

**MICRO AND MACRO-SCALE INVESTIGATION OF CEMENTING  
CHARACTERISTICS OF GAS HYDRATES**

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**INTRODUCTION**

Gas hydrates are ice-like crystalline solids that are formed through combination of gas molecules in hydrogen bonded water molecule cages under certain pressure and temperature conditions. Gas hydrates are widely considered as potential energy source. Numerous gas hydrate settings in marine sediments and permafrost formations have been identified by seismic method, bottom simulating reflection (BSR). Recently, the Mallik 2002 Gas Hydrate Production Research Well Program presented significant success in gas production from naturally occurring hydrates, which makes the potential energy source more promising.

However, there are still some arguments over the global estimates of gas hydrates. This may be mainly attributed to the uncertainty of the survey methods that are applied to explore gas hydrate distribution. For instance, interpretation of seismic data strongly depends on gas hydrate cementation behaviours, as hydrate cementation will significantly alter the elastic property of marine sediments. Gas hydrates may act on marine sediments as a part of pore fluid, or a component of sediment framework, or inter-granular cement. However, the information on the cementing properties of gas hydrates are rather limited. Furthermore, extracting gas from gas hydrates in sediments may have an adverse effect on seafloor stability, which is also related to the cementation behaviour of hydrate crystals in sediments. As a result, it is essential to gain better understanding on the cementing characteristics of gas hydrates in sediments.

**TEST METHODS**

In this communication we report on the cementing characteristics of gas hydrates in porous media using micro and macro-scale investigation. Two different experimental rigs (developed at the Centre for Gas Hydrate Research, Institute of Petroleum Engineering, Heriot-Watt University) have been used in this work. They are glass micromodel and ultrasonic rig for micro and macro-scale investigation, respectively.

The kernel of the glass micromodel consists of two glass plates, i.e., base plate and cover plate. Different patterns of 3-Dimension micro pores (20-200  $\mu\text{m}$  pore size in width) can be etched on the base plate according to specific requirements, e.g., geometrical network for simulating porous media. The glass micromodel is mounted in a steel vessel that is subjected to a confining pressure, and surrounded by coolant connected to a temperature-controlled bath (-25°C to 75°C). The working pressure of the set-up is up to 40 MPa. Test fluids can be injected and withdrawn

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through the inlet and outlet by a Quizix pump. The set-up is equipped with a digital magnifying camera (up to 500 times of magnification) to take pictures and make video recording of the system during hydrate formation and dissociation. There is a cold light to provide required illumination through a piece of optic fibre.

The ultrasonic set-up consists of a cylindrical cell, two pistons (one fixed and one moving), ultrasonic signal system, digital indicator, gas separator and backpressure regulator, Quizix pumps and a personal computer. The test cell is surrounded by cooling jacket connected to a temperature control bath (-25°C to 75°C). The maximum working pressure is 40 MPa. It is possible to simulate various overburden pressure conditions by injecting water behind the moving piston. Pore pressure is controlled by another set of pumps adjusting fluid inlet and outlet. The digital indicator is fixed to the rod tail of the moving piston to determine the displacement during depressurisation and compression, or expansion due to gas hydrate formation/dissociation. The ultrasonic system consists an ultrasonic pulser/receiver and a digital storage oscilloscope measuring the acoustic characteristics of P-wave and S-wave through the sediment samples in the presence/absence of hydrates. The gas separator and backpressure regulator are used for measuring gas production by controlled depressurisation using the Quizix pumps. The test data including pore pressure, overburden pressure, temperature, displacement of the piston, and waveforms of ultrasonic signals are acquired by the personal computer. The computer is also used to control the digital storage oscilloscope and the Quizix pumps. The ultrasonic set-up can be used in investigating various aspects of hydrates in sediments, including; geomechanical properties of sediments, detecting and quantifying gas hydrates using physical techniques, examining the effect of sediment size and/or mineralogy and/or overburden pressure on the hydrate stability zone, simulating various hydrate formation and dissociation (e.g., production) scenarios, kinetics of hydrate formation and dissociation, etc.

