



## Examination of core samples from the Mount Elbert Gas Hydrate Stratigraphic Test Well, Alaska North Slope: Effects of retrieval and preservation

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### ABSTRACT

Collecting and preserving undamaged core samples containing gas hydrates from depth is difficult because of the pressure and temperature changes encountered upon retrieval. Hydrate-bearing core samples were collected at the BPXA-DOE-USGS Mount Elbert Gas Hydrate Stratigraphic Test Well in February 2007. Coring was performed while using a custom oil-based drilling mud, and the cores were retrieved by a wireline. The samples were characterized and subsampled at the surface under ambient winter arctic conditions. Samples thought to be hydrate bearing were preserved either by immersion in liquid nitrogen (LN), or by storage under methane pressure at ambient arctic conditions, and later depressurized and immersed in LN. Eleven core samples from hydrate-bearing zones were scanned using x-ray computed tomography to examine core structure and homogeneity. Features observed include radial fractures, spalling-type fractures, and reduced density near the periphery. These features were induced during sample collection, handling, and preservation. Isotopic analysis of the methane from hydrate in an initially LN-preserved core and a pressure-preserved core indicate that secondary hydrate formation occurred throughout the pressurized core, whereas none occurred in the LN-preserved core, however no hydrate was found near the periphery of the LN-preserved core. To replicate some aspects of the preservation methods, natural and laboratory-made saturated porous media samples were frozen in a variety of ways, with radial fractures observed in some LN-frozen sands, and needle-like ice crystals forming in slowly frozen clay-rich sediments. Suggestions for hydrate-bearing core preservation are presented.

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### 1. Introduction

Natural gas hydrates, hereafter called “hydrates”, are a class of clathrate compounds formed when water molecules encapsulate small gas molecules (Sloan, 1998). These compounds typically form at relatively high pressures and low temperatures where water and guest molecules (e.g., methane, ethane, propane) are present. Gas hydrates are studied for a variety of reasons including flow assurance in pipelines, energy recovery, climate change, geohazard and drilling safety, and gas storage/transportation. Recent estimates of the volume of hydrocarbons (predominantly methane) contained in gas hydrates span a large range (from 1 to  $5 \times 10^{15} \text{ m}^3$  Milkov, 2004 to  $120 \times 10^{15} \text{ m}^3$  at standard temperature and pressure Klauda and Sandler, 2005).

Acquisition and analyses of gas hydrate-bearing core samples is critical to advancing our understanding of the occurrence and behavior of gas hydrates in nature. Scientific and engineering observations and physical property measurements on these samples provide important insights into the characteristics and behavior of hydrate bearing sediments (HBS) and hydrate-bearing reservoirs. Ideally, samples would be collected without thermal, mechanical or chemical disturbance, (i.e. the samples would remain at *in-situ* pressure, effective stress, chemical conditions, and temperature). Obtaining pristine HBS samples is difficult because of the behavior of hydrates in the sediment and the differences in conditions between the *in-situ* sample and the earth surface.

Hydrate is stable only within a specific temperature and pressure range, and this range is affected by the (possibly evolving) pore fluid chemistry during core acquisition and handling. By a combination of decreasing pressure, increasing temperature, or increasing salt (or inhibitor) concentration, hydrate dissociates into gas and either

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water or ice. Conventional coring imparts significant pressure, thermal, and chemical perturbations to the HBS samples, therefore attempts are made to retrieve and preserve the samples as quickly as possible (Tulk et al., 1999) to minimize sample changes. In the arctic setting, cold ambient temperatures may help preserve hydrate, particularly if the drilling mud is chilled.

Hydrate can be present in a number of configurations in HBS. These include disseminated (which could be either pore-filling, grain-cementing, or a combination), part of the structural frame of the medium, in veins, nodules, and in massive layers (Sloan, 1998). Depending on its configuration, hydrate may either comprise a significant portion of the HBS providing a large portion of the mechanical, thermal, chemical, or physical properties of interest, or it may be limited to filling available pore space, having a very different impact on the overall HBS properties.

In this paper we describe HBS core retrieval from the BPXA-DOE-USGS Mount Elbert Gas Hydrate Stratigraphic Test Well (Mount Elbert Well) during drilling and coring operations that occurred from February 3 to 18, 2007. Eleven samples from 131 meters of conventional (non-pressure) wireline-retrieved core were preserved onsite and distributed to a number of laboratories for detailed study. Seven of these samples were placed in liquid nitrogen (LN) immediately after retrieval and description. Four others were initially preserved in methane-pressurized vessels and later depressurized and transferred to LN. We describe methods used to handle the core and preserve samples. In addition, we consider the mechanical forces, depressurization, heating, chemical changes, repressurization, and freezing mechanisms that disturb HBS. We show results from analytical tests and x-ray computed tomography (CT) scanning of a number of core samples to describe these cores, interpret the impact of handling procedures on core condition and gas hydrate occurrence, and recommend procedures for future gas hydrate core handling and preservation in sand-rich systems.

