Annual Report: Deepwater and Ultra-Deepwater Research

30 September 2013
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Ultra-Deepwater

Office of Fossil Energy
NETL-TRS-UDW-2013
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Cover Illustration: Oblique map view of the Gulf of Mexico, looking to the northeast and illustrating the distribution and projected depths of existing ultra-deepwater (orange), deepwater (pink), and shallow water wells (black) in U.S. waters. Water bathymetry is shown in blues, land surface features are shown in greens. (Image credit: NETL)


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Annual Report: EPAct Deepwater and Ultra-Deepwater Research

Office of Research and Development, National Energy Technology Laboratory

NETL-TRS-UDW-2013
30 September 2013

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<th>Description</th>
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<tr>
<td>API</td>
<td>American Petroleum Institute</td>
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<tr>
<td>BLOSOM</td>
<td>Blowout Spill Occurrence Model</td>
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<tr>
<td>BOEM</td>
<td>Bureau of Ocean Energy Management</td>
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<td>BSEE</td>
<td>Bureau of Safety and Environmental Enforcement</td>
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<tr>
<td>CMU</td>
<td>Carnegie Mellon University</td>
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<tr>
<td>CO₂</td>
<td>Carbon Dioxide</td>
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<tr>
<td>CSILs</td>
<td>cumulative spatial impact layers</td>
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<td>CT</td>
<td>Computed Tomography</td>
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<tr>
<td>DEHP</td>
<td>Di-Ethyl Hexyl Phthalate</td>
</tr>
<tr>
<td>DIDP</td>
<td>Di-Iso Decyl Phthalate</td>
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<tr>
<td>DOE</td>
<td>Department of Energy</td>
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<td>DOI</td>
<td>Department of Interior</td>
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<tr>
<td>EDS</td>
<td>energy dispersive spectrographic</td>
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<td>EDX</td>
<td>Energy Data Exchange</td>
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<tr>
<td>EIS</td>
<td>electrochemical impedance spectroscopy</td>
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<td>EOS</td>
<td>equations of state</td>
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<tr>
<td>FCGR</td>
<td>fatigue crack growth rate</td>
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<td>FE</td>
<td>fossil energy</td>
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<td>FLD</td>
<td>fast lubrication dynamics</td>
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<tr>
<td>FMECA</td>
<td>failure mode effects, and criticality analysis</td>
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<td>GOM</td>
<td>Gulf of Mexico</td>
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<td>H₂S</td>
<td>Hydrogen Sulfide</td>
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<td>Acronym</td>
<td>Description</td>
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<tr>
<td>HPHT</td>
<td>High Pressure/High Temperature</td>
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<td>HWTF</td>
<td>High-Pressure Water Tunnel Facility</td>
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<td>IAM</td>
<td>Integrated Assessment Model</td>
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<td>ICV</td>
<td>Image Correlation Velocimetry</td>
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<tr>
<td>LAMMPS</td>
<td>Large-scale Atomic/Molecular Massively Parallel Simulator</td>
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<tr>
<td>LISST</td>
<td>Laser In-Situ Scattering and Transmissiometry</td>
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<td>LDA</td>
<td>Laser Doppler Anemometry</td>
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<td>LPS</td>
<td>linear polarization resistance</td>
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<td>NETL</td>
<td>National Energy Technology Laboratory</td>
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<td>NIST</td>
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<tr>
<td>ORD</td>
<td>Office of Research and Development</td>
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<tr>
<td>OREDA</td>
<td>Offshore REliabilty DAtabase</td>
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<tr>
<td>OSU</td>
<td>Oregon State University</td>
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<tr>
<td>OCTG</td>
<td>oil country tubular good</td>
</tr>
<tr>
<td>Penn State</td>
<td>The Pennsylvania State University</td>
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<tr>
<td>Pitt</td>
<td>University of Pittsburgh</td>
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<tr>
<td>psi</td>
<td>pounds per square inch</td>
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<tr>
<td>RES</td>
<td>Research and Engineering Services</td>
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<tr>
<td>ROV</td>
<td>remotely operated vehicle</td>
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<tr>
<td>RPSEA</td>
<td>Regional Partnership for Secure Energy for America</td>
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<td>RUA</td>
<td>Regional University Alliance</td>
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<tr>
<td>SAFT</td>
<td>Statistical Associating Fluid Theory</td>
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<tr>
<td>SCNGO</td>
<td>Strategic Center for Natural Gas and Oil</td>
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<tr>
<td>Acronym</td>
<td>Description</td>
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<td>Technical Report Series</td>
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<td>Deepwater and Ultra-Deepwater Research</td>
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1.0 Executive Summary

In FY13 offshore energy resources research conducted by the National Energy Technology Laboratory (NETL) Office of Research and Development (ORD) focused on developing a scientific base for predicting and quantifying potential risks associated with exploration and production in extreme offshore systems. Specifically, the Deepwater and Ultra-Deepwater Research (UDW) portfolio is focused on reducing and mitigating deleterious environmental events associated with deepwater and ultra-deepwater offshore drilling and production.

Increasingly, offshore domestic oil and natural gas activities are associated with remote and challenging regions, such as the deepwater (greater than 500 feet) and ultra-deepwater (greater than 5,000 feet) in the Gulf of Mexico. Development in these areas poses unique technical and operational challenges, as well as distinct environmental and societal concerns. Domestic resources of natural gas and oil will continue to play a critical role in meeting energy needs in the United States, provided they can be produced with the confidence that environmental concerns (such as air, water, and species protection) are being addressed effectively, as noted in the President’s 2012 executive order on the safe and responsible development of natural gas. The science base necessary to support stakeholder decisions stems from the ability to understand the behavior of engineered-natural systems over a range of extreme conditions.

NETL-ORD has extensive expertise in characterizing engineered natural systems associated with oil and natural gas development. This expertise has been leveraged to address key concerns and technical challenges associated with deepwater and ultra-deepwater hydrocarbon systems through the NETL-ORD Complementary Program in support of the Section 999 of the Energy Policy Act (EPACT) of 2005 and in coordination with the Research Partnership to Secure Energy for America (RPSEA).

In addition to EPACT, the NETL portfolio aligns with other Federal-scale initiatives including the Ocean Energy Safety Advisory Committee (OESC), chartered February 8, 2011, to advise the Secretary of the Interior, on a variety of issues related to offshore energy safety. In particular, the UDW Portfolio aligns with findings and recommendations of the OESC Spill Prevention Subcommittee, a multi-entity committee that seeks to address safety and potential impacts of deep offshore hydrocarbon development in the United States and adjoining regions.

In FY13 six research projects conducted within the UDW portfolio produced quantitative results to support prevention of deleterious events deepwater and ultra-deepwater offshore drilling and production. Ultimately, these projects are filling in knowledge gaps, developing new technologies, and producing results that reduce risks and improve risk-management strategies to prevent impacts associated with extreme offshore hydrocarbon exploration, development, and production. In FY13 UDW projects aligned with the three major topic areas below.

- Improved Science-Base for Materials & Wellbore Integrity: Studying performance and integrity of key offshore materials for which data in extreme environments are limited, including metallic tubulars and borehole cements.
- Improving Safety through Rapid Detection & In Situ Characterization: Tools & techniques to monitor and quickly detect potential hazards for extreme offshore hydrocarbon.

Results and accomplishments from FY13 for six projects under the UDW Portfolio are discussed in detail below.
2.0 Technology Highlights and Key Results

The focus of the EPAct Section 999 program began to shift in 2010 after the April 2010 Macondo Prospect oil well blowout and resulting spill in the Gulf of Mexico. Interest grew at the Department of Energy (DOE) in focusing on the technical basis for assessing and mitigating risks associated with safety and environmental aspects of deepwater oil exploration and production.

