

# Pressure Core Characterization Tools for Hydrate-Bearing Sediments

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## Introduction

Natural gas hydrates form under high fluid pressure and low temperature, where biogenic or thermogenic gases are available. These requirements delimit the distribution of hydrate-bearing sediments to sub-permafrost, deep lakes (>390-m water depth) or ocean sediments (>320 m). Typically, hydrates are found beneath deeper water columns due to thermal fluctuations and diffusion near the sediment surface (Xu and Ruppel, 1999).

The clathrate or cage-like structure formed by water molecules hinders the repulsion between gas molecules allows for very high gas concentration. With the high methane concentration in large areas, natural gas hydrates can become an energy resource and remain a potential source for a potent greenhouse gas. Depressurization

and/or warming cause dissociation and volume expansion leading to large-scale sediment destructuration.

A proper characterization of hydrate-bearing sediments requires coring, recovery, manipulation and testing under pressure and temperature (P-T) conditions within the stability field. This report begins with an overview of existing tools, and then describes advances in pressure core technology developed at the Georgia Institute of Technology that have been advanced to address this need.

## Pressure Core Technology: Overview

The development of pressure coring and recovery tools have involved research teams around the world, including initiatives such as the International Ocean Drilling Program and the European Union's Marine Science and Technology Program (Kvenvolden et al., 1983; Pettigrew, 1992; Amann et al., 1997; Dickens et al., 2003; Qin et al., 2005; Schultheiss et al., 2009). A depressurization of cores will cause immediate dissociation of gas hydrates. It is therefore necessary to keep the samples at all times under P-T conditions within the stability field. Pressure core manipulation and transfer technology require a longitudinal positioner/manipulator and ball valves to couple components at equalized pressures (Pressure Core Analysis and Transfer System, PCATS; Schultheiss et al., 2006).

Contact testing tools utilizing P- and S-wave velocities, strength, electrical resistivity profiles and internal core temperature (IPTC; Yun et al., 2006), and non-contact tools utilizing gamma density, X-rays and water-coupled P-waves (Pressure Multi-Sensor Core Logger; Schultheiss et al., 2006; Abegg et al., 2008) are available. Subsampling capabilities have also been developed for biological studies under *in situ* P-T conditions (DeepIsoBUG; Parkes et al., 2009).

