is pressure important?

Pressure is one of the most important variables in defining the ductile rheology of mantle minerals. Where the deformation mechanism is fixed, the effect of pressure on creep strength is given by:

$$\sigma(P)/\sigma(0) \propto \exp(PV^*/nRT) \quad (1)$$

where $\sigma(P)$ is the creep strength at pressure P , V^* is activation volume, n is the stress exponent, R is the gas constant, and T is temperature. For olivine, assuming an activation volume of $V^* = 15 \text{ cm}^3/\text{mol}$, $T = 1600 \text{ K}$ and $n = 3$, $\sigma(10 \text{ GPa})/\sigma(0) = 43$. This is to be compared with the effect of water, which will change the creep strength of olivine by a factor of ~ 10 from dry to water-saturated (Hirth and Kohlstedt 1996; Karato and Jung 2002 in press) or partial melting that will affect creep strength by no more than a factor of 2 (Kohlstedt and Chopra 1994). However, the pressure effect is small at the low pressures where studies are made, e.g. $\sigma(0.5 \text{ GPa})/\sigma(0) = 1.2$. Therefore, high-pressure studies are necessary in order to determine the flow properties in the deep Earth.

Such large effects of pressure on the flow laws can result in changes of dominant flow mechanisms with depth in the Earth. Karato and Wu (1993) suggest that dislocation flow is overtaken by diffusion flow as the creep mechanism for olivine within the upper

mantle. Their reasoning is based on the differences in the pressure dependence, i.e., in the value of V^* in Equation (1), of the two flow laws.

Pressure is an agent for phase transformation within the Earth. These high-pressure phases, with different crystal structures, will also differ in their mechanical properties. Rheology of high-pressure phases needs to be investigated within the stability field of the mineral. Because of equipment limitations, the flow properties of minerals such as ringwoodite or majorite, which set the strength characteristics of the transition zone, remain virtually unknown.

The effects of water can be well characterized only by high-pressure experiments. At low pressures ($P < 0.5$ GPa), the creep strength of olivine *decreases* with pressure under water-saturated conditions (Mei and Kohlstedt 2000b; Mei and Kohlstedt 2000a). However, at higher pressures ($P > 1$ GPa), the creep strength of olivine *increases* with pressure even at water-saturated conditions (Karato and Jung 2002 in press). Therefore results that can be extrapolated to infer creep strength (viscosity) at deep portions (> 30 km) of Earth can be obtained only by quantitative experiments at pressures higher than ~ 1 GPa.

Terminology related to strength and deformation.

Usage sometimes varies among research disciplines, so let us define a few key terms, taking the language of rock mechanics as a basis (e.g., Jaeger and Cook (1976)). It is assumed that the meaning of the *stress* (σ_{ij}) and *strain* (ϵ_{ij}) tensors are understood. *Deformation* (used already in the title without definition) refers loosely to any process that results in strain. We take positive values of stress and strain to indicate compression, following geologic convention. *Pressure*, *hydrostatic pressure*, and *mean normal stress* are all equivalently the trace P of the stress tensor, namely,

$$\sigma_{ij} = \sigma'_{ij} + P \quad (2)$$

where σ'_{ij} is the *deviatoric* stress tensor, $i, j = 1, 2, 3$, representing orthogonal directions. *Confining pressure* P_c is the hydrostatic stress generated by laboratory devices to simulate geologic overburden. Although P and P_c are different when σ'_{ij} is non-zero, the difference in most geologic settings is small and the distinction is very often ignored. Different terms for the same physical quantity are sometimes used to connote different situations. Strain can be divided into two categories: *elastic*, the recoverable portion, which owes its existence to the state of non-zero σ_{ij} , and *inelastic*, the permanent strain that remains forever after stress has been removed. Classic rock mechanics usage recognizes two categories of deformation that result in inelastic strain: *brittle* and *ductile*. Brittle deformation is associated with discontinuities of displacement and sudden loss of strength (a term to be defined shortly) and is generally not volume conservative. Ductile deformation is associated with finite displacement gradients, retention of strength, and volume conservation at scales above the nm. Note that there are important strain-producing phenomena that do not fit well into either category, in particular those that involve polymorphism (phase change) or chemical reaction. They may be ignored for the purposes of this review. The *strength* of a material (in specific circumstances called the *yield strength*, *ultimate strength*, etc.) is the maximum stress the material can support under the stated environmental conditions. *Flow* and *failure* are closely related terms for the state that exists the moment that applied stress matches strength for ductile and brittle materials, respectively.

The term *plasticity* requires special note. It was originally defined for metals as essentially equivalent to the phenomenon of flow, just defined, but was defined in terms of the yield envelope (Hill 1950) without qualification as to ductile or brittle yield. While metallurgists and most rock rheologists have retained this meaning for the term plasticity

(e.g., Evans and Kohlstedt [1995] and Poirier [1985]), many in the computational branches of geology have taken plasticity to mean any yield process, whether volume conservative or not. In this review, we follow tradition and use the terms ductile and plastic interchangeably.

Here we focus exclusively on ductile deformation, so we use the terms *rheology* (flow of materials under stress), deformation, and flow interchangeably. The rheology of a material is described by a constitutive law of the type

$$\dot{\epsilon}_{ij} = Af(\sigma'_{ij}), \quad A = A(P, T, \dots) \quad (3)$$

where all relevant state variables are included in the term A . Engineering the containment of very high pressures in the laboratory generally requires high symmetry in the sample assembly. As will be seen below, two basic types of high-symmetry strain environments exist: axisymmetric and rotational. A shorthand form of Equation (3) has evolved for each of these geometries and we will use that shorthand here. For axisymmetric deformation (whose two-dimensional analog is called *pure shear*), the values of the three principal stresses are $\sigma_1 \neq \sigma_2 = \sigma_3$, and we simplify the strain rate tensor to a scalar $\dot{\epsilon}$, which we call the *strain rate* or *shortening rate*. The deviatoric stress tensor can be characterized by the scalar $\sigma = \sigma_1 - \sigma_3$, which we call the *differential stress* or simply *stress*. For rotational deformation, the strain is approximately *simple shear*, and Equation (3) becomes a scalar relationship between the *shear strain rate*, $\dot{\gamma}$, and the *shear stress*, τ .

Despite the apparent simplicity of this language of strength and deformation, one should always keep two things in mind: (1) the terminology is not universal, and (2) the simple language belies the flow of rocks in nature, which can be counted on to be both inhomogeneous and low symmetry.

A BRIEF HISTORY OF HIGH-PRESSURE APPARATUS

To 5 GPa: Cylindrical devices

Confining pressure in rock mechanics testing serves several purposes: (1) simulating geologic overburden, (2) activating mechanisms of flow and fracture that depend on both normal and shear components of stress, and (3) suppressing brittle deformation where one is interested in gathering information on purely ductile phenomena. The scientific needs for doing measurements under pressure are illustrated in detail in several other contributions to this volume. These needs have driven the development of experimental pressure systems since the early days of rock mechanics.

The earliest experimental machines for deforming rocks under elevated confining pressure were some version of a piston pushing on a cylindrical rock held fast in a cylindrical container. A solid or fluid medium within the container, or even walls of the container itself, provided confining pressure to the rock sample. The reader may find an early history of such machines in Tullis and Tullis (1986), for example. The direction of apparatus development after 1900 was influenced primarily by P.W. Bridgman, who made such rapid advances in the design of hydrostatic equipment that the only reasonable path for rock mechanics to follow was to adapt deformation to Bridgman's vessels. Bridgman, who conceived the dynamic piston seal still commonly used, was involved in that adaptation (Bridgman 1952c), although he was no longer alone in his efforts. Continuing to this day, virtually every new deformation rig is some adaptation of a hydrostatic design. The backbone of the history of deformation machines is mostly a history of pressure vessels.

The technological breakthrough behind modern gas and liquid pressure vessels was

the Bridgman unsupported area packing (Bridgman 1914), a geometry that intensified the vessel's own pressure at the point of seal and made it possible to "reach without leak any pressure allowed by the mechanical strength of the walls of the containing vessels" (Bridgman 1952b). Bridgman regularly worked at pressures in excess of 1 GPa and occasionally reached 2 GPa with his fluid vessels (Bridgman 1935b). The application of the Bridgman-seal vessel to rock deformation led to the creation of what is commonly called in rock mechanics the "triaxial" apparatus (which is somewhat inappropriate, because the stress environment in the fluid pressure medium can be no lower than axisymmetric). Griggs, who learned the techniques of high pressure from Bridgman and shared his "gift for gadgets" (Rubey 1972), initiated this application by developing a version of the unsupported area packing around a solid moving piston (Fig. 1), thereby allowing sample shortening to be equated (with appropriate corrections for elasticity) to piston displacement (Griggs 1936). Griggs (1936) added a second innovation to the design, a pressure-compensating double piston that automatically kept pressure constant even as the sample was shortened (Fig. 1). Further development of the triaxial rig was aided by advances such as the controlled-clearance (Newhall) packing to reduce piston friction (Handin 1953; Turner et al. 1956); internal heating to achieve higher sample temperatures (Griggs et al. 1960); and the internal force gauge, which removed piston friction from the measurement of differential force on the sample (Heard and Carter 1968; Paterson 1970). This list is by no means exhaustive. A notable recent development is the proliferation in many of the world's most renown rock mechanics laboratories of one particular triaxial apparatus, the Paterson rig, which is a 0.3-GPa ready-to-use system complete with actuation and internal force gauging for both axial and high-displacement torsional deformation (Paterson 1990; Paterson and Olgaard 2000). As of this writing, there are ten such vessels in use in North America and Europe.

Practical matters related to sealing a high-pressure fluid have generally limited triaxial work to about $P = 1$ GPa, with most triaxial machines, in fact, having maximum design pressures of 0.3-0.5 GPa (Paterson 1978). At higher pressures, the solid pressure medium piston-cylinder apparatus is used. The solid-medium press used most widely for geological applications is the Griggs apparatus (Griggs 1967), adapted from the hydrostatic solid-medium piston-cylinder apparatus (Boyd and England 1960), and subsequently improved to provide a stress resolution of a few MPa (Borch and Green 1989; Green and Borch 1989; Tingle et al. 1993). Since a solid medium has a finite shear strength, measurements are limited to materials

Figure 1. The Griggs triaxial deformation apparatus, including the yoked pistons to hold pressure constant during deformation. As piston P_1 moves against the sample, piston P_2 automatically withdraws from the vessel at the same rate, holding the pressurized volume constant. After Griggs (1936), Fig. 2; reproduced with permission.

that are stronger than the medium, so very low strain rates and temperatures near the sample melting point must be avoided, but pressures of several GPa are now easily accessible. The Griggs apparatus has been regularly used to 1.5-1.8 GPa, and to a maximum pressure of 4 GPa.

A recent development is an apparatus built by Gettling (Gettling 1998; Gettling and Spetzler 1993) that combines aspects of the solid-medium and fluid-triaxial deformation machines. Gettling's is a piston-cylinder device that features precision packings that are sufficiently tight to contain solid argon (an exceedingly weak solid) but sufficiently reproducible in their frictional behavior that much of the uncertainty in stress introduced by piston friction can be accounted for by calibration. Gettling (1998) claims pressure uncertainty at $P = 3$ GPa at the extraordinary level of a few MPa. If this resolution applies also to the sum of $\sigma_1 + P$, as expected, it may mean that rock strengths can be measured with a resolution that is comparable to that of many triaxial gas rigs that operate at far lower pressures.

The practical pressure limit for all cylindrical machines is around 5 GPa. While the pressure capacity of a hollow cylinder (or any shape, for that matter) is a function of wall thickness and therefore virtually unlimited (Eremets 1996), the same is not true for the piston. As one approaches $P = 5$ GPa, the stress required to drive the piston into the pressure cylinder (which must be 5 GPa plus the stress to overcome packing friction plus the strength of the sample) causes pistons constructed of even the strongest of steels to creep. Pistons will swell and seize in a short amount of time. Note that this 5-GPa limit exists for hydrostatic piston-cylinder vessels as well. Bridgman reached higher pressures in hydrostatic vessels by confining the entire piston and cylinder in a larger vessel, thus raising σ_3 on the piston to the pressure in the outer vessel and allowing the piston to apply correspondingly more stress to the inner vessel without exceeding its strength limit. His vessel-inside-vessel multi-staged devices have been used to reach pressures of at least 10 GPa (e.g., Bridgman (1942; 1948)). Because of the complexity of those vessels, however, they are not well suited to being instrumented for deformation studies.

5 GPa and above: Anvil devices

A much simpler method for achieving an elevated σ_3 is to give the pistons a tapered shape. (The change from rectangular to trapezoidal section also merits a name change: from piston to *anvil*.) Now a component of the force applied in the σ_1 direction at the larger end of the piston/anvil is directed in the σ_3 direction at the smaller end (platen face), making $\sigma_3 > 0$ at the platen face. The drawback of this solution is, of course, that one cannot expect to contain the pressure medium over large displacements in a conventional cylindrical pressure vessel if the sides of the (moving) anvil are tapered.