NETL-ORD’s Complementary Research Program research portfolio supports the development of unbiased research and information to inform policymakers and the public, performing rapid predictions of possible outcomes associated with unexpected events, and carrying out quantitative assessments for energy policy stakeholders that accurately integrate the risks of safety and environmental impacts. The objective of this body of work is to build the scientific understanding and assessment tools necessary to develop the confidence that key domestic oil and gas resources can be produced safely and in an environmentally sustainable way.

For deepwater and ultra-deepwater resources, the general objective of this research is to develop a sufficient scientific base to be able to apply quantitative risk assessment formalism to the exploration and production of deepwater and ultra-deepwater resources. This includes: (1) experimental studies to improve understanding of key parameters that contribute to loss of control events in deepwater settings, such as delineation of materials performance and properties used for barriers and controls in these systems; (2) building a general understanding of the development of a spatial dataset that describes variation in properties of the reservoirs and engineered systems; and (3) utilizing findings from (1) and (2) in conjunction with integrated assessment models to enable the delineation of worst-case scenarios, as well as assessments of most likely scenarios relative to potential risks associated with flow assurance and loss of control. In this plan, areas of integrated assessment modeling (IAM) emphasis and focus include wellbore materials and design, subsurface properties, and the water column system (physical and environmental).

Research projects in the UDW Portfolio are working to address quantitative prediction of potential deleterious events in extreme offshore drilling and production. Successful implementation of portfolio research objectives will ultimately increase America’s domestic oil and gas supply, reduce our nation’s dependency on foreign imports, and address the environmental and social concerns associated with development. Key FY13 results and accomplishments from each research project in the UDW Portfolio are discussed below.
Characterizing the Behavior of Metal-Based Systems Used for Control Devices in Extreme Environments

Purpose and Goal

The objective of this research is to evaluate failure mechanisms and rates of failure for critical metal components (BOP’s, risers, pipelines, etc.) through observed/reported behavior in the field, augmented with experimental studies on materials behavior under simulated extreme conditions in the laboratory, in order to identify, and thereby reduce or eliminate, potential catastrophic loss-of-control events. In addition, the research examined environmental and mechanical factors that influence metal performance and degrade effective life of metal barrier and control infrastructure components.

Key Accomplishments in FY13

Assessment of Materials-Related Offshore Failures

Offshore reliability data are compiled in the Offshore REliability DAtabase (OREDA), which is maintained by Det Norke Veritas (DNV) in Høvik, Norway. The OREDA Handbook data on equipment classes, related subunits, and maintainable items utilizes a failure rate function to predict the likelihood that a failure of a component will occur within a specified period of time. Specific materials information of failed equipment classes, related subunits, and maintainable items was reviewed and assessed for possible research.

The failure mode effects, and criticality analysis (FMECA) methodology was reviewed. This was done to evaluate if FMECA can be applied to an example offshore system (e.g., topside of an offshore platform or a subsea installation) using the OREDA data. This exercise will serve as an illustrative example of how FMECA and OREDA can be used in assessments of likely scenarios relative to potential risks associated with flow assurance and loss of control. It will also demonstrate the limitations of the OREDA handbook.

OREDA identifies the frequency of failures, which may or may not be associated with materials related issues (i.e., it may be related to design shortcomings or operational issues). Moreover, catastrophic events leading to a major loss of assets or personnel are often the result of an unanticipated chain of events, which would not be predicted based on frequency data. To fill this gap, an expert survey of DNV personnel was conducted to glean knowledge on how materials related failures are identified across disciplines in the offshore industry. The survey was generically designed to probe the interviewees’ knowledge and opinion of the significance of materials in the offshore industry.

Although OREDA, the most comprehensive database for offshore related failures, does not provide detailed materials related failure mechanisms, it is valuable for evaluating and determining reliability of components from a risk-based perspective, and hence by implication, potential material related problems.

Though not traditionally used in this way, a FMECA analysis will be applied to a piece of equipment used in offshore technologies using OREDA data. As discussed, specifics on mechanisms of the materials related failures for the components within the equipment are not available. However, the analysis will demonstrate how FMECA could be used, with additional information, in directing offshore materials research.

Expert surveys are still being conducted, but the feedback already received provided information on specific systems and materials related issues in offshore equipment. The impetus for studying these systems varied depending on the respondent. Concerns on current technologies largely stemmed from reliability issues. For instance, work within sensor technologies leads the way for effective detection and evaluation of degraded systems prior to a major event. For future technologies, the behavior of materials in extreme and harsher conditions was a major concern. For instance, with climate changes, assumed
weather conditions used to predict reliability of offshore systems becomes more and more significant. The behavior or materials in such conditions becomes critical to better design and operations.

_Corrosion and Fatigue of Hammer Peened and Heat Treated Oil Grade Alloy 718_

The corrosion and corrosion-fatigue study on oil-grade alloy 718 concluded in FY13. Most of the year focused on finishing the test program and compiling information for technology transfer activities. Two students were granted degrees for their effort in the research program:

1. Jared Nutter received a M.S. degree for “Corrosion Fatigue Crack Propagation of Oil-Grade Alloy 718 in NaCl Solution.”
2. Ting Chen received a Ph.D. degree for “Pitting Corrosion and Corrosion Fatigue Crack Growth Behavior of Oil-Grade Alloy 718 in NaCl Solution.”

Investigations included significant examination of the microstructures as a consequence of the hammer peening surface modification procedure, plus changes due to subsequent heat treatment to improve bulk mechanical properties. In addition, fatigue crack growth rate and corrosion-fatigue crack growth rate studies of the various microstructures were performed as were comprehensive electrochemical investigations.

Many important findings were discovered but some of the more significant ones included the following:

- Microstructure studies revealed sparse amounts of inclusions spread throughout the grains and along grain boundaries, including nitrides, carbides, and globular delta phases for all aging treatments. After aging treatment, gamma prime and double prime precipitates were formed in the gamma matrix, as well as, discontinuous platelet shaped delta phases along the grain boundaries. The precipitates were much smaller for the two-step aging treatment than the one-step aging treatment.

- The corrosion-fatigue crack growth results showed no obvious effect of 3.5 wt% NaCl solution on the crack growth rates of oil-grade alloy 718 in the three different aged conditions. However, crack growth rate increased in a 21 wt% NaCl for all specimens compared with tests in air and a 3.5 wt% NaCl solution. Crack growth rate differences were more pronounced in the lower ΔK regions where the effects of stress and strain on the crack tip are less dominant. Moreover, aging treatments led to lower corrosion-fatigue crack growth rates of oil-grade alloy 718 in all tested conditions including air. Scanning electron microscopy of the fracture surfaces revealed that the cracks propagated transgranularly under all testing conditions.

With respect to the surface modification process, the following was concluded:

Severe work hardening and high compressive residual stress were generated with surface smoothing and microstructure evolution in terms of formation of nano-sized grains and multi-directional nano-twins in the near surface region after machine hammer peening.

Two grain refinement mechanisms, including twin–twin intersections and twin–dislocation interactions, led to a microstructure evolution sequence:

- Micro-twins or Nano-twins → Nano-grains

Electrochemical tests results showed that machine hammer peening has a beneficial influence on the pitting corrosion resistance of oil-grade alloy 718 in a 3.5 wt% NaCl solution at RT, as indicated by a significant increase of the critical pitting potential (+134 mV), accompanied with lower corrosion current density and higher polarization resistance.
The improvement of pitting corrosion behavior of oil-grade alloy 718 in a 3.5 wt% NaCl solution at RT was attributed to surface smoothing, i.e., the generation of a larger compressive residual stress in the near surface region and the formation of nano-grains and nano-twins produced by the surface modification treatment.