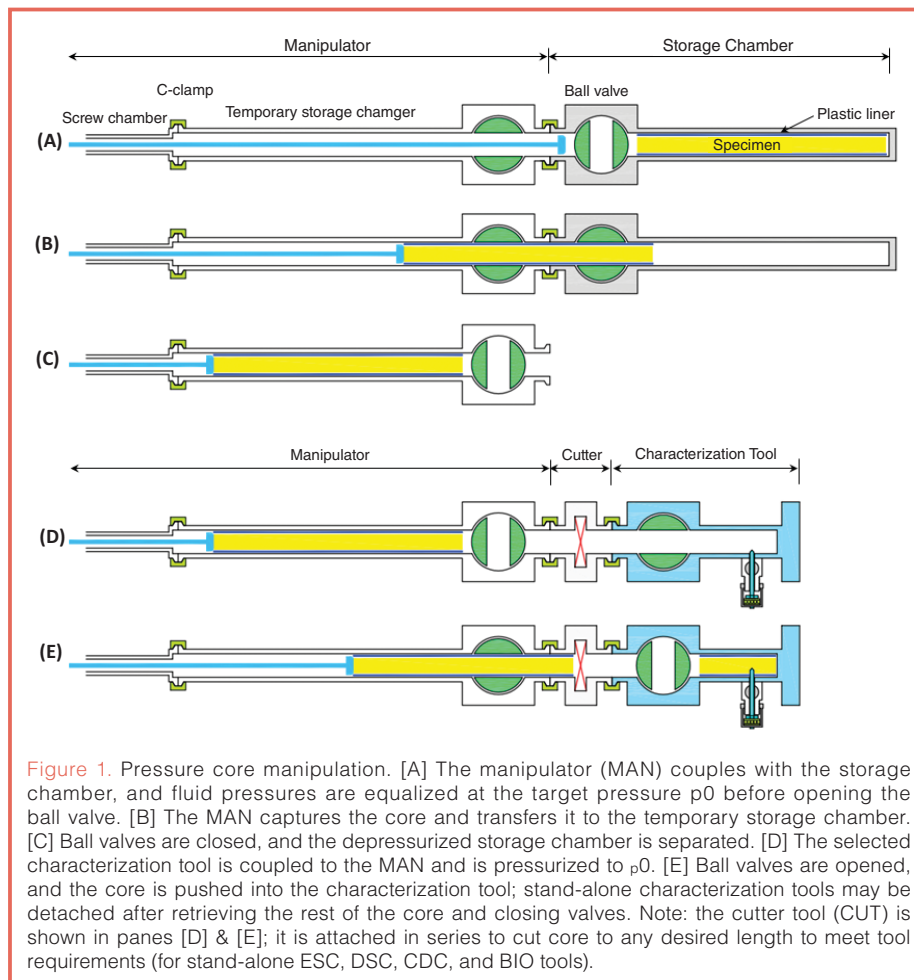


Figure 1. Pressure core manipulation. [A] The manipulator (MAN) couples with the storage chamber, and fluid pressures are equalized at the target pressure  $p_0$  before opening the ball valve. [B] The MAN captures the core and transfers it to the temporary storage chamber. [C] Ball valves are closed, and the depressurized storage chamber is separated. [D] The selected characterization tool is coupled to the MAN and is pressurized to  $p_0$ . [E] Ball valves are opened, and the core is pushed into the characterization tool; stand-alone characterization tools may be detached after retrieving the rest of the core and closing valves. Note: the cutter tool (CUT) is shown in panes [D] & [E]; it is attached in series to cut core to any desired length to meet tool requirements (for stand-alone ESC, DSC, CDC, and BIO tools).

## Pressure Core Characterization Tools (PCCTs)

Our pressure core characterization system includes core manipulation tools and characterization chambers. Tools have been selected to obtain complementary information relevant to science and engineering needs, with emphasis on the measurement of parameters used in hydro-thermo-mechanical analyses.

All tools are designed following key guidelines and objectives: simple and robust systems, portable components for fast deployment, modular design for maximum flexibility, standard dimensions and parts for affordable construction and maintenance, rust-resistance for seawater environment, capability of maintaining and operating at pressure, ability to impose effective stress, and safety for monitoring of hydrate dissociation and gas production during controlled depressurization, heating or fluid exchange (such as with liquid CO<sub>2</sub>). The modular design allows any two tools/chambers to be coupled through an identical flange-clamp system.

**Manipulator (MAN).** The manipulator is a longitudinal positioning system that is used to grab and move the core along the interconnected chambers and valves under the required P-T conditions. Figure 1 shows the typical operation sequence used to retrieve a specimen from the storage chamber into the MAN, followed by displacing the core into a test chamber. The geometric analysis of the operation shown in Fig. 1 reveals that the length of the MAN  $L_{man}$  (with its “temporary storage chamber”) is proportional to the length of the core  $L_{core}$  to be manipulated,  $L_{man} \approx 3.5 \times L_{core}$ . Our system is designed to handle 1.2-m-long cores ( $L_{core}$ ); it uses an internal telescopic screw system (stroke=2.6 m) driven by an external stepper motor, and it can position the specimen with sub-millimeter resolution. It is coupled to the 1.3-m-long temporary storage chamber by means of a dismountable flange-clamp connection. A see-through port is included to confirm the position of the MAN at any time.

**Sub-sampling.** The 1.2-m-long core can be cut into short specimens. Our cutting tool, CUT, houses either a linear or a ring-shaped saw blade within a clamp-type chamber. The saw-based cutting ensures clean surfaces and minimizes specimen disturbance. The CUT is mounted in series between the MAN and any other test or storage chamber as needed (Fig. 1d, 1e).