## 2. Core retrieval and core disturbance at the Mount Elbert Well

The Mount Elbert Well was drilled from ground surface to a casing depth of 595 m (1952 ft) using a 12¼ inch bit and water-based drilling fluids. At that point, a 9 5/8-inch casing was set, and drilling proceeded to a depth of 606.5 m (1990 ft) using a custom mineral-oil-based chilled drilling fluid. This fluid was used throughout the coring program, enabling on-site mud chillers to circulate subfreezing drilling fluid to mitigate down-hole gas hydrate dissociation and associated hole destabilization. In addition, the oil-based drilling fluid used during coring ensured that all water subsequently extracted from core samples was original pore waters. The well was continuously cored using a Doyon 14 rig to a depth of 760.1 m (2494 ft) with the ReedHycalog *Corion* wireline-retrievable coring system. Over this 153.5 m section, the 23 deployments of the system successfully recovered 131 m (430 ft) of high-quality 7.6 cm (3 inches) diameter core (85% recovery efficiency). Coring took place over 2 and a half days.

Extracted cores were laid out on the floor of the pipe shed at ambient arctic temperatures ranging from about  $-20^{\circ}\text{C}$  ( $-4^{\circ}\text{F}$ ) to  $-7^{\circ}\text{C}$  ( $20^{\circ}\text{F}$ ). From each coring run, the two 3.7 m (12 ft) sections of slotted aluminum core barrels were marked and cut into sections roughly 1 m (36 inches) in length. The individual sections were loaded into wooden boxes, and then transported via forklift approximately 11 m ( $\sim 35$  ft) to the unheated core examination trailer where the cores were laid out. Temperatures inside the trailer varied, but ranged typically from about  $-20^{\circ}\text{C}$  ( $-4^{\circ}\text{F}$ ) to  $-7^{\circ}\text{C}$  ( $20^{\circ}\text{F}$ ) during processing of the gas hydrate-bearing sections. The aluminum liners were cut open and the cores examined,

described, and subsampled. The estimated time from when the core was cut into sections to when the samples were stored varied from 20 to 45 min. Further details on the coring and core sampling programs are provided in Hunter et al. (2011) and Rose et al. (2011).

Infra-red (IR) imaging has been used in many marine gas hydrate coring programs to identify “cold spots” that may indicate locations of endothermic cooling from gas hydrate dissociation. IR imaging was not employed in this program due to the very cold ambient temperatures and the use of aluminum liners that spread temperature anomalies over a large area.

Cores were initially scraped to remove the rind of oil-based drilling fluids. Zones that were thought to be potentially gas-hydrate bearing at the time of subsampling were recognized via three primary methods; 1) temperature probes were inserted into the core approximately every two meters and ongoing temperature decline indicated the endothermic effect of gas hydrate dissociation; 2) small samples were taken and placed in liquid water, with visual release of gas bubbles indicating gas hydrate presence; and 3) gas-hydrate bearing zones were commonly observed to be well consolidated (would ring when struck with a hammer) as opposed to non gas hydrate-bearing zones, in which the sediments (particularly the sands) were often very poorly consolidated.

The cores were then subsampled according to a pre-established sampling plan. Small samples from inferred gas hydrate-bearing zones were placed in syringes to collect gas samples (see Lorenson et al., 2011). Eleven whole-round samples collected from sediments determined to be gas-hydrate bearing *in situ* were preserved in either pressure vessels (“HYPV”, 10-inch samples) or in immersed in LN (“HYLN”, 5-inch samples).

Four samples were cut to 10-inch lengths (Table 1), scraped clean, wrapped in aluminum foil, placed in pressure vessels (Parr Instruments, Moline, IL), and pressurized to 5.52 MPa (800 psi) with 99.99% pure methane gas. The vessels were then kept frozen at temperatures ranging from  $-40^{\circ}\text{C}$  to  $-4^{\circ}\text{C}$ . Prior to shipping from the well site, the vessels were inspected and it was noted that pressures had decreased to 4.48, 4.76, 4.83, and 5.17 MPa (650, 690, 700, and 750 psi), which is still above the stable methane hydrate stability pressure at  $0^{\circ}\text{C}$  of about 2.62 MPa (380 psi). This decrease in pressure can be explained by cooling, small gas leaks in the vessel seals or valves, or by hydrate formation, as the samples were maintained at pressures well within the hydrate stability zone. As a result of the pressure decline and the desire to maintain the samples within the stability zone, the vessels were further pressurized to 6.20 MPa (900 psi) then shipped overland to Anchorage, Alaska. Temperatures during transportation in late February ranged from  $-40$  to  $-10^{\circ}\text{C}$ . After arrival in Anchorage, core samples were stored in a refrigerated container maintained at  $-7^{\circ}\text{C}$ . On May 2, 2007, the pressure vessels, having pressures ranging from 4.48 to 5.17 MPa (650 to 750 psi.), were rapidly depressurized (in about 1 min), the samples removed, labeled, and placed into cloth bags and immersed in LN (taking 3–6 min), before shipment for CT scanning prior to shipment to other laboratories for further analysis.

The seven whole-round core samples placed directly in liquid nitrogen at the site (Table 1) were cut to 12.7 cm (5 inches) lengths, wrapped in aluminum foil, placed in cloth bags, and put into liquid nitrogen dewars. These dewars were stored out doors and shipped to Anchorage, and then shipped along with the four original HYPV samples.

Comparison of the hydrate sample collection depth with log data (Table 1; Fig. 1) shows that six of the samples were clearly taken within zones of high gas hydrate concentration (HYPV1, HYPV2, HYLN1, HYLN2, HYLN3, and HYLN4). The remaining four samples were taken at depths close to gas hydrate-water contacts within the C unit. Potential uncertainties in core to log depth

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