After the two-step aging treatment, a Cr-enriched oxide layer was formed, along with a nano-precipitate layer, which consisted of high precipitate fractions of $\gamma'/\gamma''$ on the top surface of the surface-treated specimens. Surface hardness increased after aging and the compressive residual stress relaxed almost entirely.

The two-step aging treatment resulted in higher susceptibility to pitting corrosion in a 3.5 wt% NaCl solution at RT. This was manifested as a significant decrease in polarization resistance and critical pitting potential. However, the hammer-peened specimen, processed at the higher feed rate (4 m/min) and followed by aging treatment, showed the best pitting corrosion resistance of the aged surface-treated samples.

The improvement in pitting corrosion behavior of oil-grade alloy 718 in a 3.5 wt% NaCl solution at RT was mainly attributed to surface smoothing and the larger compressive residual stress left after the aging treatment.

**Characteristics of Fracture in UD-165 Drill Pipe after Simulated Sour Service**

Catastrophic corrosion of drill pipes are a major concern in ultra-deep well drilling environments. Literature data indicate that approximately 20% of the drill pipe failures are caused by corrosion fatigue (Jiashen and Jingmao, 1993). Given that drill pipes are made of high strength low alloy steels that are the most economic material for construction of pipelines, there has been a significant amount research performed on corrosion fatigue of this material in Hydrogen Sulfide (H$_2$S)-containing environments, also referred to as sour environments. The main effect of H$_2$S on steels is an observed increase in fatigue crack growth rate (FCGR), particularly when H$_2$S concentration is increased in simulated production fluids (Webster et al, 1985; Bristoll and Roeleveld, 1978; Eadie and Szklarz, 1999; Maddox et al, 2005).

However, there is little available data for corrosion fatigue crack propagation for ultrahigh-strength low alloy steels needed for ultra-deep drilling (UDD) operations. To fill this technological gap, fundamental studies of crack growth were needed to develop means of corrosion protection. Ultrahigh-strength low alloy steel, grade UD-165, was selected for the crack propagation study. Fatigue test on pre-cracked samples were performed in a deaerated 5% NaCl solution buffered with Na$_2$CO$_3$/NaHCO$_3$ and in contact with H$_2$S. The partial pressure of H$_2$S ($p_{H2S}$) was 0.83 kPa (0.12 pounds per square inch [psi]) and the pH of the solution was adjusted to 7, 9, or 12 at 20°C. The solution is used by the industry to simulate a generic production fluid.

As an example, Figure 1 shows the fracture surface of UD-165 after the fatigue experiment in the pH7 solution. This sample failed catastrophically.
Figure 1: SEM image of UD-165 that failed catastrophically after corrosion fatigue test in pH=7 solution at 20°C. Red color indicates presence of sulfur products from corrosion reaction between iron and H$_2$S.

As solution pH increased to 9, the sample did not fail catastrophically. Figure 2 shows an SEM micrograph of near the crack tip area after the corrosion fatigue testing in the pH=9.

Figure 2: SEM images of the polished surface after cyclic stress testing in 5% NaCl/NaHCO$_3$/Na$_2$CO$_3$ solution at p$_{H2S}$ = 0.83 kPa at 20°C, pH=9 showing crack morphology near the crack tip.

The crack propagated intergranularly and transgranularly. A few secondary cracks were observed near the crack tip. At site 1, energy dispersive spectrographic (EDS) analyses detected the presence of 68 at% iron and 17 at% S. Higher concentrations of Fe and S were detected in such pockets (Site 1 in Figure 2) compared to narrow cracks (Site 2 in Figure 2). Although Fe- and S-rich corrosion products were detected along the primary crack, it appeared that they did not impede diffusion of hydrogen to the crack tip, so that fatigue crack propagation was controlled by hydrogen diffusion.
In the pH=12 solution, the crack tip became plugged with (Fe,S)-rich corrosion products as shown in Figure 3. This indicated that the crack propagation was not affected by H₂S. It also appeared that fatigue crack propagation was controlled by different mechanisms associated with the presence of H₂S. Therefore, determining H₂S speciation in this solution as a function of solution pH, temperature, and H₂S partial pressure, will be very beneficial in quantifying the role of H₂S in the degradation of UD-165.

**Figure 3:** SEM images of the polished surface after cyclic stress testing in pH=12 in 5% NaCl + NaHCO₃ + Na₂CO₃ solution at p$_{H₂S}$ = 0.83 kPa and 20°C, showing an un-branched crack filled with (Fe,S)-rich corrosion products near the crack tip.

*Catalytic Properties of H₂S in Corrosion Degradation of High-Strength Steels*

Hydrogen sulfide (H₂S) has been identified as one of the most detrimental aqueous corrosive solutes with respect to the corrosion resistance of metallic materials. It dissolves in water and water-based (aqueous) solutions. However, its solubility in water and aqueous solutions, and its formation of dissolved species, depend upon temperature, pressure, and solution acidity (pH).

The typical solution that industry uses to simulate production fluids in the laboratory is a deaerated 5% NaCl solution, buffered with NaHCO₃/Na₂CO₃ and in contact with H₂S. The solution corrosiveness can be managed by controlling the partial pressure of H₂S as well as solution pH (Figure 4).

**Figure 4:** Schematic representation of sulfide corrosion environment severity regions: “o” shows the relative position on the diagram for a 5% NaCl/NaHCO₃/Na₂CO₃ solution in contact with p$_{H₂S}$ = 0.83 kPa (0.0083 bar; 0.12 psi) at 20°C.
According to Figure 4, for partial pressure of H$_2$S ($p_{H2S}$) of 0.83 kPa (0.12 psi) in contact with buffered 5% NaCl, pH=12 at 20°C, the solution can be considered as “mild”. Therefore, an alloy operating in this environment is expected to be resistant to sulfide corrosion. It is worth noting that Figure 4 was developed for oil country tubular goods (OCTG) with yield strength of 758 MPa (110 ksi). Materials of higher strength or with inhomogeneous regions, such as welds, are expected to follow a similar trend but the precise positions of the boundaries between regions will be different, and they have yet to be established (The Institute of Materials, 1995).

To understand the possible mechanism involved in sulfide corrosion, the reacting species must be considered. Literature data indicate that in acidic environments H$_2$S reacts directly with steel to produce iron sulfide through formation of an (Fe-HS) intermediate (Brown et al, 2003). If this mechanism prevails in neutral and alkaline solutions then it will be necessary to determine how changing solution pH and temperature affects corrosion through changing H$_2$S solubility and speciation. Thermodynamic modeling is useful in showing the way.

The HCh chemical simulation software has been used to monitor dissolved H$_2$S in a 5% NaCl solution at 25°C as a function of pH and H$_2$S partial pressure. Changes in solution pH were modeled by varying the concentrations of the solution using HCl and NaOH (Shvarov, 1999).

The first series of thermodynamic simulations has shown that the concentration of H$_2$S is expected to depend greatly on partial pressure of H$_2$S but not on pH. However, the concentration of HS$^-$, which is the primary dissociation product of H$_2$S, is expected to increase with increasing H$_2$S partial pressure and solution pH.