Sealing against a moving tapered surface can be achieved for small displacements through use of a crushable gasket material; so if one is satisfied with anvil displacements that are less than gasket thickness, one can reach high values of σ_1 at the platen faces without exceeding the failure criterion of the anvils. The first application of this principle is generally credited to Bridgman's (Bridgman 1952a) opposed-anvil device, which is capable of 10 GPa, although Bridgman used the same principle many years earlier in confining samples in a 5-GPa shear apparatus (Bridgman 1935a). It is also the underlying principle of the Drickamer cell (see below). Note that the original purpose of anvil displacement in these designs was pressure generation rather than sample deformation, although as we will see below, the possibility of the latter was not ignored.

A slight variation, which allows for larger volumes at a given pressure, is to combine the function of pressure container and pressure generator by clustering several anvils in a quasi-spherically symmetric pattern. These are called multianvil devices. Four-anvil

tetrahedral (Hall 1958; Houck and Hutton 1963; Lloyd et al. 1959), six-anvil cubic (Carter et al. 1964; Houck and Hutton 1963; Osugi et al. 1964), and eight-anvil octahedral (Kawai and Endo 1970; Walker et al. 1990) have been constructed. The eight-anvil devices are multistage, with the eight anvils of the final stage contained within the working volume of an intermediate cubic stage. They are often referred to, therefore, as 6/8 devices. Hydraulic actuation of the several anvils was sometimes independent or in opposed pairs (Carter et al. 1964; Hall 1958), but better symmetry of anvil displacement was achieved, along with higher pressures, when the anvils were driven by wedge-shaped guide blocks that then required only one hydraulic actuator (Lloyd et al. 1959). There are at least two versions of the wedge-type cubic multianvil device: a split-sphere device (Houck and Hutton 1963) and the DIA (Osugi et al. 1964; Shimomura et al. 1985); the latter will be describe below in detail.

Wedge guide block machines achieve high pressures more reliably than machines with multiple actuators, and for that reason they have proliferated, especially the DIA and 6/8 devices (e.g., see review by Onodera (1987)). Note, however, that their design function is exclusively hydrostatic pressure generation. Advances in high-pressure rheology required that the quasi-isotropy within the pressurized volume be broken; those advances are the main subjects of this review.

Another reason for the recent proliferation of multianvil devices is a serendipitous advantage they have over pressure cylinders: optical and/or X-ray transparency along a line of sight leading directly to and from the sample (Fig. 2). In the case of diamond (or sapphire) anvil cells, the anvils themselves are transparent. For devices where anvils are opaque, lines of sight are always available in the narrow gaps between anvils, and the gaskets compressed in those gaps can be made of X-ray-transparent weak material (e.g., pyrophyllite). The analytical potential of high-energy X-rays and their recent availability at very bright synchrotron sources has been the motivation for development of a new generation of deformation machines that have the pressure capacity of anvil devices and the lowered symmetry of deformation machines.

MEASUREMENT METHODS AT HIGH PRESSURE

Stress measurement

Measurement of deviatoric stress is key to quantifying the rheological properties of a material. Stress provides information about strength relative to the laboratory time scale. Stress, measured in conjunction with ductile strain, can yield flow laws within the context of the state of the sample. The crucial ingredient to facilitate such studies is the development of a stress meter, or piezometer.

In experiments operating below 5 GPa, the deforming force is applied directly to the sample and various strategies are designed to measure the magnitude of this deforming force. Stress is then given as the ratio of force to sample area. Typically, a load cell placed in series with the deforming piston defines this force along the deforming column. Combinations of friction between the force gauge and the sample and forces that support the piston, such as in the multianvil system, continually degrade the ability to relate a measured force to sample stress. These problems begin to interfere with the resulting accuracy at 0.3 GPa and completely overwhelm the measurements by 5 GPa.

A revolutionary technique for stress and strain measurements under high-pressure (and temperature) conditions *in situ* has now become feasible using high-energy X-rays generated by a synchrotron radiation facility. These new tools are just now being explored and their limitations defined. The exceptional quality of these tools rests in the fact that they are directly monitoring the sample. Stresses are measured in the sample.

Figure 2. Sample assembly in the DIA cubic apparatus, shown with three of the six anvils removed and with the sample assembly itself, i.e., the cube, shown in cross section. Analytical synchrotron X-rays enter and exit along a horizontal line through the sample and normal to the section as shown here. The X-rays pass through the vertical gaps between anvils. Drawing by M. Vaughan.

Different positions in the sample can be isolated to test for stress uniformity. Measurements of stress and strain can be time resolved with a precision of about one minute. Strain is also obtained by images of the sample. Distribution of strain with position and time can be defined. All of this is done with a current accuracy of a few tens of MPa in differential stress and 10^{-4} in strain.

Strategies for measuring stress are found through use of the X-ray diffraction or pressure-sensitive probes (such as ruby fluorescence), and are being made available by exploiting powerful synchrotron X-ray sources. The basic principle of X-ray piezometers is that lattice strain is sensitive to elastic strains and not to plastic strains. Changes in distances between atoms are expected to balance stress fields that are present. These stress fields may be created by dislocations or external forces, but departures from the lattice spacing that the material experiences at zero pressure and stress are sensibly related to the stress field within the sample. Furthermore, the coupling between the strain and stress is expressed by the elastic moduli. There are two styles of X-ray piezometers, one based on stress gradient and the other based on strain anisotropy.

Stress gradient. Where the inertia term can be neglected, variation in the stress field is restricted to obey the mechanical equilibrium relations:

$$\sum_{j=1}^3 \frac{\partial \sigma_{ij}}{\partial x_j} = 0$$

Three equations, one for each value of i , represent the vector force balance equations. The first of these equations is:

$$\frac{\partial \sigma_{11}}{\partial x_1} + \frac{\partial \sigma_{12}}{\partial x_2} + \frac{\partial \sigma_{13}}{\partial x_3} = 0$$

Thus, if there is a variation of the normal stress, σ_{11} , with x_1 , there must also be a variation of the shear stresses, σ_{12} , in the x_2 direction and/or a variation of σ_{13} in the x_3 direction. The peak magnitude of the shear stress is related to the variation of the normal stress (or pressure) and the length scales of two types of stress. This has been exploited in the diamond anvil cell geometry by measuring the radial gradient of pressure to deduce the axial (along the axis of the diamonds) variation of shear stress. This is illustrated in Figure 3. The shear stress, τ , vanishes along the center plane of the sample and along the central axis of the cell where $\partial P / \partial r$ is zero. The maximum shear stress is at the sample-diamond interface and is given by

$$\tau_{\max} = (\partial P / \partial r)_{\max} d / 2$$

where d is the thickness of the sample. This technique was developed by Sung et al. (1977) and was further used by Meade and Jeanloz (1988a,b; 1990). These experiments relied on ruby dust distributed across the sample as a pressure marker for delineating the

Figure 3. Illustration of stress field in a diamond-anvil cell. The radial distribution of pressure, illustrated at the top, is coupled to the shear stress distribution along the axial direction. Force couples indicate the sense of the shear stress distribution within the cell.

pressure gradient. As Wu and Bassett (1993) illustrate, the pressure marker can also be an X-ray diffraction standard such as gold. The objective is to measure the radial pressure profile to define the shear-stress distribution. Often, the peak shear stress is taken as the yield point of the solid.

Stress gradients coupled with deviatoric stress affect the diffraction peak shape as described in detail by Weidner(1998) and Weidner et al. (1998). In this case, heterogeneities in the stress field broaden the diffraction peaks. Since the length scale of stress variation is the same for normal and shear stress, then the magnitude of the heterogeneity for all stresses is similar. Weidner et al. (1998) model the relationship between the observed strain (that creates the broadened diffraction peak) and the stress magnitude and conclude that Young's modulus is the appropriate coupling elastic modulus, or:

$$\sigma_{\text{differential}} = E \epsilon_{\text{differential}}$$

where the differential strain is deduced as the “strain broadening” contribution to the peak profile. Since for the peak-broadening measurement, the full-width, half-height of the diffraction peak is used to define the strain broadening and represents roughly two-thirds of the scattering diffraction planes, then most of the sample must be experiencing the inferred shear stress. Deviatoric stress can originate from dislocations, from heterogeneities in material or properties, or any number of sources. Peak-broadening analysis is often used in commercial applications to determine stresses induced by processing. For example, internal stress generated by welding is often analyzed by this method. In high-pressure studies, the stress is most commonly generated by compressing powdered samples (e.g., Chen et al. 1998). As a rheological tool, this approach resembles an indentation experiment where the indenter is the same material as the sample. Weidner et al. (1994) studied the high-temperature and -pressure strength of diamond with this method. Chen et al. (1998) studied plastic deformation of olivine, wadsleyite, and ringwoodite. Flow can also be studied with this piezometer, with the constraint that this becomes a relaxation experiment (Weidner et al. 2001).

Strain anisotropy. A uniaxial stress will introduce elastic strains in the sample. The strain parallel to the axis of compression will generally be larger than the strain perpendicular. The material's elastic moduli quantitatively define the relationship between these strains and the imposed stresses. X-rays sample the distance between lattice planes (called *d*-spacing) whose normals are parallel to the diffraction vector, which is the bisector between the incident X-ray and the detector. The lattice spacing is insensitive to strain history (plastic strain) but reflects the elastic strain field. Figure 4 illustrates a Debye ring that would be observed from a powder sample in a stress field using a monochromatic X-ray beam and an area detector. The lattice spacing is related to the distance from the center of the image to the Debye ring through Bragg's law. For the angle Ψ of zero, the lattice spacing reflects that measured parallel to the x_1 direction. For Ψ of ninety degrees, the X-rays are sampling grains aligned parallel to the x_2 direction. The strains deduced from these measurements compared with ambient conditions are related to the stresses by:

$$\epsilon_{ij} = \sum_{k=1}^3 \sum_{l=1}^3 s_{ijkl} \sigma_{kl}$$

where *s* is the elastic compliance tensor.

In the actual case, more than one set of diffraction planes will produce Debye rings similar to the one illustrated here. Each will be approximately circular in shape, but distorted by the stress field. The amount of distortion may be different if the elastic

Figure 4. X-ray diffraction line on a two-dimensional detector from a sample under stress. The distance from the center to the ring defines the scattering angle, 2θ , and hence the lattice spacing through Bragg's law. The presence of the stress field distorts the Debye ring from its normally circular shape. The usual stress field is axisymmetric, with $\sigma_2 = \sigma_3$. The diffraction vector at $\psi = 0$ lies at an angle θ from the direction of σ_1 . θ is typically only 3 to 4 degrees.

compliance tensor is not isotropic. To evaluate this in detail, we need to be careful to account for the crystal orientation when defining the compliance tensor. Thus, in a non-hydrostatic stress field, crystallographic planes with different orientations change their spacing by different amounts. For cubic crystals, the relationship between the Ψ dependence of strain and the differential stress, $\Delta\sigma$, is given by:

$$\epsilon_{hkl}(\Psi) = \Delta\sigma / 3 \{1 - 3\cos^2\Psi\} \{(S_{11} - S_{12}) [1 - \Gamma(hkl)] + S_{44} \Gamma(hkl) / 2\}$$

Each diffraction peak corresponds to a set of lattice planes defined by the Miller index, hkl . In an anisotropic crystal, the elastic moduli will depend on the particular set of planes that are considered. The function, $\Gamma(hkl)$, varies from 0 to 1 and accounts for this variation as it multiplies the different elastic compliance moduli, S_{ij} . Thus, for a particular diffraction plane, hkl , the right hand bracket contains a constant value. Then ϵ varies in a fixed manner with the angle, Ψ .

The above expression is based on the assumption that each grain experiences the same stress field, or the Reuss condition. A similar relation can be derived by assuming that each grain satisfies the Voigt condition of iso-strain. In this case the equation is altered by replacing the elastic shear compliances, S_{44} and $(S_{11} - S_{12})$ by their Voigt equivalent. This relation for cubic symmetry is generalized by Singh et al. (1998) for all crystal symmetries.

This phenomenon gives rise to a couple of strategies for a piezometer. Stress has a manifestation even for a single value of Ψ . For an elastically anisotropic material, the strain for a given crystallographic plane will depend on the orientation of crystal with respect to the applied stress and the stress magnitude. Therefore by measuring the strain for several diffraction peaks corresponding to different orientations, one can determine the stress magnitude using the known elastic constants of the crystal. Such a measurement can now be done in a multianvil high-pressure apparatus using a synchrotron X-ray source (Weidner et al. 1992). The accuracy relies on knowing the elastic anisotropy at the pressure/temperature conditions of the experiment.

A more robust strategy comes from mapping out the d spacing as a function of Ψ . The values of the elastic moduli are still required, but the details of anisotropy are less critical to yielding precise measurements. With a precision of 10^{-4} for the lattice spacing

and a typical elastic modulus of 200 GPa, it should be possible to resolve differential stresses of ~ 20 MPa. Singh has reduced the elasticity equations to a closed-form solution for several crystal symmetries and has given a concise summary (Singh et al. 1998).