**Instrumented Pressure Testing Chamber (IPTC).** The chamber was developed to sample fluids and to measure P- and S-wave velocities, undrained strength, electrical

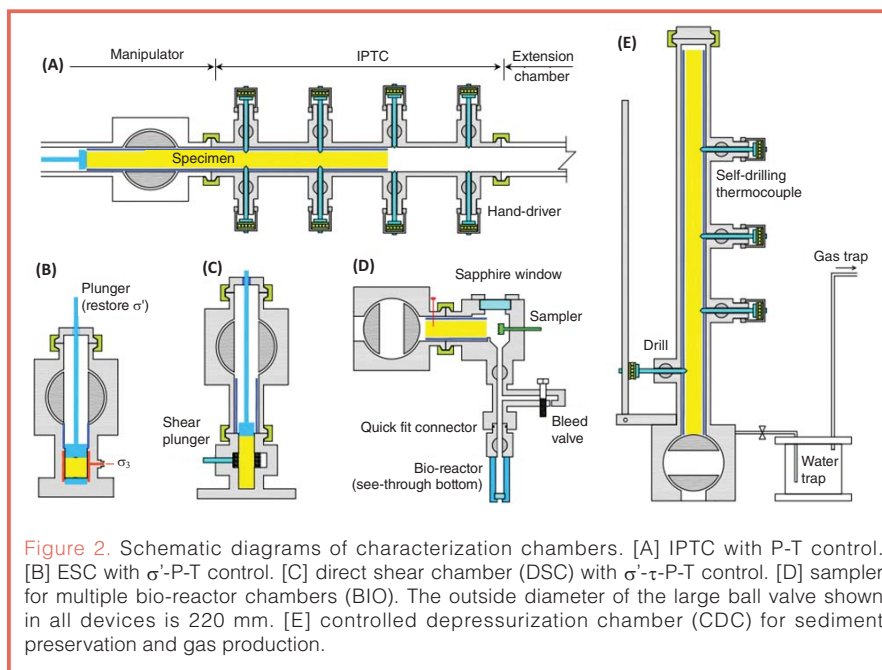


Figure 2. Schematic diagrams of characterization chambers. [A] IPTC with P-T control. [B] ESC with  $\sigma'$ -P-T control. [C] direct shear chamber (DSC) with  $\sigma'$ - $\tau$ -P-T control. [D] sampler for multiple bio-reactor chambers (BIO). The outside diameter of the large ball valve shown in all devices is 220 mm. [E] controlled depressurization chamber (CDC) for sediment preservation and gas production.

conductivity, and internal core temperature (Fig. 2a; details in Yun et al., 2006). Additional tool developments have been implemented by the USGS, within the context of the Gulf of Mexico JIP. This cylindrical chamber has two sets of four diametrically opposite port pairs. The first pair drills holes (ID=8 mm) in the plastic liner so that contact probes in successive ports can be pushed into the specimen. In characterization mode, the IPTC is coupled to the MAN on one side and an extension chamber on the other, and measurements can be conducted at any position along the core length. The eight access ports make the IPTC a versatile chamber for conducting well-monitored production studies in view of reservoir calibration models.

**Effective Stress Chamber (ESC).** Pressure cores are recovered and stored at fluid P-T conditions needed to preserve hydrate. However, physical properties such as stiffness and shear strength are functions of both hydrate saturation and effective stress, with the relative effective stress increasing as hydrate saturation decreases. The ESC maintains P-T stability conditions and restores the effective stress ( $\sigma'$ ) that the sediment sustains *in situ* (Fig. 2b). It was designed and laboratory-tested at Georgia Tech in 2006 under Joint Oceanographic Institutions (JOI) sponsorship, and it was first deployed in the field by the Korean Institute of Geoscience & Mineral Resource in collaboration with Geotek (Lee et al., 2009).