Taking into account temperature on H$_2$S concentration in the thermodynamic model for 0.01 bar (0.145 psi) H$_2$S from 25 to 200°C showed that the concentration reaches a minimum around 0.3 to 0.4 mmol/kg H$_2$S between 100 and 200°C. A similar analysis for HS$^-$ concentration found almost no change with respect to temperature, showing that HS$^-$ is expected to only be a function of solution pH and H$_2$S partial pressure, whereas dissolved H$_2$S is expected to vary with H$_2$S partial pressure and temperature, though the effect is less dramatic as the temperature increases above 100°C.

These thermodynamic simulations were used to assess the effects of changing environments on sulfide-assisted corrosion mechanisms. If the corrosion processes are largely dependent on the concentration of dissolved H$_2$S, then increasing temperature reduces the concentration and results in a significant decrease in the corrosion rate. Little or no change would be expected due to changing solution pH. However, if the concentration of HS$^-$ dictates the overall corrosion rate, then the opposite behavior would be observed. Increasing pH would greatly increase the corrosion rate, while changing temperature should have little effect. In both cases increasing the partial pressure of H$_2$S should increase the corrosion rate.

Research in FY14 will attempt to confirm the modeling results. The experimental exposure system has been designed and will allow for electrochemical measurements in a well-stirred autoclave vessel. High strength steel, Grade S-135, and ultrahigh strength steel, Grade UD-165, were selected for these experimental verification studies. Selected experiments have been performed using a computer-controlled potentiostat to perform linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS) measurements to investigate the corrosion behavior in real-time.

Role of Yield Strength and Microstructure on Fatigue Crack Growth Rate in Sour Environments

Offshore pipelines are subject to large fatigue stress from drilling, Vortex Induced Vibrations, and thermal transients particularly during shut down. These cause significant fatigue demand during the operation of the pipeline. Thermal transients typically are very low frequency events with high amplitudes, which can lead to lateral buckling. The loading rates associated with the transients are very slow; hence, the
toughness properties of the material in environments at low loading rates are important. The pipelines are also subject to high stress and thus need to have high yield strength and good ductility. Some work has been performed to understand the response of carbon steels in dynamic applications like risers and flow lines in sour service. The effect of H₂S on steels is typically observed as an increase in the fatigue crack growth rate (FCGR), with FCGR at high ∆K values being as high as 50X those for corresponding tests conducted in air. There are, however, key factors that affect the mechanism of fatigue crack growth and the environmental factors that control it, and these must be examined in more detail.

An important goal in developing better understanding of material behavior in sour service environments rests upon understanding and quantifying the effects of yield strength on the FCGR, as well as the response between yield strength and fracture toughness. It is also important to be able to measure the hydrogen flux and diffusion coefficient of hydrogen in the various steel microstructures. The role of microstructure in trapping hydrogen is critically important and needs to be addressed in order to build a comprehensive failure mechanism model. As such, the effects of sour service over a range of pH and H₂S concentrations have been investigated through:

- FCGR measurements
- Fracture toughness measurements
- Diffusible H and trapped H measurements

As part of the overall effort to understand the role of yield strength and microstructure on the sour service properties of the steels, significant scientific and practical value can also be gained by characterizing the behavior of the welds using these same testing procedures.

The role of yield strength and microstructure on FCGR in line pipe steels in a mildly sour environment was, therefore, studied since significant historical information exists for these materials. Controlled testing of newer line pipe steels was evaluated against the historical data to evaluate the FCGR performance of various line pipe grade steels in sour environments.

The conclusions from the program are:

- FCGR of all grades of line pipe steels increases with decreasing frequency and reaches a plateau for the X52, X65, and X80 grades. This suggests that FCGR propagation in all of the line pipe steels is associated with a hydrogen driven mechanism. No plateau was observed for X100 and 4137M grades, and was likely due to K_{1SSC} behavior.

- The plateau FCGR appears to be independent of the yield strength at the ∆K=1000 Nmm⁻³/² in the mildly sour environment. At a higher value of ∆K, 1211Nmm⁻³/², FCGR decreases with increasing yield strength.

- The effect of yield strength at the higher ∆K appears to be associated with the shorter diffusion distances in the higher strength steels, coupled with the higher hydrostatic stresses which enhance the concentration of hydrogen.

- The X-52 vintage pipe had the coarsest grain size and exhibited almost complete transgranular crack propagation during the fatigue test. The newer X-52 and the X-65 pipe steels had a finer ferritic/bainitic microstructure and showed some signs of transgranular crack propagation. The X-80 steel was predominantly bainitic with acicular grains.

- Local misorientation maps showed a qualitative increase in plasticity around the crack with a corresponding increase in yield strength from X-52 vintage to X-80. Transmission electron microscopy imaging of the region close to the fracture surface shows dislocation sub-cell
formation as well as significant dislocation activity. Increased mobility of dislocations due to the presence of hydrogen would be consistent with this observation.
Determining the Physical and Chemical Behavior of Cement Barriers Used in Ultra-Deep Water Systems

Purpose and Goal

The primary function of well cement is to provide casing support and zonal isolation for the life of the well. Foamed cement is a gas-liquid dispersion that is created when a gas, typically nitrogen, is stabilized as microscopic bubbles within a cement slurry (Harms, 1985; Nelson, 2006). Foamed cements are ultralow-density cement systems used in formations unable to support the annular hydrostatic pressure of conventional cement slurries (Nelson, 2006; Harlan et al., 2001). The use of foamed cement for its lightweight density is well documented in literature (Benge & Poole, 2005; Harlan et al., 2001; White et al., 2000; Kopp et al., 2000; Frisch et al., 1999; Benge et al., 1996; Thayer et al., 1993; Harms & Febus, 1985). More recently, foamed cement use has expanded into regions with high-stress environments, for example, isolating problem formations typical in the Gulf of Mexico (Rae & Lullo, 2004; White et al., 2000; Judge & Benge, 1998; Benge et al., 1996).

Current testing methods are limited to atmospheric conditions. However, it is well known that elevated pressures in the wellbore environment have a profound effect on foamed cement properties. Foamed cement stability depends on time evolution of the gas bubble-size distribution (BSD) and varies as it is pumped and placed in the well.

NETL researchers have produced the first high resolution x-ray Computed Tomography (CT) three dimensional images of atmospheric generated foamed cement across a range of foam qualities. CT imaging enables the assessment and quantification of the foamed cement structure, quality, and BSD in order to provide a better understanding of foamed cement. In addition, NETL researchers have produced the first x-ray Computed Tomography (CT) three dimensional images of field generated foamed cement across a range of foam qualities and pressures. Foamed cement was collected utilizing the same full scale industrial equipment and methodology used to generate cement in a well.

In addition to its light-weight application, foamed cement has a unique resistance to temperature and pressure-induced stresses and long-term sealing through resistance to cement-sheath stress cracking (White et al., 2000). In comparison to conventional cement, foamed cement is ductile and will deform when the casing is pressurized (Kopp et al., 2000). As such, foamed cements are often used to prevent stress cracking in the cement sheath due to temperature and pressure cycles (Benge et al., 1996). Industry standards require the measurement of mechanical parameters to ensure the integrity of the primary cement job. Therefore, this research is measuring the physical properties of foamed cement typically used in deep offshore wells in the Gulf of Mexico.

The general objective of this work is to develop a sufficient scientific base to be able to apply quantitative risk assessment formalism to evaluate the exploration and production of deepwater and ultra-deepwater resources. Adequate definition of materials performance and properties is critical to this effort. The outcome of this project is to provide a better understanding of the effects that foam cement production, transport downhole, and delivery to the wellbore annulus have on the overall sealing process.