This methodology was first applied in a diamond-anvil cell by Kinsland and Bassett (1977) by passing the X-ray beam through a Be gasket, perpendicular to the axis of the diamonds. The multianvil system has not been accessible by this technique because the anvils themselves cast a shadow and limit the range of Ψ where the diffracted signal can be observed. However, we have recently experimented with cubic BN anvils. These are nearly as hard as diamond but are about an order of magnitude less expensive. Furthermore, they are transparent to X-rays. Thus, by using these anvils it is now possible to observe diffraction spectra in any plane relative to the incident beam.

Here we have illustrated this tool with monochromatic X-rays and angle dispersive measurements. Energy dispersive methods are equally valid for these measurements and are sometimes advantageous. Spectra need to be collected for different values of Ψ through collimators that fix the two-theta value. Weidner's group has designed and used a conical slit system for this purpose as illustrated in Figure 5. Two concentric cones, whose angle is the desired diffraction angle for white, energy dispersive diffraction, create the slit itself. Solid-state detectors with energy discrimination are placed at specific values of Ψ behind the slit system. Each detector is calibrated independently. Lattice spacings are used as illustrated above to define the differential stress.

Figure 5. Conical slit assembly in use at National Synchrotron Light Source. The cone defines the two-theta angle and is constructed by two concentric cones with this angle. The X-rays that pass between the two cones originate from the sample at that angle as all other X-rays are blocked by the cones. Multiple energy discriminating detectors, located behind the slit assembly, record the X-ray signals. A YAG crystal located inside the slit assembly fluoresces in the X-ray beam, emitting visible light where an X-ray photon passes. The visible light is magnified by a microscope assembly and recorded by a digital camera.

Strain rate measurement

Synchrotron X-rays can also be used to measure sample length. Direct images of the sample can be obtained using an incident beam whose dimensions are larger than the sample. Platinum or gold foils above and below the sample are easily viewed. The X-ray image is obtained by projecting the X-ray onto a fluorescent screen that is viewed with a CCD camera through a magnifying system. A typical image is shown in Figure 6. The

horizontal lines in this image are gold discs placed at the ends of the sample. The black edges are the shadows of the opaque tungsten carbide anvils. Cubic boron nitride anvils are transparent and can be used to view the entire sample because they do not cast such a shadow. Figure 5 illustrates the position of the microscope for these measurements. The fluorescent screen is located on-axis inside the conical slit system. We determine that, by comparing two images, strains of 10^{-4} can be measured. For images taken 100 seconds apart, this allows resolution of strain rates of 10^{-6} s^{-1} .

Figure 6. X-ray shadowgraph of MgO sample during compression. The horizontal dark lines are images of gold foil that bound the sample whose length is ~ 1 mm. The horizontal dimension is defined by the opening between the high-pressure anvils. The shadow across the top is caused by a platinum foil wrapped around the end-plug.

The X-ray image yields the total strain of the sample, which is the sum of the elastic and plastic strain or:

$$\epsilon_{\text{ob}} = \epsilon_{\text{el}} + \epsilon_{\text{pl}}$$

In an experiment both the elastic and plastic components may vary with time. Thus, to deduce the plastic strain rate, it is necessary to account for the variations of all strain components. The elastic strain, however, is reflected in the diffraction pattern and can be represented by a unit cell length, a . This cell-edge length is obtained from analysis of all of the diffraction peaks recorded with a diffraction vector parallel to the sample axis. In elastically isotropic samples, there is no ambiguity in defining this value. Anisotropic samples in a differential stress field will exhibit lattice spacings that reflect the aggregate properties. In this case, an average cell dimension based on several diffraction peaks should be obtained. We define the Kung ratio, R_k , as:

$$R_k = (d/d_0) / (a/a_0)$$

where d is the length of the sample measured with an image and a is a unit cell length measured from diffraction; the zero subscript refers to a reference value such as ambient conditions. This ratio, which is normalized to one, represents the length of the sample in the units of the cell dimension. This number is proportional to the number of unit cells that make up the length. The Kung ratio remains constant if all deformations are elastic. Thermal expansion, pressure, and stress will not change the Kung ratio if the sample responds elastically. Plastic strain changes the Kung ratio. In this case the number of unit cells that define the length changes. Thus, the Kung ratio can be used as the proxy for plastic strain, or:

$$\epsilon_{\text{pl}} = [R_k(2) - R_k(1)] / R_k(1)$$

where (1) and (2) represent two states of the material.

Strain is not always uniform in the sample. Visual tools as described here provide opportunity to map the strain field as a function of space and time during the experiment. Vaughan et al. (2000) demonstrate that one can embed strain markers in a sample during preparation and view the position of these markers during the experiment. Figure 7, from this work, shows a grid made from a TEM gold sample grid, in the sample at different P, T conditions. The sample was a sintered olivine cylinder. The cylinder was sliced into

two halves, the grid inserted, and the sample halves placed back together. The sample assembly included hard end-plugs in a DIA apparatus, so as to produce shortening during compression. The grid size is initially about 70 microns. With these images, it is possible to map the strain as a function of position in the sample and as a function of time by comparing successive images.

Figure 7. A TEM gold grid embedded on the central plane of a cylindrical sample. Images at different conditions illustrate different amounts of strain. The initial pressurization distorted the grid because surfaces of sample assembly parts were not perfectly flat. Subsequent strain can be mapped by following specific strain markers.

MODERN TECHNIQUES FOR DEFORMATION AT HIGH PRESSURES

In current technology, deformation at $P > 10$ GPa is the realm of anvil devices. We describe in detail four methods currently in use or in active development: (1) sample assembly modification to hydrostatic multianvil devices, (2) sample assembly modification to the DAC, (3) the D-DIA, and (4) the RDA.

Sample assemblies

Sample assemblies used in these machines share several common features of those used in hydrostatic devices, serving common requirements such as high pressure, high temperature, and control of chemical activity in the sample. With the exception of the specialized environment of the DAC, these common features include a pressure medium, such as soft fired pyrophyllite, ZrO_2 , $\text{MgO}/5\%\text{Cr}_2\text{O}_3$, castable ceramic or epoxy; a furnace, for example, metal foil, LaCrO_3 , or graphite; and a soft medium surrounding the sample itself, for example, BN, MgO, NaCl, or glass (the latter two occasionally as liquids). Descriptions of such assemblies can be found in, e.g., Kawai and Endo (1970), Liebermann and Wang (1992), Walker (1991), and Weidner et al. (1992). For control of chemical environment the sample can be encapsulated along with buffering compounds (see e.g., Rubie et al. 1993).

Non-hydrostaticity is introduced by introducing hard platens that impart shear or

normal stresses to the sample. Several of the methods for achieving this are compared in Figure 8. They provide axisymmetric deformation in the multianvil devices (Figs. 8a-8c), simple shear in the multianvil (Fig. 8d), axisymmetric deformation in the DAC (Fig. 8e), and rotational (approximately simple shear) deformation in the RDA (Fig. 8f). We compare these assemblies briefly here and in more detail below.

Figure 8. Starting ($P = 0$) configurations of sample columns used for deformation experiments at very high pressures. For simplicity, details such as sample encapsulation, thermocouples, and furnace electrodes are not shown. (a) Stress relaxation. The sample is loaded elastically during pressurization. The furnace and outer and inner pressure media shown here also apply to (b)-(d). (b) Constant stress/strain rate tests. The porosity within the crushable ends of the column takes up most of the column shortening during pressurization. (c) Specialized version of (b), with a fluid in the column assuring strictly hydrostatic loading of the sample during pressurization. (d) Simple shear, high strain. (e) Column for deformation in the diamond-anvil cell. Use of a separate piston is optional, i.e., the diamond anvils can also act as pistons. In some applications, a ruby piston can also serve as a differential stress gage. (f) Assembly for the rotational Drickamer apparatus, also shown in Figure 13.

The adaptations to the DIA and 6/8 discussed in the next section use a column such as shown in Figure 8a, in which the source of deviatoric stress in the sample is the closure of the anvils during initial pressurization. At the scale of Figure 8a, the shortening of the column (elastic plus inelastic) is approximately 20% of the column length (Durham and Rubie 1998), so the “crushable” material at the ends of the column, present to protect thermocouple wires and furnace electrodes, has a porosity somewhat less than 20% of the column length. To avoid deforming the sample during pressurization in devices capable of deforming at fixed pressure (currently only the D-DIA) one uses a column such as in Figure 8b, where the length of crushable material is sufficient to absorb all anvil displacement during pressurization. When a strictly hydrostatic environment is required during pressurization, such as when the sample is a fragile single crystal, it is also possible to create a liquid cell around the sample before the pressurizing load is applied (Fig. 8c) (Durham, unpublished results).

Figure 8d shows the method for converting axial displacement to high-strain, simple-shear deformation in the multianvil cell (Karato and Rubie 1997). As discussed below, this design was first introduced as a method for imposing larger strains in the stress-relaxation mode of the hydrostatic multianvil device. However, it can also be used in the D-DIA, presumably with slightly more crushable material at the ends, to carry out constant stress or constant strain-rate tests to very high simple-shear strains.

The very simple deformation assembly for the DAC (Fig. 8e) reflects the greatly reduced available volume and the fact that laser heating can be used in place of a resistance heater to achieve local heating of the sample. Finally, the assembly for the RDA (Fig. 8f) is the rotational analog to the axisymmetric columns in Figures 8a-8c. Because rotational deformation and pressurization are entirely decoupled in the RDA, there is no need for crushable material in the cell.

Modifications to the sample assembly in a multianvil press

Fujimura et al. (1981) developed one of the first techniques for converting the environment of the 6/8 device from hydrostatic to non-hydrostatic, thus making the 6/8 device a sort of deformation apparatus. By making the elastic character and/or mechanical strength of a sample assembly anisotropic, one can create a deviatoric stress in the assembly (e.g., Fig. 8a). This deviatoric stress can be relaxed by plastic flow upon heating. This technique has been used in numerous studies (Bussod et al. 1993; Durham and Rubie 1998; Fujimura 1989; Green et al. 1990; Karato et al. 1998; Karato and Rubie 1997; Weidner et al. 2001; Weidner et al. 1998). One typically achieves the anisotropic strain by embedding a rigid sample column in the compliant pressure medium. As the assembly is pressurized at room temperature (where most relevant geologic materials are very strong), a large deviatoric stress is generated on the sample. When temperature is increased, the sample softens and begins to flow, and the stress gradually relaxes. The analysis by Karato and Rubie (1997) (see also Durham and Rubie [1998]) showed that the mode of deformation in most cases is this “stress relaxation,” i.e., the magnitude of deviatoric stress changes significantly within a single experiment.

This technique has several limitations. First, pressurization and plastic deformation are not completely separated; therefore, deformation during pressurization likely occurs unless special care is taken in sample assembly to absorb the initial-stage shape change of sample assembly (Durham and Rubie 1998; Karato and Rubie 1997). Second, deformation by this method is “stress relaxation,” and the interpretation is complicated because of the change in stress that could cause the change in deformation mechanisms. Third, the amount of inelastic displacement is limited to the amount that can be imposed elastically during pressurization, usually well under 100 μm . By using a thin sample sandwiched between two pistons cut at 45°, Karato and Rubie (1997) were able to convert this very small displacement to relatively large strains ($\gamma \approx 1\text{-}2$) (Fig. 8d), yet the maximum strain is not large enough to obtain steady-state fabrics. Quasi-constant displacement rate tests can also be made with multianvil apparatus through the continuous movement of the hydraulic ram (Bussod et al. 1993; Green et al. 1990), although one must realize that pressure is increasing steadily and significantly in such tests.

The multianvil deformation technique has been applied to ~25 GPa, ~2000 K with a sample dimension of ~1-2 mm diameter and ~2-4 mm long, or a thin (~0.2 mm) disk sample with a similar diameter (Chen et al. 1998; Cordier and Rubie 2001). Sacrificing pressure for sample size, Green et al. (1990) deformed 3-mm diameter x 6-mm length samples in a 6/8 device. The major advantage of this technique as compared with a diamond-anvil cell technique is that rheological properties can be investigated under more homogeneous high temperature and pressure conditions with a better-controlled

chemical environment. Also, because relatively large samples can be used, microstructural evolution during deformation and its effects on rheology can be investigated (Green et al. 1990, 1992; Karato 1998a; Karato et al. 1998).

The limited plastic deformation is a serious shortcoming of the method. The displacement available is generally not sufficient to probe important rheological questions about lattice preferred orientation and strength in the steady state. Second, because the stress magnitude changes during an experiment, dominant mechanisms of deformation may change in a single run, making interpretation of mechanical data and microstructures difficult. This last point, however, may turn out to be an advantage for the study of rheology at a very small strain-rate (small stress) that is relevant to Earth.