The original design was based on a zero lateral strain boundary condition. We have updated this chamber to accommodate a stress-controlled boundary condition using a jacket. The resulting triaxial stress configuration consists of  $\sigma_3'$  applied with the jacket and  $\sigma_1'$  applied by a piston that is advanced through the ball valve and acts directly on the pressure core. The piston and the base pedestal house the sensors needed for the measurements of physical properties,

including stiffness (wave velocities), thermal conductivity, and electrical resistivity.

A salient advantage of the flexible wall configuration is the ability to conduct precise fluid conductivity measurements by preventing the preferential flow along the sediment-steel boundaries in rigid wall chambers. This chamber is particularly well suited to monitor production studies under *in situ* effective stress conditions, including assessment of sediment volume change upon dissociation.

**Direct Shear Chamber (DSC).** Two constraints guided the design of the DSC tool. First, the imperfect boundaries that result when cutting heterogeneous cores under pressure cause stress concentration during vertical loading; thus, we selected a “double direct shear” geometry to cut across the specimen away from end effects. Second, overcutting during coring leaves a gap, and the core tends to tilt during shear; therefore, we adopted a double shear plane configuration to avoid bending action. Consequently, the DSC consists of a thick wall stainless steel ring that is pushed to shear the central third of the specimen (Fig. 2c). The DSC includes the piston to restore effective stress (similar to the ESC), a liner trap to capture the plastic liner before the specimen enters the shear chamber, and a small, lateral built-in frame to push the side piston that displaces the ring (Fig. 2c). The maximum shear displacement ( $\delta_{max}$ ) is 15 mm, allowing both peak and residual shear strengths to be determined. The

result is strength and volume change data under *in situ* conditions that are necessary for model calibration, production design, and stability analyses.

**Sub-sampling Tool for Bio-Studies (BIO).** Assessment of bioactivity in deep-water sediments without incurring depressurization cycles is crucial to the survival of some barophilic microorganisms. The BIO chamber is loaded with a core segment using the MAN; afterwards, it is detached from the MAN for all successive procedures (Fig. 2d). Its operation involves (1) nitrogen-liquid replacement, (2) core face cleaning and chamber sterilization, (3) sub-sampling using a rotary sampling head, and (4) sample deposition into the bio-reactor that is pre-filled with nurturing solutions (volume=10 mL). All operations can be observed through a sapphire window. Bio-reactors are readily replaced by closing a system of two ball valves and decoupling a quick connect fitting. This device allows the collection of a large number of specimens from a single core segment under *in situ* hydrostatic pressure.

**Controlled Depressurization Chamber (CDC).** Successful pressure coring operations may produce more pressure cores than the available storage. In this case, recovered cores can be selectively depressurized to conduct further studies under atmospheric pressure. The CDC is designed to help preserve the core lithology and to gain valuable information during depressurization, with minimal demand on personnel resources. This stand-alone device has a built-in drilling station to perforate the liner at selected locations in order to reduce the longitudinal expansion of the specimen. A pressure transducer and a thermocouple monitor the gas P-T conditions inside the chamber. In addition, three self-drilling thermocouples are deployed along the CDC; these are driven into the core to monitor the internal sediment temperature during depressurization. Finally, a 2-L water trap and a 55-L gas trap are attached in series to the needle valve that controls the rate of depressurization; these traps allow measurement of the water and gas produced (Fig. 2e).

### Measurement of Physical Properties

Multiple sensing systems have been developed to characterize the sediment and to determine hydrological, thermal, chemical, biological, and mechanical parameters within the chambers, under controlled pressure, temperature, and effective stress conditions as described