Key Accomplishments in FY13

Atmospheric-Generated Foamed Cement

Two industry standard foam cement systems have been generated using the current test method (API RP 10B-4 – Recommended Practice on Preparation and Testing of Foamed Cement Slurries at Atmospheric Pressure) across a range of foam qualities (10% - 40% gas volume). All atmospheric-generated foamed cements have been scanned using the industrial CT scanner. Image and data analysis is complete. A Technical Report Series (TRS) entitled “Computed Tomography and Statistical Analysis of Bubble Size
Distributions in Atmospheric-Generated Foamed Cement” is available: http://www.netl.doe.gov/onsite_research/gasandoil/NELT-TRS-2-2013_FoamedCement_20130809.pdf. In addition, a TRS detailing the CT imaging and statistical analysis of the second set of atmospheric-foamed cements is in progress.

Physical/mechanical properties of both foamed cement systems have been determined using the identical samples used for the CT imaging and analysis. Permeability, porosity, compressive strength, Young’s modulus (E), and Poisson’s ratio (ν) were measured across a range of foam qualities. Test results include a modified ASTM C109/C109M relationship correlating different geometries of cement. Results indicate a relationship between physical properties and foam quality. A draft TRS correlating the foam quality with the mechanical properties (permeability, porosity, and strength) is complete and undergoing review by the team. Ultrasonic velocity measurements have started and are ongoing.

Field-Generated Foamed Cement

Collecting representative samples of foamed cement from the same full scale industrial equipment used to generate foamed cement in a well presented a number of challenges. In order to collect a representative sample of foam cement for CT Scanning from standard oilfield industry foam cement equipment a method had to be developed to remove a small amount of foam cement from the high pressure treating lines while keeping the foam cement under constant pressure. The foamed cement sample needed to be kept under the same pressure while curing and a method had to be determined to transport the set foam cement from the job site to the CT scanning facility in Morgantown, West Virginia. All of this had to be accomplished without putting anyone at risk of injury or damaging any equipment.

Two sets of field-generated foamed cements have been collected by our industry collaborators. The first set (from Schlumberger) has been scanned using both the medical and industrial CT scanner. CT imaging and data analysis is nearly complete. A second set of field-generated cements (from Baker Hughes) was recently collected and CT-scans, image and data analysis has been started and is ongoing. A draft TRS detailing the CT imaging and statistical analysis of the Schlumberger field-generated samples is underway. Posters have been prepared for laboratory tours (including the recent visit from Secretary of Energy Moniz in Morgantown. Several invited presentations were given at technical conferences.

Modeling

The spatial distribution of gas bubbles in foamed cement system is being modeled. The models will allow for the realistic incorporation of: (1) variable gas volume fraction with depth/pressure, (2) time dependent non-Newtonian rheology of the suspending phase as the hydration reaction proceeds in the suspending cement phase, and (3) interplay during globally imposed shear flow between the local flow profile, emergent coarse-grained rheology of the cement/gas bubble composite, and spatial correlations between bubbles and bubble motion (bubble entrainment, shear-induced bubble rearrangements, etc.). In particular:

- Modeling to determine the spatial distribution of gas bubbles in cement foams and its impact on the mechanical properties and failure probability of the as-set foam.
- Transferring knowledge of the bubble distribution at laboratory conditions to field conditions at depth, where the gas fraction of the foam decreases due to high pressure from the overburden.
- Determining correlation between atmospheric and pressure generated foamed cement systems. This is significant to better understand the correlation between current American Petroleum Institute (API) test method and foams generated under pressure.
- Determining correlation between stability, BSD, permeability, and porosity of foamed cement systems.
Brownian and fast lubrication dynamics (FLD) simulations of foamed cement were performed. The FLD simulations were performed using Matrix Laboratory (MATLAB). The FLD MATLAB code is being developed based on Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS), a classical molecular dynamics code, which is being developed, run, and validated on a regular basis. LAMMPS is distributed as an open source code by Sandia National Laboratories.

- Poiseuille geometry in the FLD code was implemented; output is being investigated.
- A flow profile for regular droplet arrays at a typical air volume fraction at atmospheric conditions was measured.
- Data from the atmospheric foamed cements that were CT scanned at NETL were collected.
- The FLD MATLAB code is being validated in other simple flow geometries.
Quantifying Complex Fluid-Phase Properties at High Pressure/High Temperature

Purpose and Goal

This project addresses significant challenges associated with the efficient, safe, and environmentally responsible exploitation of Ultra-Deepwater petroleum resources. These challenges are twofold: (1) insufficient quantification of the thermodynamic and transport properties of the petroleum constituents under the extreme conditions associated with the reservoirs and (2) lack of accurate equations of state (EOS) models that correlate the thermodynamic and transport properties with High Pressure/High Temperature (HPHT). We have combined well-defined experiments, theory, and robust computational models to address these critical issues. The focus of the experimental effort is to develop a comprehensive database of thermodynamic and transport properties of Ultra-Deepwater reservoir fluids (both pure and multi-components systems). These unique data are used to develop improved EOS models accurate up to temperatures of 500°F and pressures up to 40,000 psi. The availability of such models should promote and improve the safe and efficient development and utilization of Ultra-Deepwater petroleum resources.

Key Accomplishments in FY13

Successfully Identified Fluid That Can Serve as a “Deepwater Viscosity Standard” for Experimental Studies

The project team successfully identified an appropriate fluid for use by experimentalists studying the viscosity behavior under extreme conditions of hydrocarbons. Researchers worldwide have sought a standard of this type to support viscosity studies of petroleum fluids found in ultra-deep formations (such as those below the deepwaters of the Gulf of Mexico). The fluid identified by this project team allows this team and other researchers to have a fluid with which they can reliably calibrate their viscometer at the relevant conditions. We have determined, using both the rolling ball viscometer and the NETL HTHP Couette viscometer, that the perfluropolyether oils known as DuPont Krytox 102 is an excellent candidate. Krytox 102 has a viscosity of about 26 cP as shown in Figure 5 below. This viscosity is sufficiently close to 20 cP and has been suggested, in collaboration with the National Institute of Standards and Technology (NIST), as Deep-water Viscosity Standard. These results have been well received when presented to the rheology community at the 12th International Association of Transport Properties conference and at the subsequent 18th Symposium on Thermophysical Properties, in Boulder, Colorado. Seven foremost researcher groups in Australia, Spain, United Kingdom, and the United States have already asked our group to provide Krytox 102 samples from the same lot for their studies of this fluid with their viscometers.
The formation of solid deposits in crude oil can clog processing lines during production, potentially leading to severe equipment damage. This is a major issue experienced during the oil recovery process for HPHT ultra-deep petroleum reservoirs such as those typically encountered beneath the deep waters of the Gulf of Mexico. Hence, HPHT liquid-solid phase transition data for hydrocarbons such as heavy n-alkanes, cyclics, and aromatics are essential for determining operating conditions that minimize or avoid the formation of solids. There is currently a substantial amount of literature data on the high-pressure solidification behavior of paraffinic hydrocarbons, up to carbon number 60, but there are fewer available solidification data for cyclic hydrocarbons. The studies that have been done on solidification behavior of aromatic hydrocarbons are limited to pressures below 100 MPa. Perhaps more importantly, there are no high-pressure solidification data for saturated cyclic hydrocarbons.