Diamond-anvil cell

The highest pressures obtainable in the laboratory are in the diamond-anvil cell. This device loads the sample uniaxially and, thus, naturally produces a deviatoric stress environment. The first examinations of differential stresses in the diamond cell were motivated by the need to produce a hydrostatic environment. Use of the diamond cell as a deformation device was pioneered by Kinsland and Bassett (1977) and Sung et al. (1977). The former used X-ray transparent gaskets and passed the X-ray beam through the gaskets, perpendicular to the diamond-cell axis. Stress was measured from the shape of the Debye rings as discussed above. The measurements provided the first estimates of the room temperature strength of MgO at high pressures. Sung et al. (1977) developed the methodology for using pressure gradients to deduce shear stress. In this case, a sample is sandwiched between two single crystals of diamond, and radial distribution of pressure is determined by measuring the shift of fluorescence lines of ruby crystals located at various points in the sample space. Then, assuming that the pressure gradient is supported by the sample strength, one can estimate the sample's strength from the equation for force balance. This technique has been used extensively by Meade and Jeanloz (1988a,b; 1990) at room temperature to the pressure of ~40 GPa.

Chai and Brown (1996) developed a diamond-cell piezometer based on the splitting of the ruby fluorescence lines. They find that the R_2 line reflects the average stress, that the splitting between the R_1 and R_2 lines is sensitive to the differential stress, and that the character of the sensitivity depends on whether the load is applied parallel to the c -axis or the a -axis. This phenomenon suggests experiments that can be done with a diamond-anvil cell at room temperatures using single crystal ruby as the pressure/stress calibrant.

The major advantage of diamond cell studies is the high pressures that can be used under which a sample can be plastically deformed. Wenk et al. (2000) recently deformed Fe at ~220 GPa. However, there are major limitations with this approach: homogeneous heating is difficult in a diamond-anvil cell, and almost all previous results were obtained at room temperature. In addition, the strain rate is not well constrained even though deformation is likely to be time-dependent in these tests. Furthermore, the sample space is so small that some important effects such as grain-size sensitivity of strength are difficult to measure.

Poirier et al. (1981) and Sotin and Poirier (1990) report on a sapphire cell that enables larger sample volumes than a diamond cell and is more versatile for higher temperature studies. They calibrate the stress field from the characteristics of the loading system and observe the movement of strain markers in the sample. This enables them to define strain rate along with the stress determinations.

Deformation-DIA

Samples of MgO and polycrystalline tantalum were deformed in the deformation-DIA (D-DIA) at the National Synchrotron Light Source (NSLS) in February 2002. This

marked the first materials test ever above $P = 10$ GPa in which virtually all relevant rheological independent variables were under full and independent operator control. We provide here a detailed description of this new apparatus.

The D-DIA (Wang et al. 2002) is a modification of the cubic anvil device known as the DIA (e.g., Osugi et al. (1964) and Shimomura et al. (1985)) a single-stage, wedged guide block machine. The hydrostatic DIA usually operates to ~ 15 GPa and ~ 2000 K. The D-DIA modification gives independent displacement control to one pair of opposing anvils, reducing the cubic symmetry of the DIA to tetragonal, thus allowing high-strain deformation experiments to 15 GPa.

The original DIA consists of symmetric upper and lower guide blocks, four wedge-shaped thrust blocks, and six anvils, as indicated in Figure 9. Four of the anvils are mounted on the inside faces of the thrust blocks and the other two are mounted on the inside central faces of the guide blocks, the square fronts of the anvils thus defining a cubic volume at the center of the apparatus. The operation of the DIA can be visualized by recognizing that the eight inner inclined surfaces of the guide blocks define a virtual regular octahedron whose dimension changes with the separation distance between the guide blocks. The six anvils are aligned with the six apices of this virtual octahedron, and parts are machined such that the (fixed) distance from anvil face to associated apex of the virtual octahedron is precisely the same for each anvil. As the guide blocks close or open, all displacements of the anvils are symmetric about the center. The force of a single hydraulic actuator (or ram) applied along the vertical axis in Figure 9 closes the guide blocks and, thus, compresses the cell hydrostatically. Ram forces of 100 T are typically required with various truncation sizes (typically 3 to 6 mm) to reach maximum pressures near 20 GPa (see e.g., Shimomura et al. (1992) and Utsumi et al. (1992)). By the cubic symmetry of the DIA assembly, this force is divided into three equal portions along each of the three orthogonal directions defined by opposing pairs of anvils.

Figure 9. The original hydrostatic DIA apparatus. A single hydraulic ram (large arrows) drives 2 wedge-type guide blocks toward one another. The 6 anvils (truncated pyramids on the guide blocks and wedge blocks; only 5 are visible here) are thus driven toward one another, symmetrically compressing a cubic sample assembly (not shown) at the very center.

Independent control of one anvil pair in D-DIA is provided by two additional hydraulic actuators—called differential rams—within each guide block (Fig. 10). These rams may be thought of not as parts that are added, but rather as a central portion of each guide block that has been cut free so that it can move vertically. A small hole is introduced into the side of each guide block to allow access for hydraulic fluid to each ram, and seals for the pressurized plenum below each differential ram are provided. The

conical shape of the differential rams is rather unimportant; it leaves more steel on the guide blocks and makes them slightly stronger. Note that in the D-DIA modification, the guide blocks not only support the forces confining the thrust blocks, they now have also become thick-wall pressure containers for the hydraulic fluid driving the differential rams. For this reason, the guide blocks of the D-DIA are complete discs rather than crossed steel members as in the original DIA (Fig. 9).

Figure 10. Computer-generated 3/4 section through the D-DIA. Comparing to Figure 9, the guide blocks are now full cylindrical shapes, but the four wedge blocks and six anvils are identical to those in Figure 9. The two differential rams, the large conical pieces within the top and bottom guide blocks, move independently of the guide blocks themselves, and can thus generate a deviatoric state of stress in the assembly at the center (not shown). In actual operation, as the differential rams displace inward toward the center, the guide blocks move apart to allow the four wedge blocks to displace outward. The displacement rates are independently controllable and are usually chosen such that the net volume of the sample assembly, and therefore the confining pressure, remains constant.

The differential rams have a (pressurized) diameter of 89 mm and are therefore capable of generating a force of 125 T at a hydraulic pressure of 0.2 GPa, sufficient to overcome the force of the main ram (up to 33% of the main ram maximum of 300 T) and apply differential force even at the highest confining pressure. Finite element analysis of the D-DIA guide blocks show that the maximum octahedral stress occurs on the inner bore in a “typical” operation where the differential load is 67% of the main ram load (presuming some force is needed to overcome packing friction and sample strength) (Wang et al. 2002). The stress at the base of the inclined surface of the guide block, the next highest point of stress concentration in the guide block, is roughly 90% of the stress in the bore. Note that in the original DIA with very large notches cut in the guide blocks, the stress concentration at the base of the inclined surfaces is twice that of the D-DIA under a similar main ram load.

The key point of independent control of the differential rams is that deformation can be imposed without the necessity of increasing confining pressure (which has been the

long-standing limitation of deformation studies in 6/8 multianvil and diamond-anvil assemblies). In normal operation, the D-DIA sample is brought to run conditions of P and T with differential rams fully withdrawn in the same manner as the hydrostatic DIA. Advancing the differential rams then introduces a non-cubic shape change to the assembly. Furthermore, by simultaneously draining hydraulic fluid from the main ram at an appropriate rate, the four side anvils retract, and the total force of the main ram as well as the volume of the sample cell can be held constant. In the synchrotron X-ray beamline) pressure itself can be monitored and, accordingly, serves as the process variable. The operation at NSLS in February 2002, showed that this procedure gave very satisfactory control of pressure over very large displacements of the differential rams (Fig. 11).

The differential rams are driven by high-precision syringe pumps, and their velocities are controllable from approximately 10^{-7} to 10^{-2} mm/s. When both rams are driven symmetrically, this translates to a strain rate on a typical 1-mm-length, 1-mm³-volume sample of $2 \times 10^{-2} \geq \dot{\epsilon} \geq 2 \times 10^{-7} \text{ s}^{-1}$. These rates are much faster than most relevant rates in geology, but as with all experimental rock mechanics work—for which these rates are typical—it is the human time scale and not the geologic time scale that governs the duration of an experiment. As with most experimental studies of rock deformation, appropriate scaling analysis is critical to apply these results to Earth. Differential ram displacements of >1 mm are possible, so strains in pure shear compression can approach 1. Deformation in simple shear, using 45°-cut pistons (see “Sample Assemblies,” above) is also possible, allowing for much higher strains, at some cost to sample volume. Finally, the D-DIA is capable of extensional, as well compressional, deformation, because the sense of motion of the six anvils can be reversed.

Rotational Drickamer apparatus (RDA)

Large strain deformation experiments can be conducted in the torsion mode, allowing a detailed study of deformation microstructures as well as rheology. Paterson and Olgaard (2000) described such an apparatus in which a torsion actuator is attached to a gas-medium high-pressure apparatus. This apparatus contains an internal load-cell, making possible a precise measurement of stress. However, the maximum pressure of operation is limited to ~ 0.5 GPa. Deformation experiments can be conducted at higher pressures by twisting a thin sample between anvils. Bridgman (1935a; 1937) was the first to apply the technique to 5 GPa with an apparatus that consisted of two fixed anvils bearing on a flat, rotating anvil. The design was later modified by Griggs et al. (1960) to remove the intermediate anvil and rotate one of the anvils instead. Similar attempts were made in the late 1960s at room temperatures and at pressures to 7 GPa (Abey and Stromberg 1969; Riecker and Seifert 1964) in which a sample was sheared, and the strength was determined by the measurements of torque on an anvil needed to deform the sample.

Figure 11 (next page). Results plotted vs. run time from a D-DIA experiment on a stacked sample of tantalum (starting length 1 mm) and MgO (starting length 1.5 mm), both polycrystalline. (a) Five traces showing, from top to bottom: measured hydraulic oil pressure in the top and bottom differential rams (scale on right-hand axis); combined centerward displacement of the two rams as measured by displacement transducers outside the sample assembly; shortening of the MgO portion of the sample; and shortening of the Ta component (scale for last three on left-hand axis). Sample length vs. time was measured directly using X-radiography. (b) Detail of the sample shortening, also indicating the strain rates (displacement rates normalized by sample length). The run was conducted by pumping hydraulic fluid to the differential rams at two different constant rate (hence deformation steps (1) and (2)), while draining fluid from the main ram in order to keep pressure at a constant level of 10 ± 1 GPa. Note that shortening rates of the samples were also approximately constant. The steady increase in differential ram pressure during both deformation steps is the result of steadily increasing friction on the gaskets squeezed between the differential and side anvils.

Yamazaki and Karato (2001) have improved these techniques by modifying the Drickamer-type high-pressure apparatus (Fig. 12). This apparatus allows large rotational shear deformation of a sample at higher pressures and temperatures. A Drickamer apparatus consists of a pair of anvils in a cylinder with a gasket material between the anvils and a thin disk of sample squeezed between the anvils to reach high pressure

Figure 12. Sketch of a rotational Drickamer apparatus (Yamazaki and Karato 2001).

Figure 13. Sample assembly for a rotational Drickamer apparatus. A thin disk of sample is sandwiched between two anvils. A sample is cut into two pieces and W5%Re and W25%Re foils are inserted vertically; these act both as strain markers and as a thermocouple. The change in shape of these foils can be measured after an experiment or during an experiment through X-ray absorption. A small cylindrical W3%Re at the center acts as an electrode for the heater and as one component of the thermocouple with a W25%Re foil.

(Fig. 13). The gasket provides an extra support for the anvils and, as a result, this apparatus yields significantly higher pressures than does a Bridgman apparatus without a gasket (Perez-Albuerné et al. 1964; Prins 1984). Because much of the force is supported by the gasket and the sample itself, the cylinder does not support a large load. Consequently, it is possible to make holes in the cylinder to provide a path for X-rays; the

thermocouple leads can be taken through these holes. The X-ray path feature is important for high-resolution stress and temperature measurements, both of which are critical for rheological studies. The Drickamer apparatus has recently been used for the *in situ* measurements of static properties using a synchrotron X-ray facility under conditions up to ~ 35 GPa and ~ 2000 K (Funamori and Yagi 1993).

In the Rotational Drickamer Apparatus (RDA) modification, one of the anvils is fixed with the frame, whereas another is attached to a rotational actuator. A gearbox with an ac-servo motor provides controlled rotation of one of the anvils. A sample is first pressurized (and heated), and then the rotational actuator is started. The sample is twisted between the two anvils, and the geometry of deformation is rotational shear. The strain (and strain rate) increases from zero to the maximum value, which is determined by the motor rotation speed and sample thickness. With the current motor and gear combination, a shear strain rate from ~ 0 to $\sim 10^{-3} \text{ s}^{-1}$ can be realized. This design has three advantages. First, the motion of the anvil for deformation is *orthogonal* to the motion of the anvil for pressurization. Therefore, pressurization and deformation can be clearly separated. Second, unlike axisymmetric deformation, the shape of the sample does not change appreciably during rotational deformation, allowing very large strain deformation and making possible studies of microstructural evolution. Third, because of the radial gradient in strain (strain rate), microstructural evolution and/or rheology at different strain-rates can be investigated in a single run.