## RESULTS

In the micromodel tests, the cementation behaviour of hydrates was visually observed using three different gases as hydrate formers, including methane, a natural gas and carbon dioxide. Tetrahydrofuran (THF) was also used to simulate massive hydrate formation. Figure 1 shows gas hydrate cementation features in the pores. In Figure 1a, methane hydrates are formed at 6.5 MPa and 0.2 °C from dissolved gas. As shown in the figure, methane hydrate particles do not seem to adhere to the glass wall and were dispersed inside the pore. Furthermore, the hydrates have a more geometrical shape due to slow rate of hydrate formation, as a result of low solubility of methane in the aqueous phase. Figure 1b shows hydrates formed from a natural gas mixture at 7.6 MPa and 1 °C. Again hydrates are formed from dissolved gas only. However, more hydrates are observed here, partly due to higher solubility of the natural gas in the aqueous phase, as well as flowing water saturated with natural gas through the micromodel, in an attempt to simulate natural convection and advection in subsea sediments. At the final stages of the test, the flow of natural gas saturated water was temporarily stopped for few hours to give time to the system to stabilise. After commencing the flow of natural gas saturated water, further hydrate formation was observed, partly in the form of appendix to the existing gas hydrates (as identified with the arrows). As shown in the figure, hydrates seem to grow inside the pores and can provide cementing characteristics when the pore space has a high saturation of gas hydrates. In the third series of the tests carbon dioxide was used. To further enhance the formation of gas hydrates, carbon dioxide in the form of free gas was also present in the micromodel. As shown in Figure 1c, hydrates are formed in the water phase, gas-water interface and the gas phase. The hydrates formed in the water phase (i.e., excess water environment) tend to form within the pore space.

However, as a result of massive hydrate formation significant cementation characteristics is observed. In the last series of the tests, THF was used to further increase the amount of hydrates formed. As shown in Figure 1d, some pore spaces are filled with gas hydrates, without any detectable free water. Therefore, in the THF test where hydrate saturation are very high, they seem to cement the grains together. The above results show that in the case of glass micromodel, hydrates prefer to form inside the porous media and cementation occurs when the hydrate saturation is very high.

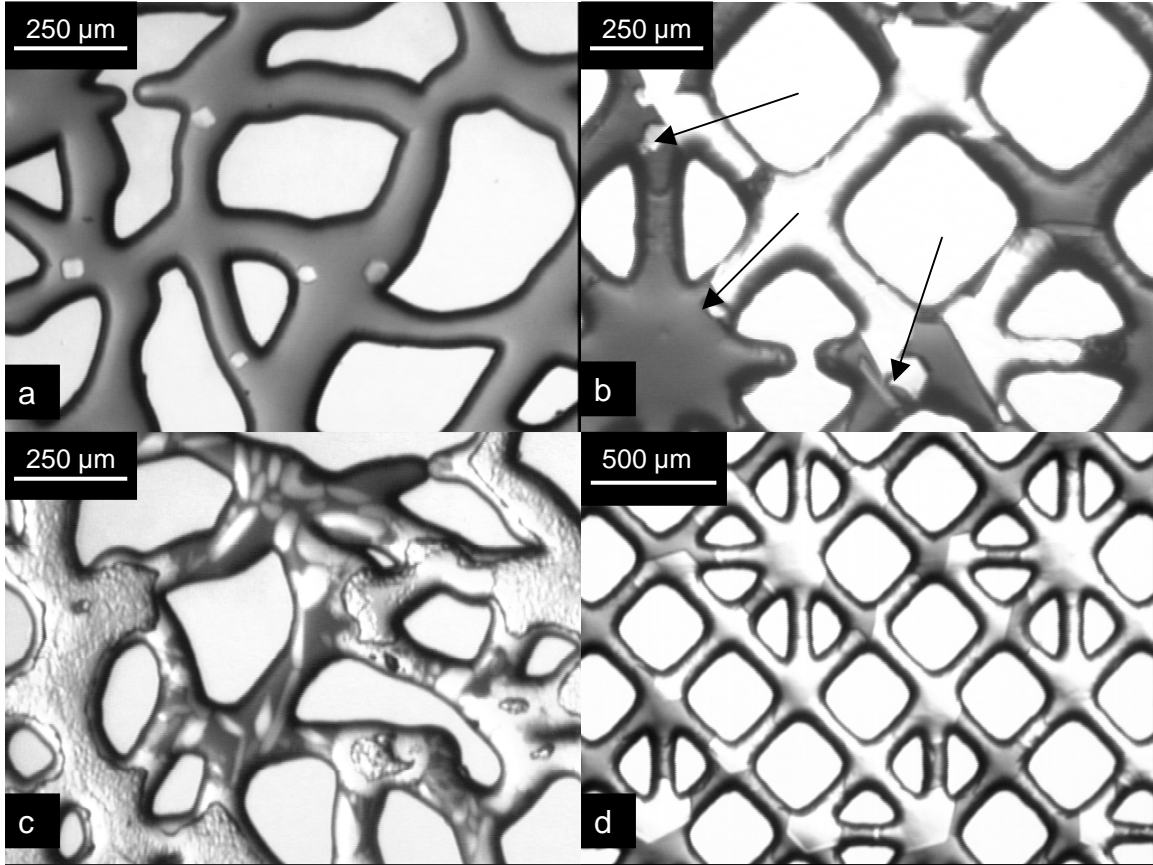


Figure 1: Cementation behaviour of gas hydrates in the glass micromodel tests.