The NETL research team has recently addressed the gap in available aromatic and cyclic hydrocarbon solidification data by measuring solid-liquid transition data for cyclooctane, methylcyclohexane, ethylcyclohexane, cis-1,2-dimethylcyclohexane, cis-1,4-dimethylcyclohexane, trans-1,4-dimethylcyclohexane, p-xylene, m-xylene, o-xylene, and 2-methylnaphthalene. A visualization technique coupled with a high-pressure, variable-volume view cell is used for these solidification measurements at pressures to 300 MPa and over a wide range of temperatures starting at 293K. The solidification transitions were successfully duplicated and often triplicated over an extended period of time to insure reproducibility of the results. The NETL research team has also provided the parameters needed in the Simon equation, a popular equation used to correlate solidification temperatures and pressures, which allows for estimation of the solidification conditions within the range of temperatures and pressures of the study. These findings have been reported in a manuscript titled, “Investigation on the Solidification of Several Pure Cyclic and Aromatic Hydrocarbons at Pressures to 300 MPa,” released in the September 2013 issue of the journal Fuel, volume 111, p. 75-80, 2013.
Experimental Results Fill HPHT Density Data Gap in the Open Literature

Our group has provided experimental density data for hydrocarbons in Table 1 below, ranging at temperatures to 500°F (260°C) and pressures to 40,000 psi.

Table 1: Experimental Density Data for Hydrocarbons

<table>
<thead>
<tr>
<th>n-alkanes</th>
<th>Iso-alkanes</th>
<th>Cyclic</th>
<th>Aromatics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Propane</td>
<td>2-Methylpentane</td>
<td>Methyl cyclohexane</td>
<td>Toluene</td>
</tr>
<tr>
<td>n-Pentane</td>
<td>2,2,4-Trimethyl pentane</td>
<td>Ethyl cyclopentane</td>
<td>p-Xylene</td>
</tr>
<tr>
<td>n-Octane</td>
<td></td>
<td>cyclooctane</td>
<td>m-Xylenes</td>
</tr>
<tr>
<td>n-Decane</td>
<td>Ethylcyclohexane</td>
<td></td>
<td>o-Xylenes*</td>
</tr>
<tr>
<td>n-Hexadecane</td>
<td>cis-1,2-Dimethyl cyclohexane, *</td>
<td>2-Methyl naphthalene*</td>
<td></td>
</tr>
<tr>
<td>n-Octadecane</td>
<td>trans-1,4-Dimethyl cyclohexane*</td>
<td></td>
<td></td>
</tr>
<tr>
<td>n-Eicosane</td>
<td>cis-1,4-Dimethyl cyclohexane*</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cyclohexane</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Density data was obtained at 50°C and 150°C.

Experimental Results Fill Density Data Gap for a Reference Fluid

Di-Ethyl Hexyl Phthalate (DEHP) has been suggested as a reference fluid for viscosity measurements in the HPHT regions consistent with ultra-deep reservoir conditions. DEHP is preferred over the current fluid reference, Di-Isodecyl Phthalate (DIDP), since DEHP is commercially available in high purity while DIDP is only available as a mixture of phthalates esters of the isomers of isodecyl alcohols.

An exhaustive literature review revealed a lack of density data in the open literature for DEHP at both HPHT conditions. The NETL-Regional University Alliance (RUA) researchers have now filled this literature gap by reporting HPHT DEHP density data in the August 2013 issue of the Journal of Chemical Thermodynamics, 63, p. 102-107, 2013. The density data, obtained using a variable-volume, high-pressure view cell technique, were reported for five isotherms (373, 424, 476, 492, and 524K) and pressures from ambient to 270 MPa (40,000 psi). The NETL-RUA team modeled the experimental data with both cubic-based and Statistical Associating Fluid Theory (SAFT)-based EOS. As described in detail in the journal manuscript, the NETL-RUA results demonstrated that a cubic-based EOS fails to reliably predict the density of DEHP at high temperatures and pressures, which is not surprising given the high molecular weight of DEHP. In contrast, the NETL-RUA researchers demonstrated that reliable densities were predicted with the SAFT-based equations of state combined with first order group contribution methods, which increases the range and applicability of the fundamental SAFT-based equation approach.
GOM Integrate Assessment Modeling (IAM) Project: Assessing Risks and the Potential for Environmental Impacts for Deepwater, Ultra-Deepwater, and Frontier Resources

Purpose and Goal

Production from offshore drilling in U.S. federal waters, currently limited to the Gulf of Mexico (GOM) and California, now roughly equals production from the Alaskan North Slope and is a key component of the U.S. energy independence. Increasingly, exploration in these maturing offshore regions is moving into deep and ultra-deep water environments (EIA 2011). At the same time more attention is being placed on frontier areas that garner strong public and environmental concerns, such as offshore of the Alaska North Slope (Beaufort and Chukchi Seas), offshore eastern United States, and the eastern GOM. The 2010 Deepwater Horizon disaster clearly demonstrated a need for an integrated and improved evaluation of natural and engineered attributes (geologic, engineering, water column and atmospheric) associated with these systems that allow researchers to rapidly evaluate and assess risks and environmental implications of development in these regions. In addition, this experience highlighted the need for independent estimates of potential loss of control-related scenarios (such as maximum potential flow rates for a failed well).

This project was initiated in FY12 by NETL-ORD and focuses on conducting quantitative risk assessments and research of spatial trends in the subsurface and water column of the deepwater and ultra-deep water GOM. The goal of this study is to estimate and evaluate potential risks related to exploration, development, and production in this region. Based on these evaluations this study seeks to identify key knowledge and research gaps related to hydrocarbon activities in the extreme offshore GOM, as well as offer numerical simulation tools and key data inputs needed to support rapid predictions in the case of an unexpected loss of control event. Many of the datasets necessary for these assessments are available from existing resources (including other governmental agencies, industry, etc.). Thus, the intent of this effort is not to reproduce what others have already developed but rather integrate these various data sources so assessments of safety and environmental issues can be conducted. In addition to publically available resources, proprietary information from industry partners and/or commercial vendors will be pursued. Some of these may require special handling; as a federal entity, NETL is in a unique position to serve in this role where proprietary data may be required for derivative products but the primary datasets must remain confidential.

This effort builds off of NETL’s existing expertise in capturing and analyzing geospatial variability in key attributes, including past work on tight gas resource assessments, Marcellus shale characterization, and the Natcarb atlas. This comprehensive project leverages that experience, allowing NETL researchers to conduct assessments of potential social, environmental and production risk factors, provide recommendations on future technology needs, and assist in responses to unexpected events (such as future Macondo-like disasters). NETL will also use these data to assess geospatial trends in potential release rates as a function reservoir and well variability. This assessment will rely on the development and use of an IAM that will form the basis of a quantitative risk assessment platform for offshore resources, ultimately incorporating information developed in other tasks within the NETL deepwater effort (such as failure rates for key components, long-term behavior of wellbore cements, etc.). In the FY12 effort, NETL will build on experience from estimating flow rates for the Macondo well by utilizing a surrogate reservoir model coupled to a reduced order pipe-flow model (via the IAM) to explore variations in flow rate a function of likely variability in reservoir attributes (exploiting the quantitative evaluations noted above). The product will be the aforementioned IAM that can be used to predict flow rate ranges through failed wells and a geospatial evaluation of potential maximum flow rates, for use in environmental risk assessments. This initial IAM will be expanded in out years to incorporate detail for other components of the engineered-natural system.
The ultimate objective of this project is to complete development of a suite of science-based tools (Energy Data Exchange [EDX] and IAM) that will allow for the unbiased assessment of potential risks and environmental impacts associated with offshore drilling in the deepwater Gulf of Mexico. These tools will be used to produce unbiased assessments of potential risks associated with hydrocarbon development in deep offshore settings. EDX and the GOM IAM tools will incorporate the best-available information to characterize the complexities of deep offshore, natural-engineered systems targeted for hydrocarbon development by industry. This includes properties of geologic formations and pore-filling media/fluids, wellbore characteristics, and water column attributes (biologic and physical). Impacts of system variability and uncertainty on quantified risks will be taken into account through formalized uncertainty quantification.