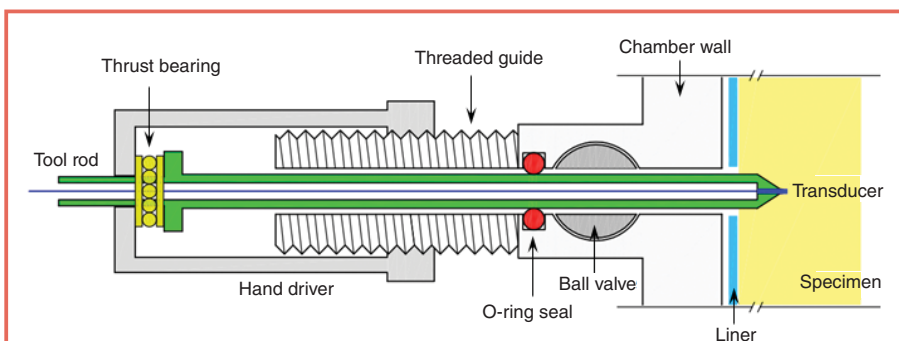


Figure 3. Tool Position. The displacement of sensors, subsampling tools, and drills are controlled under pressure using a screw-based positioning system where the driver advances along the threaded guide while pushing the tool rod (shown in green). Transducers at the tip of the rod are wired through the central hole in the tool rod.

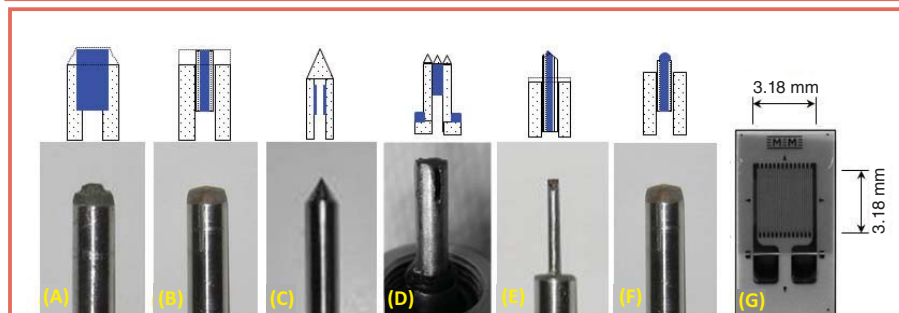


Figure 4. Measurement tools and sensors. [A] Bender elements for S-wave generation and detection. [B] Piezocrystals for P-waves. [C] Penetrometer for strength measurement. [D] Pore fluid sampler. [E] Electrical needle probe for resistivity profiling. [F] Thermocouple instrumented tip. [G] Strain gauge for thermal conductivity determination (TPS-NETL; Rosenbaum et al., 2007).

above. Their deployment in the various devices support the comprehensive characterization of natural hydrate-bearing sediments under *in situ* pressure, temperature, and/or stress conditions, and permit detailed monitoring of gas production tests.

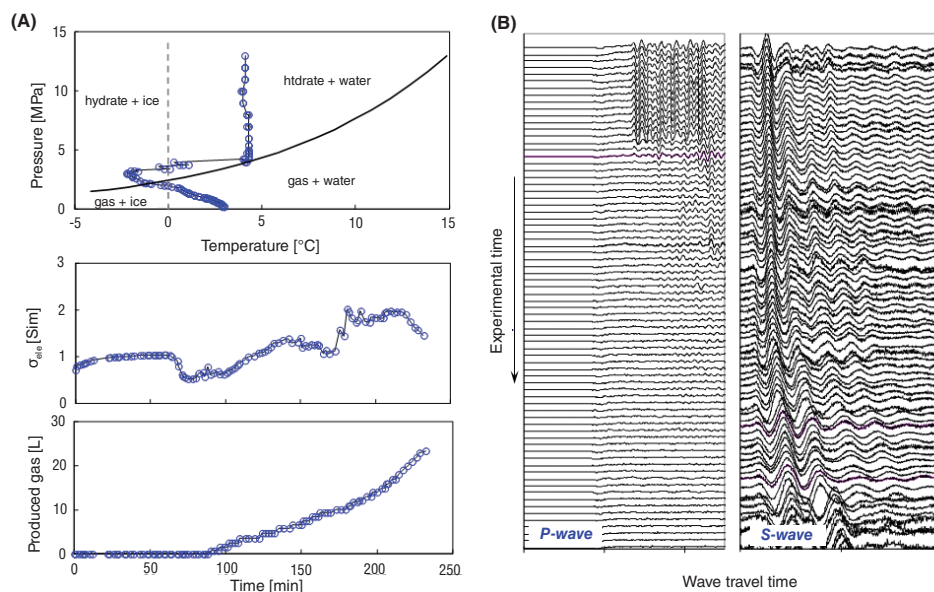
**Tool Position Control.** All contact instruments, sensors, and drills are mounted on polished rods (7.9 mm diameter) that are advanced into the specimen using externally controlled threaded positioning systems to overcome the 1.7 kN force at the maximum working fluid pressure of 35 MPa (Fig. 3). The ball valve between the threaded guide and the chamber permits replacing tools under pressure.