The diameter of the anvil tip in the current RDA is 4 mm (sample diameter is ~ 1 -2 mm, thickness ~ 0.2 -0.4 mm). With a load of ~ 30 T, a pressure of ~ 15 GPa can be generated using tungsten carbide anvils. The maximum pressure is determined primarily by the strength of anvil materials (and the diameter of sample space) and is expected to be more than ~ 30 GPa when a harder material such as sintered diamond or cubic-BN is used. The rotational Drickamer apparatus constructed at Yale University has been tested at $P = 12$ GPa, $T = 1473$ K. A polycrystalline sample of MgO was deformed homogeneously to the maximum shear strain of ~ 3 . Two methods can be employed to estimate the stress magnitude. First, X-ray diffraction techniques can be used for deformation experiments with the RDA in a synchrotron radiation facility in the manner discussed previously. At this writing, such experiments are planned but have not yet been carried out. To adopt the diffraction technique for rotation geometry, a conical shape window is made in a cylinder to collect diffracted X-rays for a range of angles (Fig. 14b). One complication with this apparatus is the radial variation of stress. This is inevitable in the torsion test (e.g., Paterson and Olgaard 2000); Fig. 14a). In measuring the stress magnitude by X-ray diffraction, one must consider the spatial resolution. Important factors that control the spatial resolution are the cross-sectional area of the X-ray beam and the 2θ value used for diffraction (with energy dispersive mode). A smaller beam size and a larger 2θ are preferred for a better spatial resolution of stress measurements. However, a smaller beam size results in a weaker signal. Also the 2θ values are limited by the energy of X-ray and the d -spacings as well as the geometry of the apparatus (we will use ~ 10 -50 $\times$ 10-50 μm^2 beam [slit] size and $2\theta \approx 8$ -10 $^\circ$). To allow for control of 2θ , the apparatus is set on a mobile stage (with a remote control through a stepping motor) whose angle and position with respect to the beam line can be adjusted to optimize the conditions. The resolution of the stress measurement is also dependent on the detector. We anticipate that a spatial resolution of 100-200 μm and the stress resolution better than 0.1 GPa will be obtained by this technique. The use of a hollow specimen could help solve the problem of stress heterogeneity (Fig. 14d).

We note that this technique not only allows us to estimate the stress magnitude, but also provides information as to the grain-scale stress (strain) distribution in a polycrystalline material (either homogeneous stress or homogeneous strain (Funamori et

Figure 14. (A) Stress distribution in a sample in a torsion test (n is stress exponent, $\dot{\gamma} = A\tau^n$). (B) X-ray diffraction geometry. Stress magnitude can be estimated from the difference in d -spacing collected from two different diffracted beams through two windows that are cut at 45° with respect to the vertical axis. (C) A top view of a sample and the X-ray path. In order to determine the stress, X-ray diffraction must occur in a region of a sample where the shear direction is normal to the X-ray beam. (D) The geometry of a hollow sample, where stress heterogeneity is less.

al. 1994). Such information is useful in understanding the physics of deformation of polycrystals (Kocks 1970).

Another method is to use dislocation density as a stress indicator. This technique can be used when the deformation mechanism is dislocation creep and when stress has been held at a constant value for at least a few percent strain. The method does not work well if stress is inhomogeneous. We have recently used this technique for olivine and are able to estimate the stress magnitude with an uncertainty of ~ 10 -15% (Jung and Karato 2001; Karato and Jung 2002 in press). The reliability of such measurements for high-pressure phases is not as high as that for olivine because the calibration curve for dislocation density vs. stress relationship is not available. However, the relation between dislocation density and stress is nearly universal,

$$\rho = \alpha b^{-2} (\sigma / \mu)^2$$

(ρ : dislocation density, α : a constant of order unity, b : the length of the Burgers vector, σ : stress, μ : shear modulus (e.g., Kohlstedt and Weathers 1980) We consider that such measurements should provide at least semi-quantitative data on the stress magnitude.

We will perform both single-layer deformation experiments and two-layer deformation experiments. A single-layer deformation experiment is simpler to interpret, but the big advantage of a two-layer design is that one can determine the relative strength of two samples very accurately. Our experience with this apparatus showed that we can deform our samples to strains up to at least $\gamma \approx 10$ at ~ 15 GPa. Such a large strain is essential for the study of deformation fabric.

SUMMARY AND PERSPECTIVES

We have summarized some of the recent developments in quantitative characterization of stress and strain and the controlled generation of deviatoric stresses under high pressure (and temperature). These developments will allow us to explore

rheological behavior of Earth materials under much deeper conditions than heretofore possible. Such studies will, for the first time, provide the critical data sets necessary for understanding whole mantle dynamics through modeling and/or through the analyses of observations such as seismic anisotropy. Further technical developments need to be made to make these techniques robust. First, stress measurements using different techniques must be benchmarked. Three techniques are currently used to measure stress under high pressure: (1) Measurement of force using an external load-cell with a low-friction sample design (Tingle et al. 1993), (2) synchrotron X-ray measurement of orientation dependence of d spacing (Singh et al. 1998), and (3) inference from dislocation densities (Karato and Jung 2002 in press). Below ~ 4 GPa, all of these techniques can be employed to determine the strength of a standard materials such as olivine. A comparison of results of these measurements will provide a measure of reliability of each technique. Second, a number of issues still remain regarding the sample assembly. They include a proper control of thermodynamic environment, particularly water fugacity (or water content) and oxygen fugacity. Third, in almost all of these techniques under high pressure, it is almost inevitable to have deformation during pressurization. An experimental procedure (e.g., annealing; a very low strength medium surrounding the sample) must be established to minimize the effects of initial stage deformation.

It must also be emphasized that the sample preparation and characterization are as critical as careful mechanical tests. Samples with negligible porosity must be used and grain size and water content must be measured both before and after each experiment (control of other thermodynamic variables such as oxygen fugacity is also important; see Rubie et al. (1993)). In almost all laboratory deformation experiments, conditions are often close to the boundary between dislocation and diffusion creep (Karato et al. 1998), and, therefore, precise measurements of grain size are needed to interpret the data. Also, water content must be measured both before and after each experiment. Water is known to have significant effects on rheology, but its content in samples is difficult to control during high-pressure experiments.

Using the results of mechanical tests and sample characterization, dominant microscopic mechanisms of deformation must be identified. A meaningful comparison of *strength* of different materials (or for the same material for different conditions) can only be made when the deformation mechanism is the same, and the comparison must be made based on an appropriate *scaling*. For instance, if the dominant mechanism of deformation is diffusion creep, then the data must be compared by normalizing with respect to the grain size.

With the use of these new techniques (combined with careful characterization of samples and the analyses of deformation mechanisms), we will be able to cast first light on the rheology of the more than 90% of Earth's mantle that has not been accessible by quantitative studies. Results of such studies will be critical to our better understanding of dynamics and evolution of this planet.

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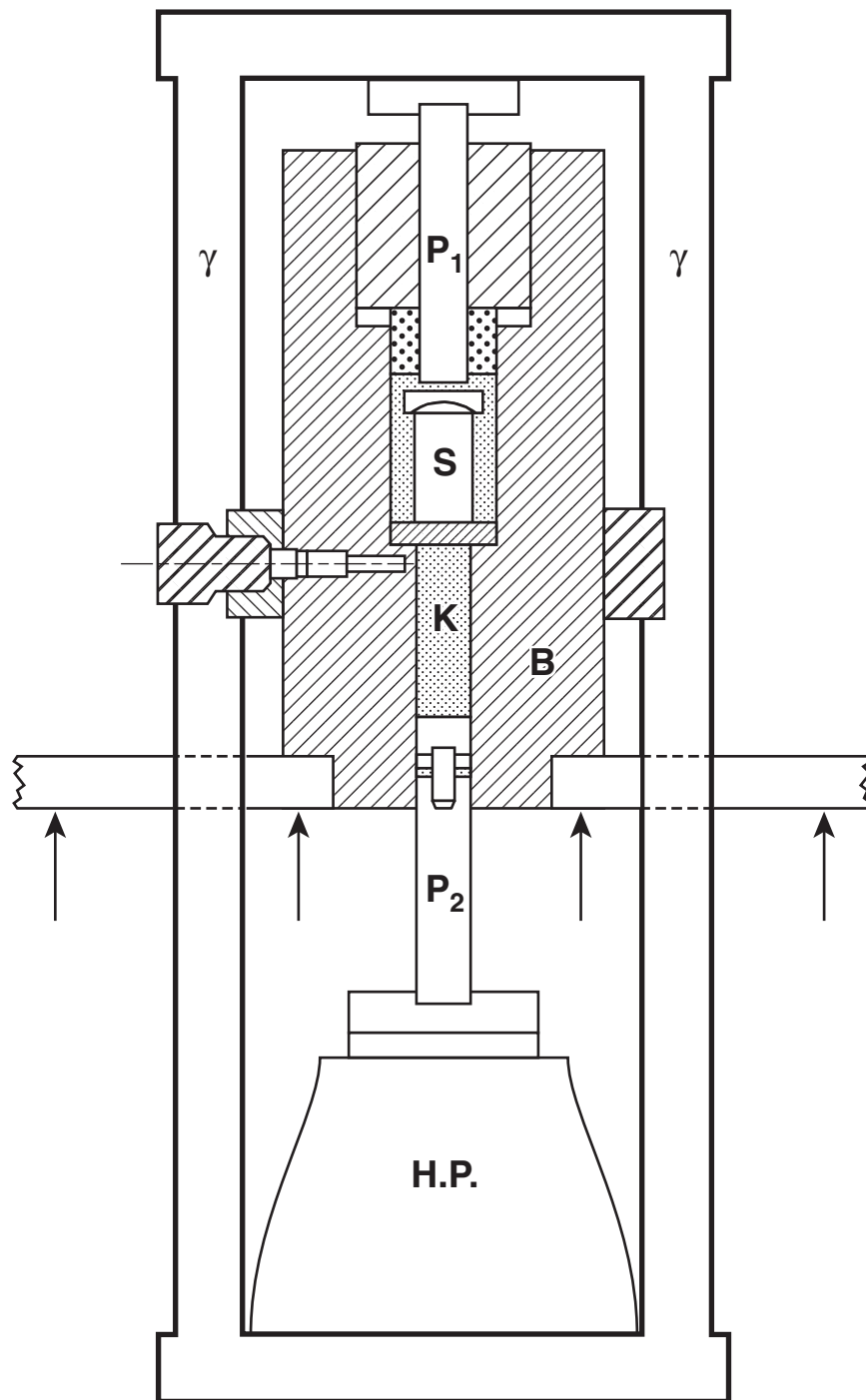


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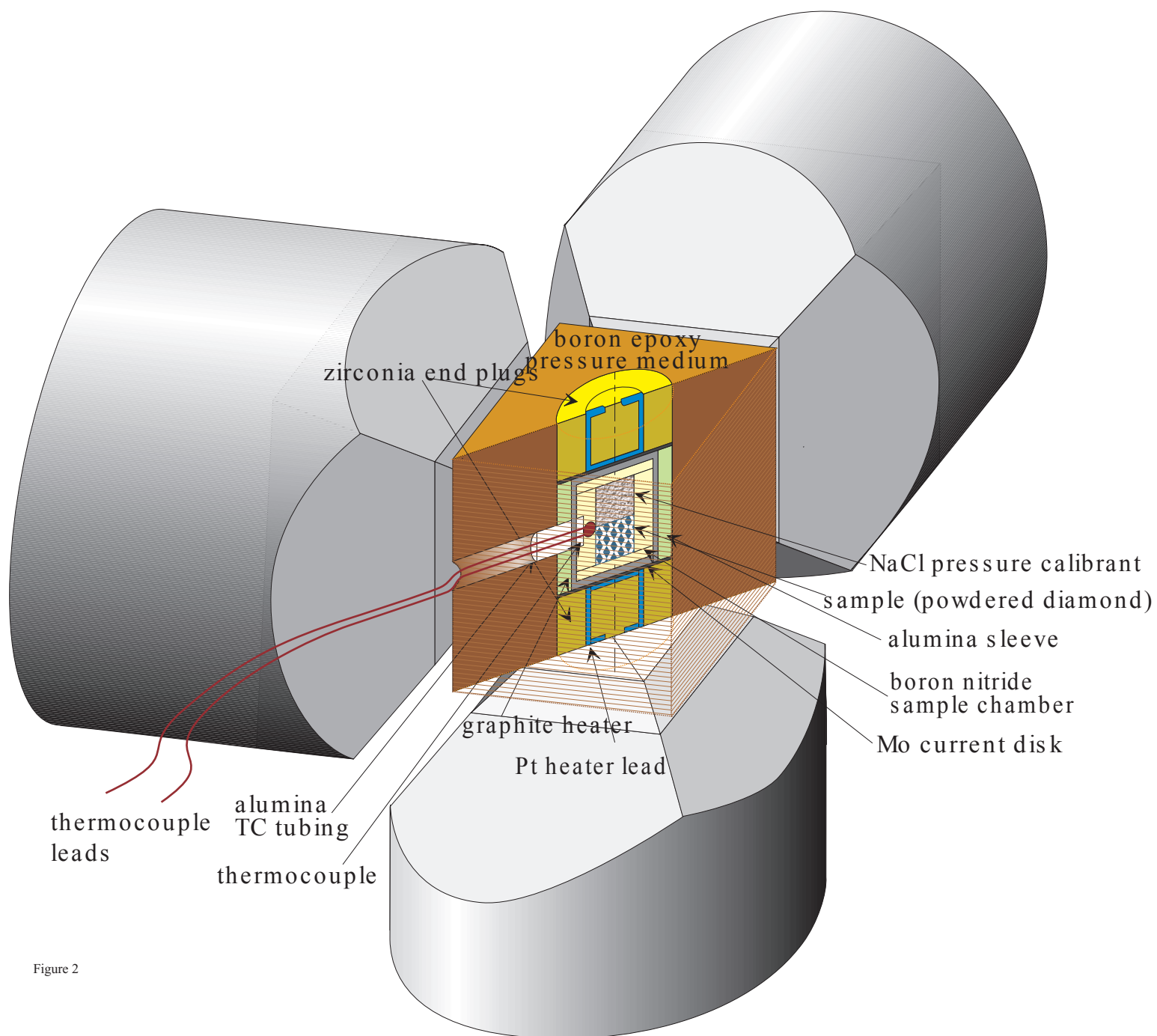