Key Accomplishments in FY13

Recent natural and anthropogenic uncontrolled hydrocarbon release events, such as from Hurricanes Katrina and Rita and the BP Deepwater Horizon disaster, identified significant gaps in the ability of current tools and technology to predict risks associated with offshore hydrocarbon production as well as the capabilities to respond to deleterious events of varying scope, magnitude, and duration (e.g., Graham et al., 2011). Knowledge of these gaps along with industry’s continued advance into new, unpredictable environments, particularly deep and ultra-deepwater, and deep subsurface environments, exemplifies the need for a comprehensive system-wide tool that integrates science based data to simulate the complexities of natural and engineered-natural systems to estimate potential risks and impacts. No publically available solution currently available incorporates subsurface reservoir, wellbore, and water column data and simulation tools to allow for spatial prediction in support of spill prevention, and support rapid response to unexpected hydrocarbon release events. Instead, systems available tend to focus on single components (e.g., subsurface reservoir characteristics, or blowout characteristics, or surface spill characteristics, or oil weathering characteristics, or response options, etc.). Therefore, there is no integrated system to capture the inherent complexities found in and between natural and engineered-natural systems that heavily influence the behavior of an uncontrolled release event (e.g., Reed et al., 1999). Additionally, many of the integrated models available that are capable of combining multiple components tend to be proprietary and can require a significant investment to obtain access to the software and/or underlying support data. Furthermore, a large range of datasets are utilized by these solutions as model inputs, including proprietary databases, field data collected by industry, and authoritative, open-source datasets provided mainly by federal and state governmental agencies and academia. However, these datasets are often dispersed amongst sources, varying in quantity and quality, and unavailable in a single location making it difficult to access data quickly when needed, such as during the response and recovery to any loss of control event.

Development of Data Inputs in Support of GOM-IAM for Receptors

One component of evaluating and identifying key risks, knowledge, and R&D gaps related to offshore GOM hydrocarbon development involves understanding what receptors will be impacted. In the offshore environment key receptors are associated with the water column and near-shore systems, in particular offshore infrastructure, transportation systems, and biologic communities. There are numerous data elements associated with these receptor systems, so the focus of this effort was to identify, value, and spatially concatenate and analyze high value components of these systems. In FY13, the GOM IAM team utilized select publically available, authoritative spatial datasets and developed a new approach to display and concatenate key spatial data attributes for the water column system portion of the GOM in support of understanding potential impacts of a hydrocarbon release event. These data were binned within four major socio-economic ocean use sectors traditionally affected by oil spills: (1) oil and gas industry infrastructure, (2) commercial transportation, (3) commercial fisheries, and (4) tourism (Aldy, 2011; Graham et al., 2011).
These four concatenated datasets were then used to estimate their spatial distribution and annual economic value to provide qualitative and quantitative cumulative spatial impact layers (CSILs) (Figure 6). Ultimately, these CSILs interpretations will be used in combination with modeled output scenarios from the GOM IAM tools to identify broad spatial trends of risks and potential impacts associated with different modeled loss of control scenarios. However, the spatial approach developed to produce the CSILs, and the CSILs for the GOM may also be applied to other systems and relevant for other analytical purposes. The results of this work in FY13 is forthcoming in an NETL publication in FY14, (Bauer, J., Nelson, J., Romeo, L., Eynard, J., Sim, L., Halama, J., Rose, K., and Graham, J., 2014, A Spatial Approach to Analyze Broad Risks and Potential Impacts Associated with Uncontrolled Hydrocarbon Release Events in the Deep and Ultra-Deepwater Gulf of Mexico, NETL-TRS) and is also anticipated to be submitted to a peer-reviewed journal for publication as well.

Figure 6: RB Map From Bauer et al., in review, cumulative spatial impact layer related to the estimated annual economic value ($USD) per 4.77 x 4.77 km grid cell in the Gulf of Mexico based on various activities related to the oil and gas, commercial transportation, commercial fishing, and tourism ocean use sectors.

Development of Data Inputs in Support of GOM Subsurface

In FY13, the Gulf of Mexico Ultra-Deepwater Subsurface team has imported data for more than 4,000 Gulf of Mexico deep- and ultra-deep water boreholes into NETL’s Petra database, including well logs, directional surveys, and borehole characteristics such as drilling depth and water depth (Figure 7). Thanks to collaboration with the Department of the Interior-Bureau of Ocean Energy Management (DOI-BOEM) and Bureau of Safety and Environmental Enforcement, NETL has access to raster logs as well as sand, reservoir, and production information compiled by Bureau of Ocean Energy Management (BOEM). These databases, along with NETL’s own ongoing interpretations, add more than 2,600 located reservoir tops in 1,600+ wells to the NETL database in the deep- and ultradeep- water. Average sand properties, correlated to the reservoir locations, are available for more than 2,000 of these reservoirs. Depth calibration of the raster logs is ongoing, but more than 1,500 wells have been calibrated as of November 2013. Using federal lease blocks as a proxy for spatial distribution, 47% of the lease blocks for which NETL has data have depth calibrated well logs, 43% have located reservoirs, and 42% have associated average sand data. A model was also completed for the Amberjack field in Mississippi Canyon to use as test input for reservoir and borehole/flow subteams to calibrate and test these reduced order models that are currently under development in support of this project.
Figure 7: Map showing the distribution and types of subsurface datasets compiled and utilized by GOM IAM researchers in FY13.

Along with the Gulf of Mexico subsurface data framework, a draft literature review discussing the Gulf of Mexico subsurface will be completed by the end of first quarter, FY14. The literature review serves as both a project to familiarize NETL's UDW subsurface team with the Gulf of Mexico's geological past and as a tool for researchers seeking organized, vetted information in future research endeavors. It will provide information on paleogeography, depositional history, structure, tectonics, and the petroleum system.
Deepwater and Ultra-Deepwater Research

Figure 8: Map showing the distribution of wells across the GOM. Black box is MC109 area. Inset shows the subsurface interpretation by the GOM IAM team for two sand-rich reservoirs that produce hydrocarbon from M109 area.

**Water Column Systems Model Development**

To assist with risk assessment to prevent future hydrocarbon spills and provide a comprehensive tool for response planning, we have developed an integrated water column modeling system for simulating offshore oil spills resulting from deepwater (>500ft) and ultra-deepwater (>5,000ft) blowouts. The water column modeling system is comprised of multiple models for the plume phase, the advection and diffusion dominated transport phase, an intermediary model between the two, and other models for handling the physical properties of oil/gas and the weathering of crude oil. In FY13 the beta version of this model, title the Blowout Spill Occurrence Model (BLOSOM) was completed. The development of this modeling system which spans a spill at the sea surface and follows the fate of the hydrocarbon through the water column to the shore (Figure 9) has allowed for the adaptation of the entire system towards ultra-deepwater blowouts instead of simply assigning a deepwater plume model to a traditional oil spill process model.