Another series of experiments were conducted using the ultrasonic rig with the objective of understanding the cementation behaviour of hydrates in macro-scale. In these experiments, THF at various concentrations (to control the amount of hydrates formed) in water were used as hydrate former. This was to achieve the required high saturation of hydrates, and avoid any effect of remaining free gas on the frequency spectrum. Glass beads of 0.1 mm in diameter were filled into the test cell and compacted by applying certain overburden pressure to simulate unconsolidated sediments. In the experiments, after compaction the porosity was approximately 41%. Hydrates were formed at similar conditions of 0.4 °C, pore pressure of around 7 MPa, and overburden pressure around 14 MPa.

Acoustic characteristics before and after hydrate formation were monitored. Here we report the resulting changes in Fast Fourier Transform (FFT) of acoustic signals. Figure 2 shows the results in frequency spectrum. In Figure 2, the dashed lines presents the base FFT that was carried out with the same glass beads pack saturated with water, while the solid line presents the FFTs of THF hydrate-bearing sediments at different saturations. The FFT gave different widths and the

widths increased with hydrate saturation, and all the widths except that with approximately 100% pore volume filled with hydrates were narrower than the base FFT. This means that the presence of THF hydrates in pores dominantly scattered the acoustic waves. This may suggest that THF hydrates mainly acted as discrete particles or small lumps (probably as observed in the micromodel tests) that scattered the acoustic energy, although one of them had a hydrate saturation of around 79% of the pore volume.

The FFT of the experiment with 100% pore volume fill with hydrates became wider than the base FFT, and the maximum peak of its FFT shifted to higher frequency. This suggests that only if hydrate saturation was high enough, for example, nearly full of all the pore space, a complete cementing effect could be expected, as shown by the last curve in Figure 2.

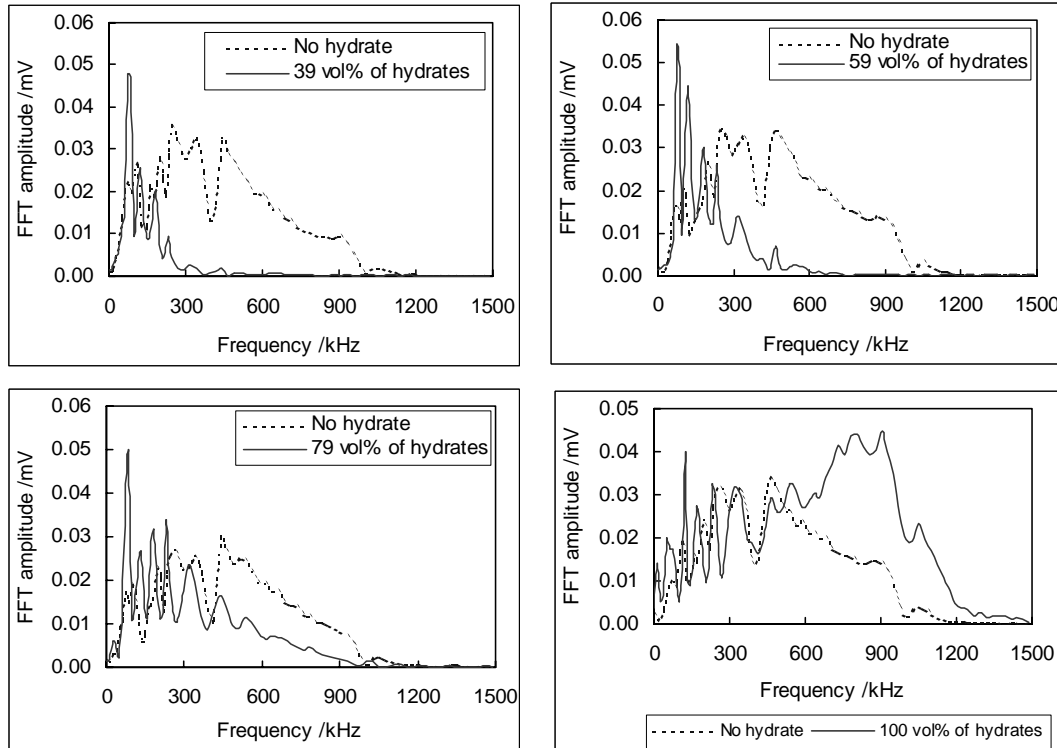


Figure 2: FFT response as a function of the amount of THF hydrates in the glass beads.

## CONCLUSIONS

Visual observation in the micromodel experiments shows that gas hydrates and THF hydrates seem not to adhere the pore walls. Hydrate crystals are likely to form in the centre of the pores and cementation could only occur at high hydrate saturations.

In the ultrasonic experiments, FFT response to the amount of hydrates in the glass bead pack suggests that hydrates more likely act as discrete particles or small lumps and did not tend to cement glass beads together when hydrate saturation was insufficiently high. However, hydrate cementation may be expected when hydrate saturation was very high.

It should be noted that silica glass has different chemical and mineral surface properties, which may result in different cementation features in comparison to some in-situ sediments.