**Sensors.** Transducers are mounted at the tip of tool rods and wired through the central bore. Available instruments are shown in Figure 4. Small-strain wave velocity measurements employ bender elements for S-waves and pinducers for P-waves (Fig. 4a and 4b; peripheral electronics and test procedures as described in Lee and Santamarina, 2005a, 2005b).

While large-strain strength data can be gathered using the DSC (Fig. 2c), we have developed a strength-penetration probe as well (Fig. 4c). This device determines the sediment strength using a cone-shaped stud equipped with a full-bridge strain gauge inside. The measured tip resistance during probe penetration reflects the sediment undrained shear strength (Yun et al., 2006).

Fluid conductivity can be determined using the flexible wall system built within the ESC (Figs. 2b), and can be inferred using the fluid sampling tool (Fig. 4d). This is a self-drilling drainage port with a pressure or volume control to drive the interstitial fluids out of hydrate-bearing sediment. The pressure difference can be selected to preserve hydrates within stability conditions.

Electrical resistivity is measured using an electrical needle probe that is gradually inserted into the specimen to determine a radial resistivity profile with millimeter-scale spatial resolution (Fig. 4e; details and measurement procedure in Cho et al., 2004). We have also developed a multiple electrode system at the base of the effective stress cell that allows us to conduct a surface-based electrical resistivity tomography within a specimen.



**Figure 5.** Monitored gas production tests using IPTC: [A] Evolution of pressure, temperature, electrical resistivity, and produced gas (Krishna-Godavari Basin; Yun et al., 2010); [B] Typical wave signatures during gas production: P-wave signatures eventually fade out after gas production; S-waves detect the evolution of the skeleton shear stiffness during hydrate dissociation and gas production (Ulleung Basin; Yun et al., 2011).

The thermal probe consists of a thermocouple deployed at the tip of a tool rod. When pushed into the sediment, the thermal probe monitors the temperature inside the core (Fig. 4f). Internal temperature measurements can be used to monitor phase transitions during controlled gas production studies and to determine thermal conductivity. In addition, the transient plane source (TPS) sensor for thermal conductivity measurements—developed at the U.S. Department of Energy National Energy Technology Laboratory (Fig. 4g; Rosenbaum et al., 2007)—can be installed on the tools or on the pedestal of the ESC and DSC.

## Monitoring Dissociation – Gas Production

All PCCT chambers allow core-scale gas production tests by depressurization, heating, or chemical injection (e.g., inhibitors or carbon dioxide). Monitoring data include pressure, temperature, produced gas and water, stiffness (seismic wave velocities), fluid conductivity, and electrical resistivity. Figure 5 shows examples of data gathered during the depressurization of natural hydrate-bearing sediments.

## Conclusions

Pressure core technology is needed for the proper evaluation of natural hydrate bearing sediments. The set of pressure core characterization tools (PCCTs) described in this review allow the manipulation, sub-sampling, and extensive assessment of natural gas hydrate bearing sediments under *in situ* pressure, temperature, and effective stress conditions.

In addition to pressure core testing, comprehensive characterization programs should include sediment index properties analyzed within the framework of available data for natural hydrate bearing sediments, and tests with remolded specimens with synthetic hydrate. Pressure core technology can also be deployed to study other gas rich hydrocarbon formations such as deep-sea sediments, coal bed methane, and gas shales.

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