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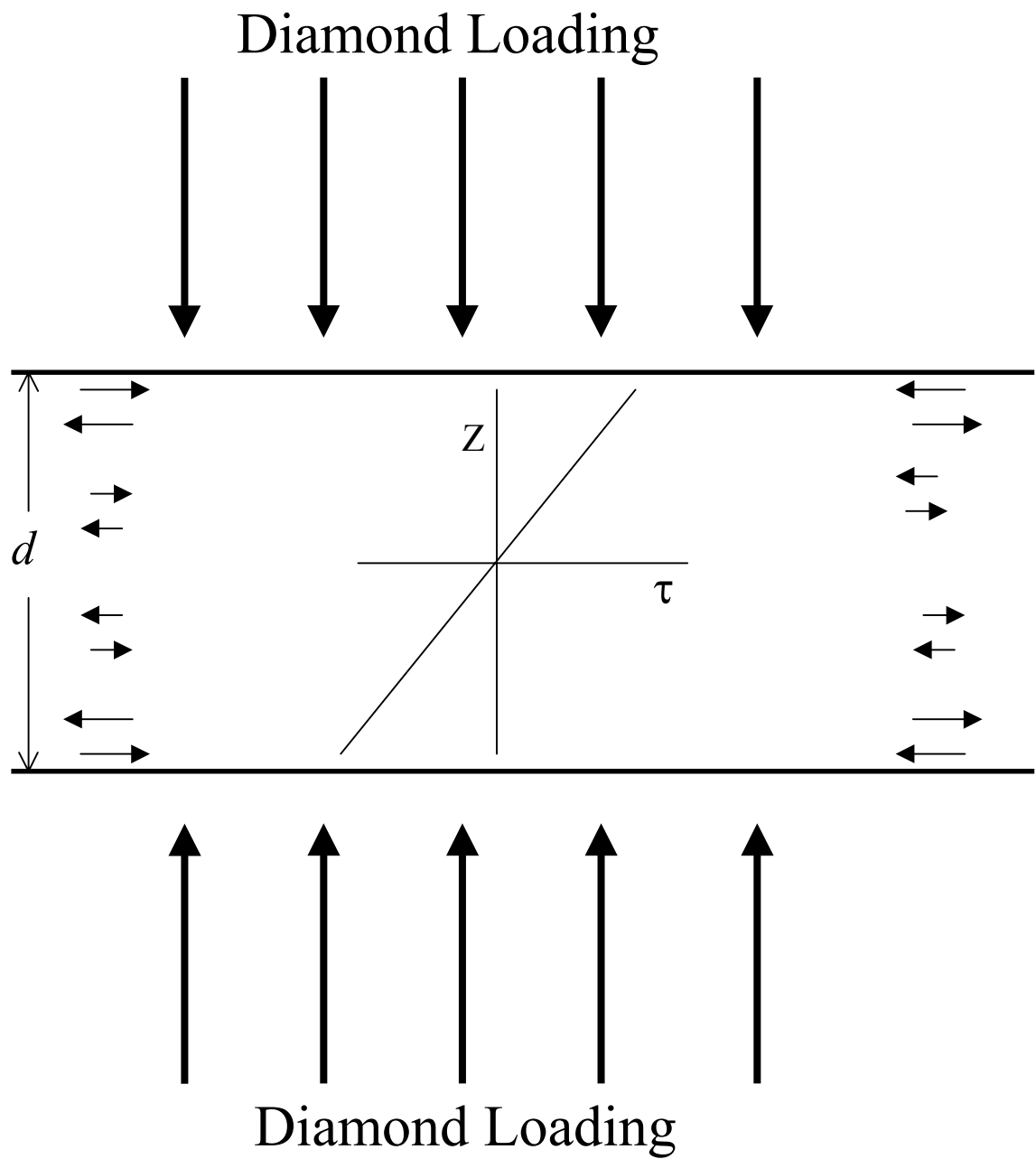
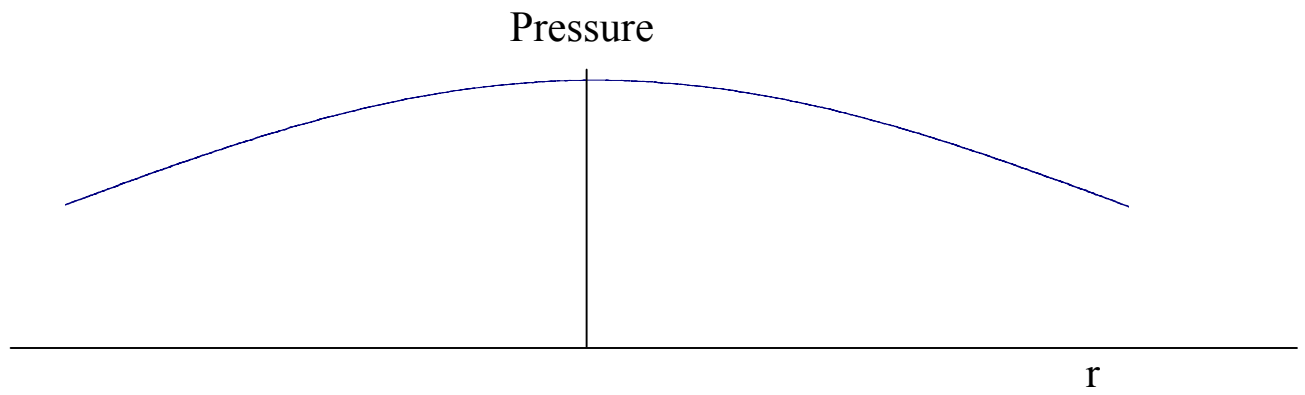


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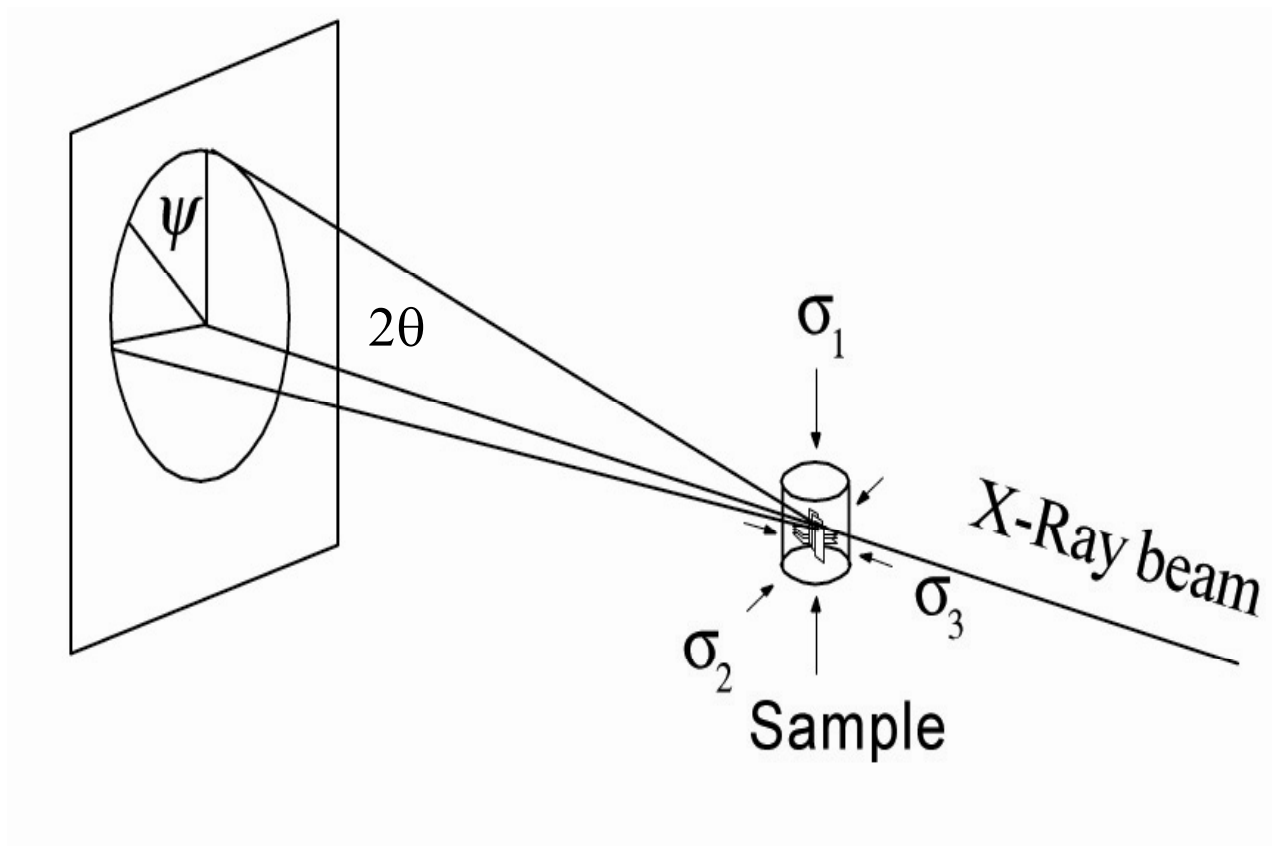


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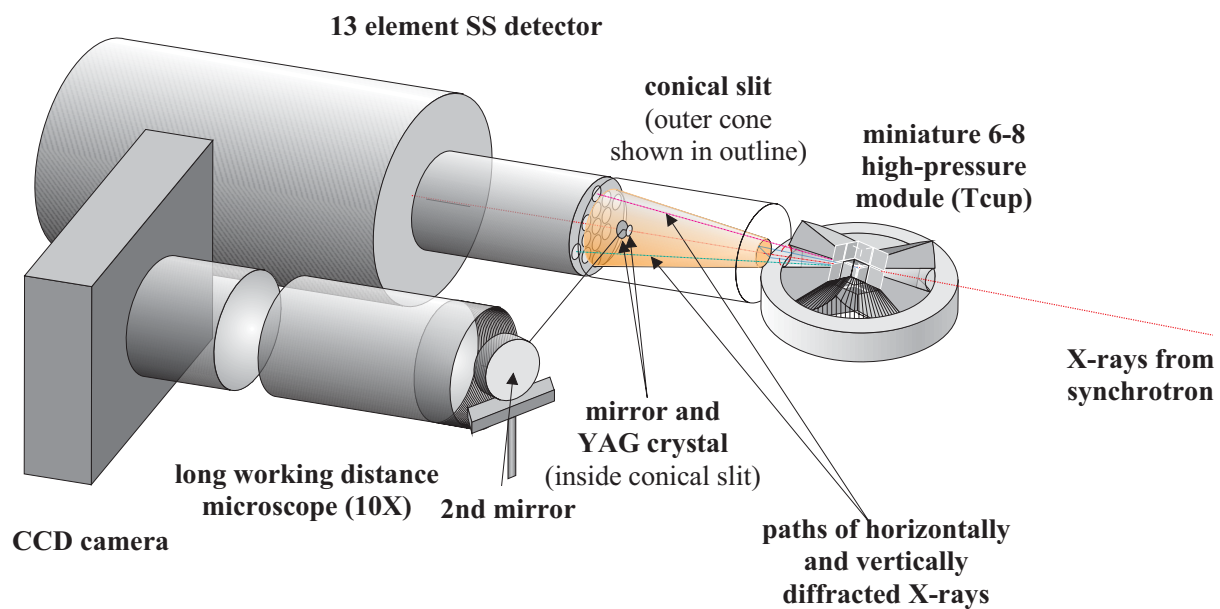


Figure 5



Figure 6

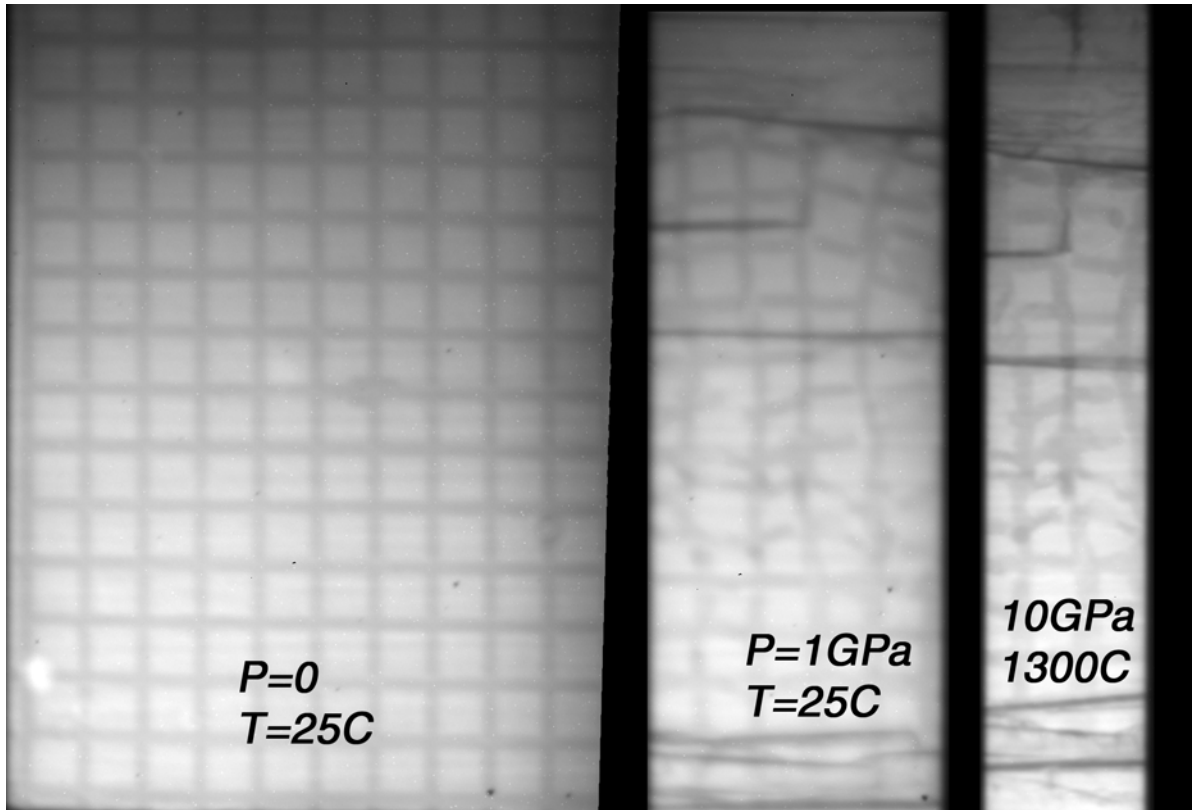


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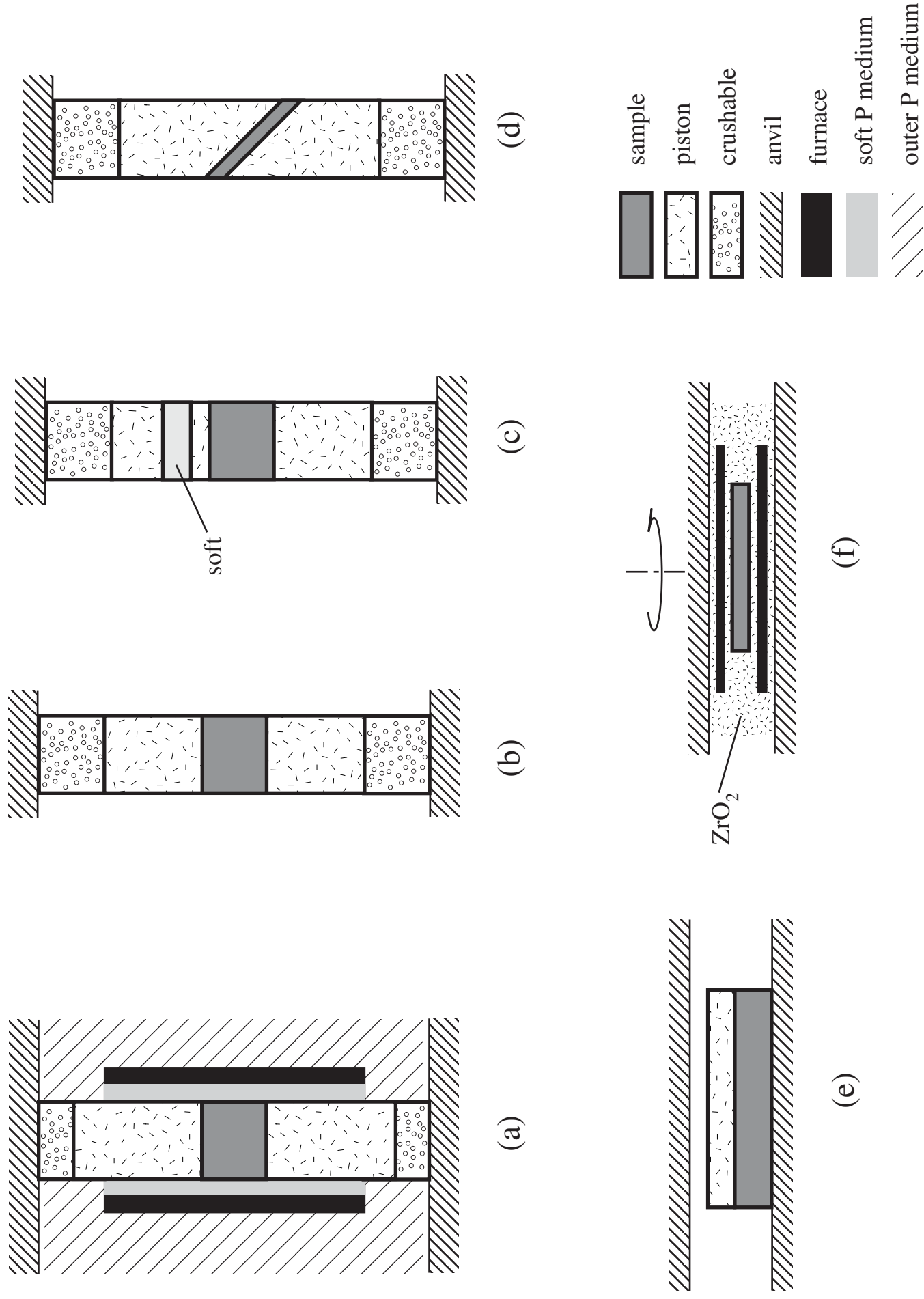


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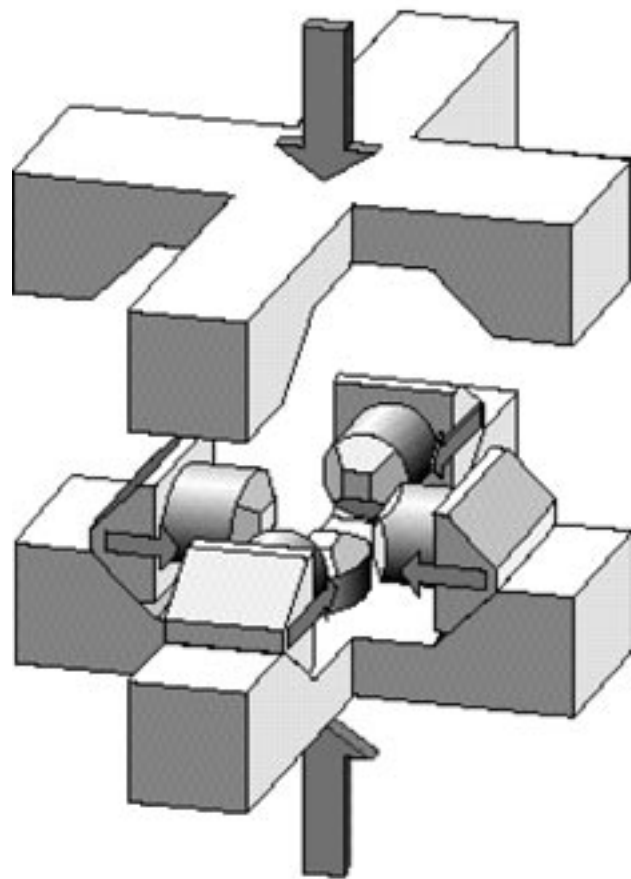


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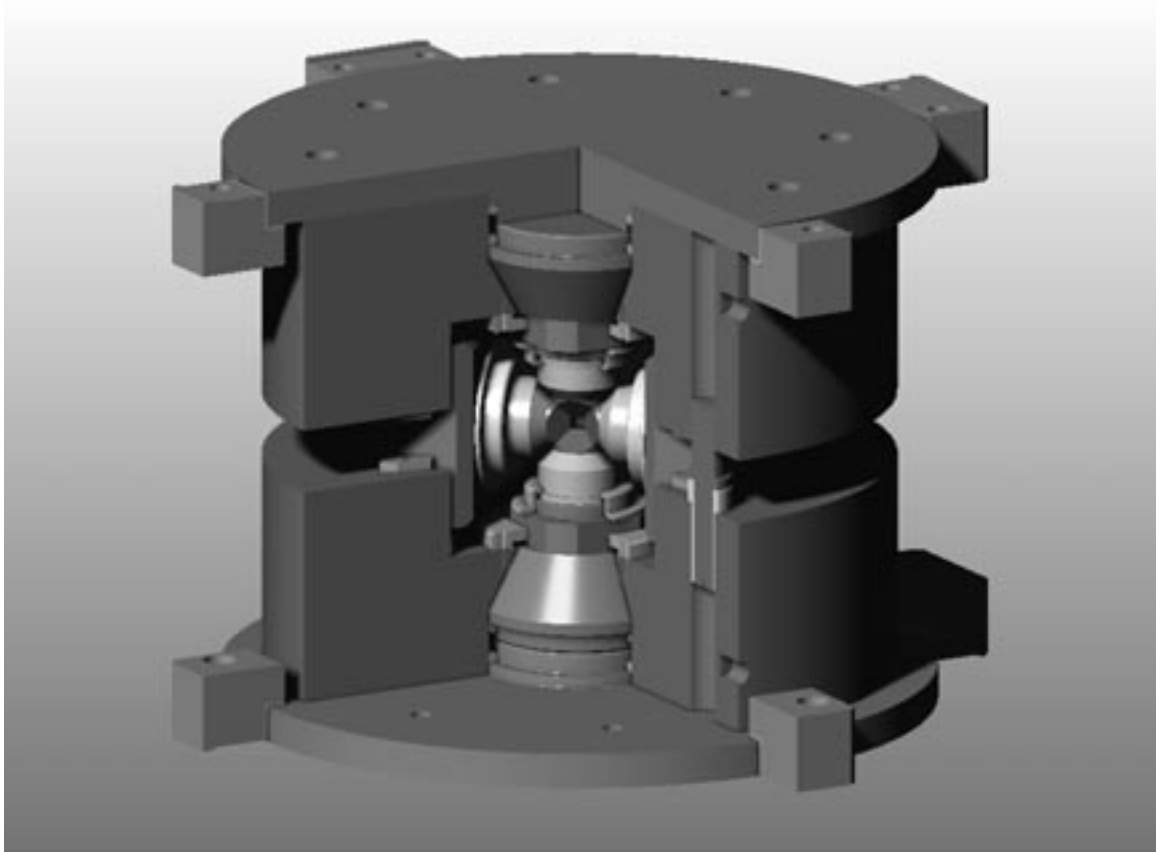


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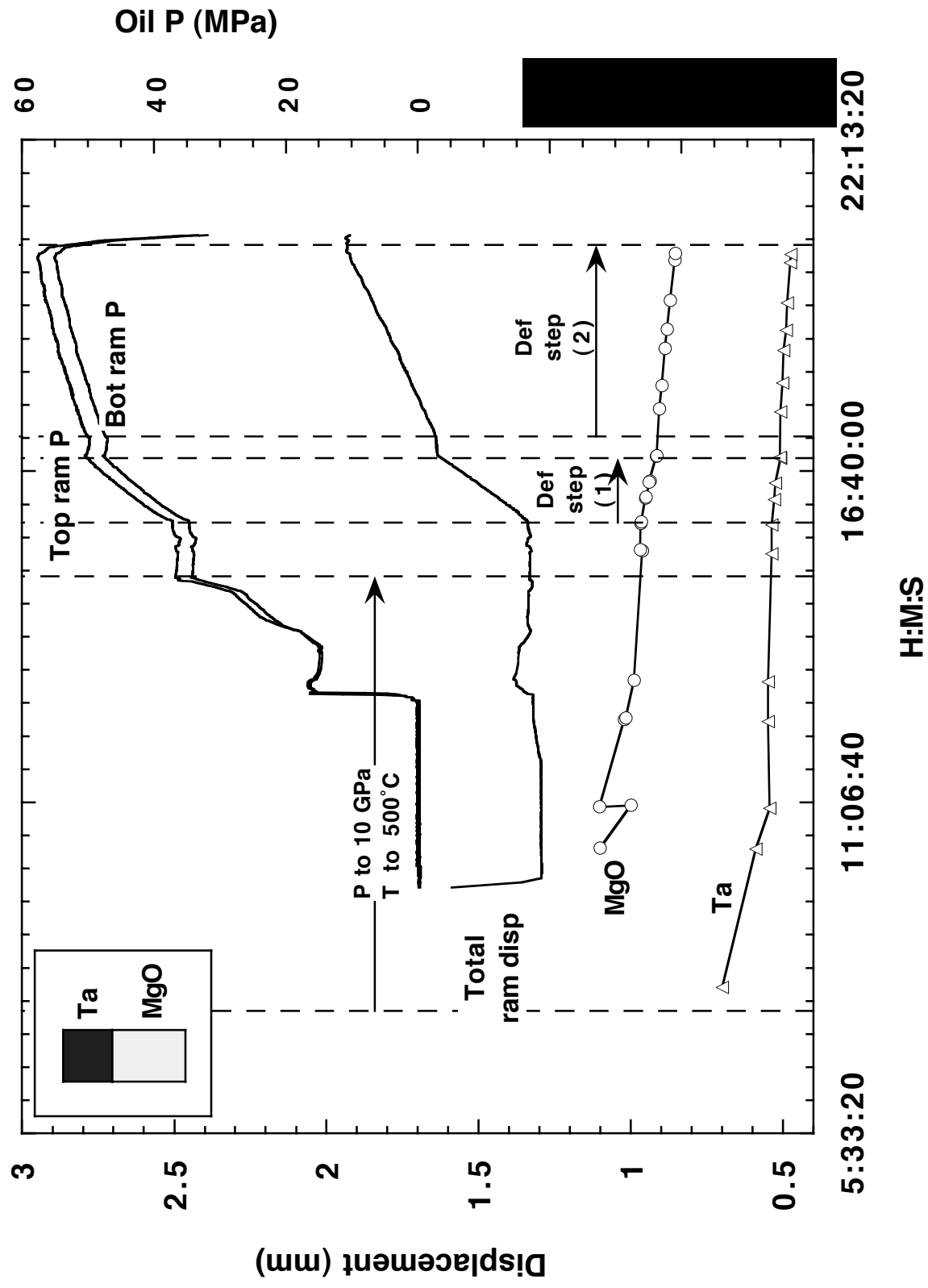


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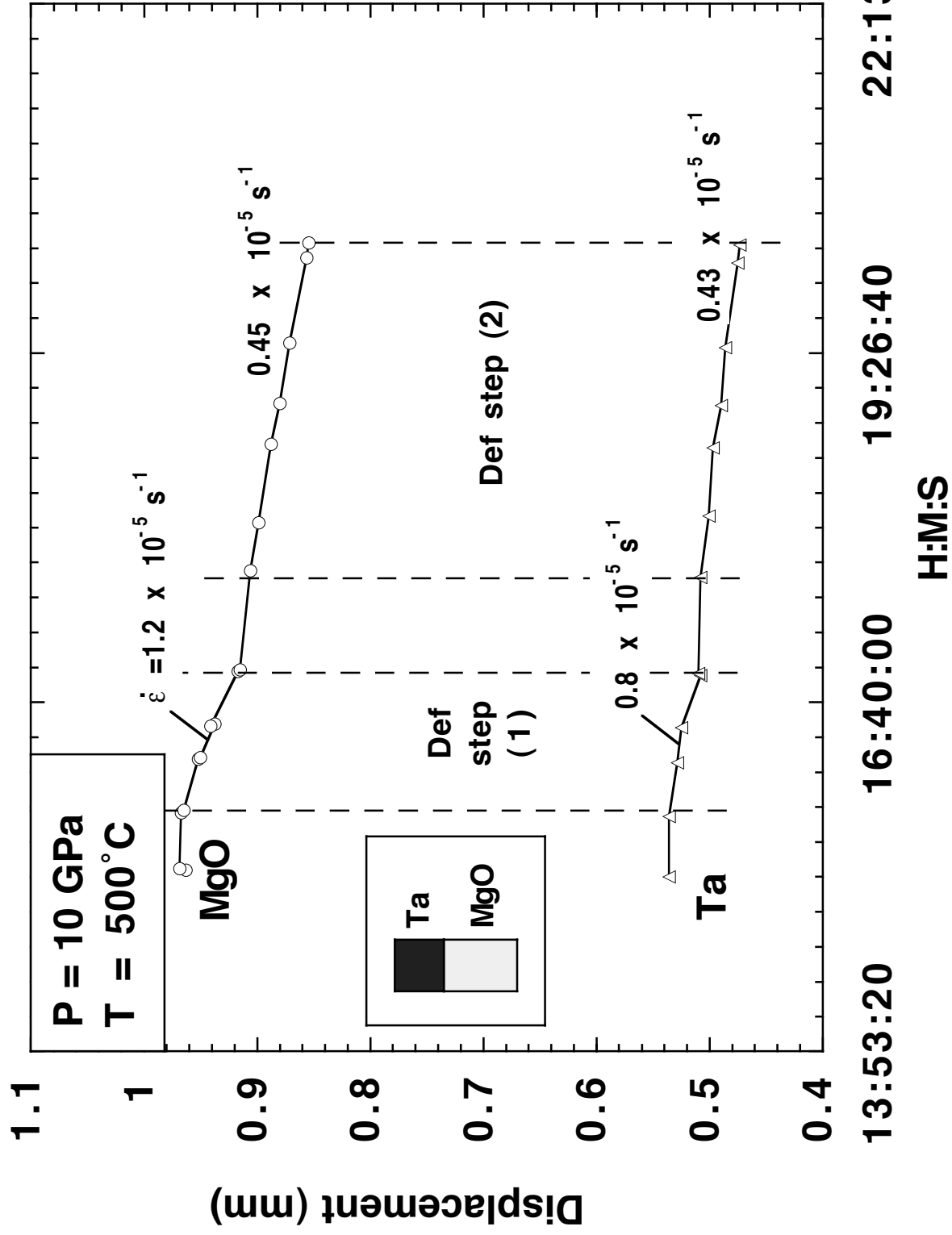


Figure 11b

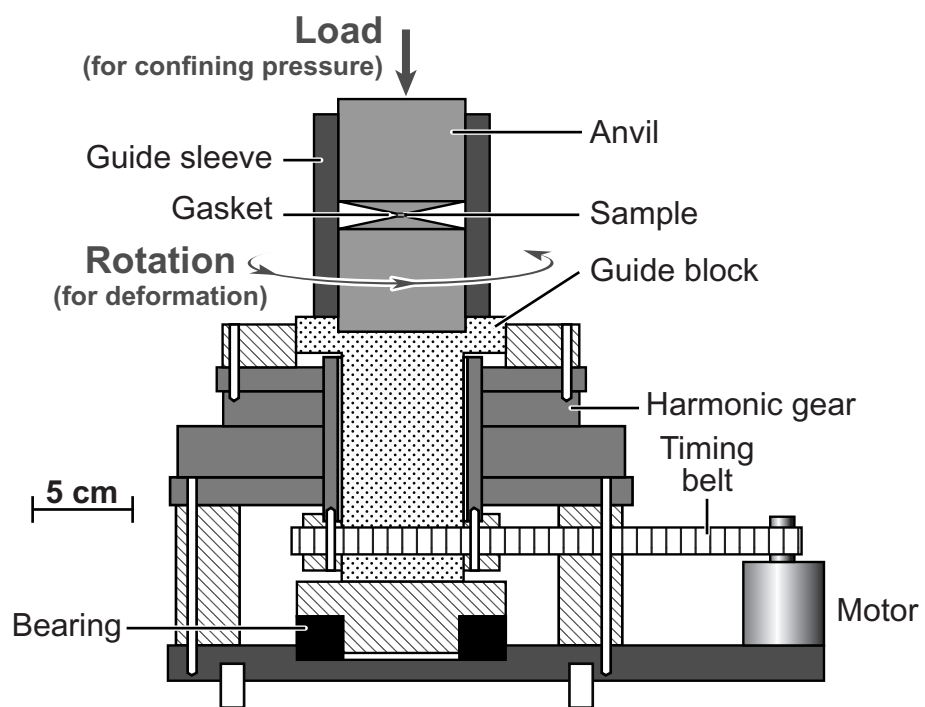


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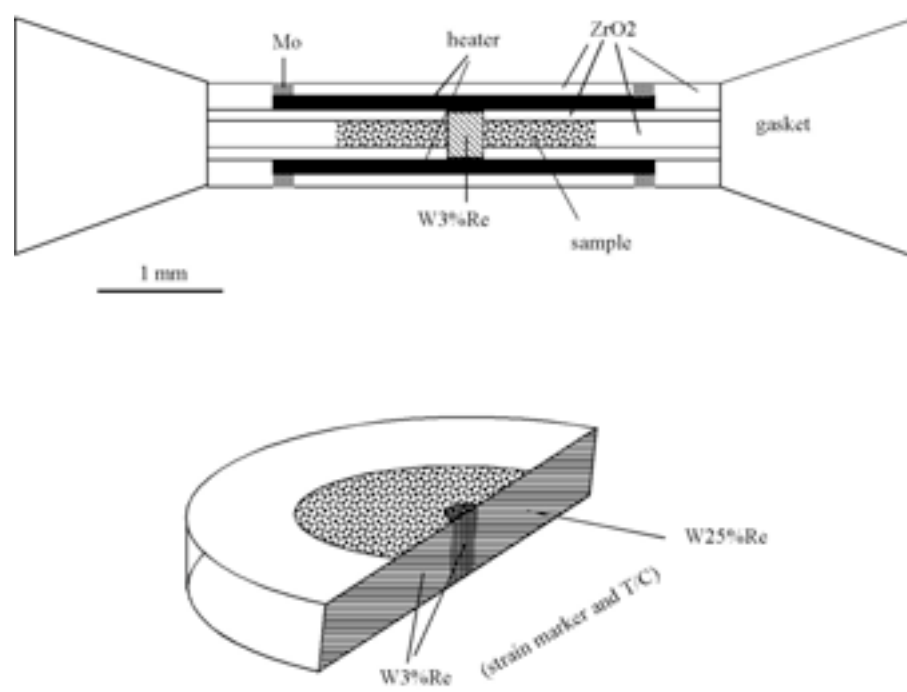


Figure 13

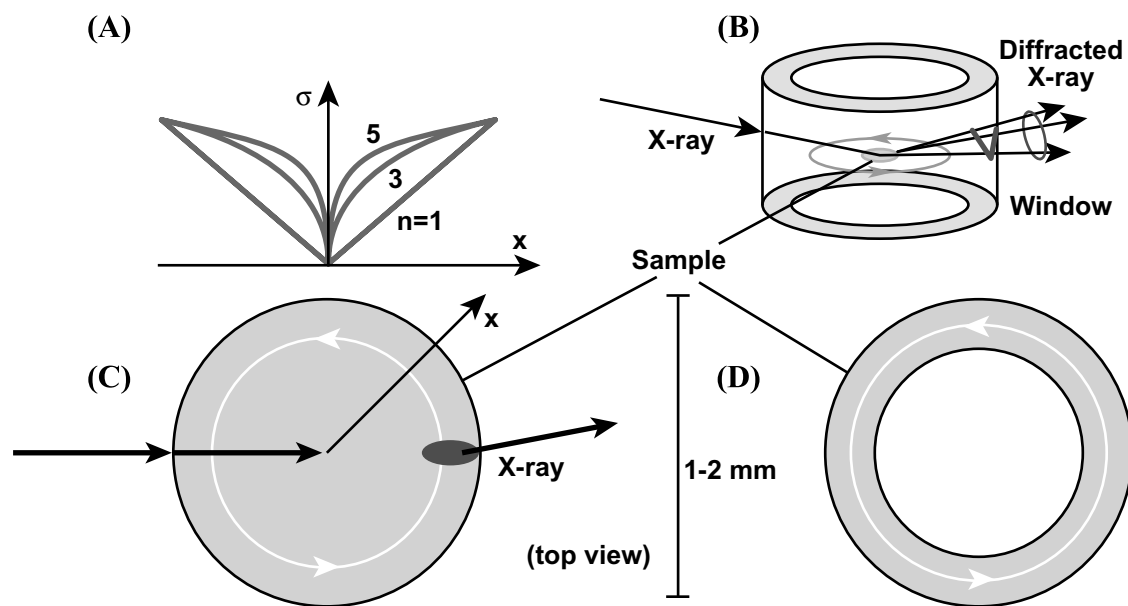


Figure 14