In FY13 BLOSOM was invited to participate in a model inter-comparison study on plume dynamics and droplet-sizes, particularly in the presence of dispersants, hosted by the API. The API-led effort includes a wide diversity of plume modelers from Texas to Norway to determine the relative strengths of each model and identify areas of larger uncertainties. In addition, the study seeks to use the modeling effort to better understand the formation of subsurface plumes and the effect of dispersants on these plumes, phenomenon that are presently poorly constrained and for which there is not substantial real world data. The study will be ongoing until mid-December 2013 with a presentation of results planned in early 2014.
Figure 9: Screen shot of the BLOSOM tool showing a preliminary run of BLOSOM, for a hydrocarbon release event in the GOM, Mississippi Canyon Block 109.

Development and Integration of Tools and Datasets Related to GOM Offshore Systems in Support of GOM-IAM

This project is utilizing the EDX and GOM IAM tools developed in FY13 to conduct assessments of potential social, environmental, and drilling risk factors, and identify technology needs to help reduce risks associated with offshore drilling in deepwater GOM. Finally, the IAM and EDX-based tools developed by this project will provide an independent response system that can be utilized by researchers, industry, and regulators to develop and evaluate response plans in the event of a loss of control event (Figure 10).

Figure 10: Schematic representation of technical and scientific components of the GOM-IAM project illustrating the relationship between the three components of the GOM-IAM under development and the data delivery system (Geocube).
The GOM IAM project team worked with the EDX Operations and Development Team to develop a new web-based mapping tool. The intent of this system was to provide timely access to up-to-date information spanning the subsurface to the shore for the GOM region in support of the risk assessments and impacts modeling underway by this project. In addition, this system is anticipated to serve as a knowledge transfer product from this effort and make access to this type of spatial data and information more accessible to other research that coincides with the GOM region but is focused on other aspects of energy R&D. The GOM IAM team spent significant time assembling key, publically available information and data to support our efforts. The team leveraged that data to support development of the first NETL Geocube.

NETL’s Geocube is a flexible, customizable web mapping application that concatenates key spatial datasets and information relevant to energy research needs for a given region. NETL’s Geocube aims to connect users to data and information spanning the subsurface to the shore via NETL’s EDX, (edx.netl.doe.gov), integrating EDX’s repository of data and information with current data feeds for spatial information. The first NETL Geocube was released in FY13 and focuses on the offshore Gulf of Mexico (GOM) region and supports NETL’s GOM IAM risk assessment for the Gulf of Mexico deep and ultra-deepwater offshore. The GOM Geocube integrates numerous spatial datasets allowing the user to view and analyze spatial information related to the offshore GOM region including subsurface geologic data, oil and gas infrastructure, water column physical and biological information, and numerous human marine and coastal activities, including tourism and recreational and commercial fishing. Users have the ability to add additional data layers either via a search of NETL’s EDX system or by uploading their own datasets. Ultimately, this tool seeks to offer NETL researchers and other users an advanced tool to search, find, display, and download key datasets and custom web maps in support of better understanding a variety of questions related to engineered-natural systems such as the U.S. region of the Gulf of Mexico. In FY14 the GOM IAM team anticipates integrating data from the Geocube with the GOM IAM modeling tools in support of risk and impact assessments under development as part of this project.

https://edx.netl.doe.gov/gom-geocube/.
Improving Deepwater Drilling Safety through Enhanced Understanding of Multiphase Flow Dynamics of Hydrocarbon Mixtures

Purpose and Goal
This project is an experimentally driven project for obtaining fundamental information useful for reducing risks and improving response and planning in the event of a spill associated with offshore drilling in deepwater and ultra-deepwater environments. The goal is to develop a video analysis tool for both quantification of natural or man-made oil/gas plume rates and determination of the presence of gas hydrates from video from an undersea remotely operated vehicle (ROV). Major accomplishments and their relevance are described below.

Key Accomplishments in FY13

Video Analysis Performed on Simulated Submerged Oil Leaks Performed in Large Tow Tank

Members of the NETL team analyzed data from NETL sponsored experiments performed at the end of last fiscal year in the large (2.4m wide, 1.5m deep, 70m long) University of California Berkeley (UCB) Towing Tank in which dye-colored, turbulent water jets were studied. The water jets were measured with Laser Doppler Anemometry (LDA) and high speed videography. A video analysis technique called Image Correlation Velocimetry (ICV) was used to measure the flow rate of the dye colored water jets. The ICV measurements showed good agreement with the LDA measurements. A manuscript describing the experiments and results was submitted to the Journal of the Society of Petroleum Engineers in May, 2013.

Video Analysis of Submerged Natural Gas Leaks at UCB

Members of the NETL team returned to the UCB Towing Tank to perform experiments in which a submerged natural gas leak was simulated using compressed air jets in the tow tank. The submerged air jets were recorded with high speed videography. The purpose of these experiments was to visualize the behavior of a submerged natural gas leak to see if the gas leak rate can be measured using the velocity of visible features.

A compressed gas leak will “choke” if the pressure inside the gas line is more than two times the pressure of the surrounding water. Choking happens when the velocity of the gas reaches the speed of sound in the gas. If a gas leak is choked, compressed gas theory can be used to calculate the leak rate. Only the size of the hole from which the gas is leaking and the pressure inside the gas line need to be known. Gas well testing data from the DOI-BOEM indicates the well pressure is high enough to produce choking for a significant number of gas wells in the Gulf of Mexico.

For submerged gas leaks that are not choked, high speed video showed a distinct pulsing flow. This has been observed in experiments by others who have reported that the pulsation frequency is an indicator of the gas leak rate. Below is a snapshot of pulsed air flow taken in the Berkeley Tow Tank (Figure 11).
Participated in Small-Scale Oil Leak Jet Experiments at OHMSETT

NETL was invited by the Department of Interior, Bureau of Safety and Environmental Enforcement (BSEE) to participate in small-scale oil leak experiments at the BSEE OHMSETT facility during August of 2013. The OHMSETT facility is the largest water tank in the United States for oil spill research. A small (1/4" diameter exit) oil leak jet was injected at a depth of 10ft. Laser In-Situ Scattering and Transmissiometry (LISST) was used to measure the size distribution of oil droplets with and without Corexit dispersants. The oil leak jets were recorded with high-speed video. Figure 12 contains a snapshot of Endicott crude oil being injected into seawater in the OHMSETT facility. The process of breakup of the crude into droplets is shown.
Deepwater and Ultra-Deepwater Research

DOI/BSEE Awards $738K to Accelerate Development of the ROV Video Technique

Stemming from the work discussed here and supported by the UDW Portfolio, in September 2013 DOI-BSEE awarded this project team $738K for a separate but related proposal. The goal of the new project is to accelerate the development of technology to rapidly and accurately determine flow rates from video obtained by a ROV video of deepwater oil/gas leaks or eruptions. UC Berkeley and OHMSETT are partners in this project.

Extensive Set of Experiments Completed with Structure II Hydrate Forming Gas

An extensive experimental series was completed in NETL’s High-Pressure Water Tunnel Facility (HWTF) in which a gas mixture of methane (87.4%)/ethane (8.1%)/propane (4.5%) gas (referred to as C1C2C3) was used to form bubbles that were studied in 35 salinity seawater. In contrast to pure methane that forms Structure I hydrate, this mixture forms a Structure II hydrate, which requires less severe conditions for stability compared to methane, i.e., higher temperatures or lower pressures. This means a Structure II hydrate could transport gases higher in the ocean water column owing to its increased stability.

A total of 103 individual bubbles were studied in the HWTF over 3 temperature ranges (2-3°C, 6-7°C, and 10-13°C), 3 simulated depths (mainly of 1,000m, 2,000m, and 3,000m) and at dissolved gas concentrations up to that required for stable hydrate formation on or from the bubble. Of these bubbles, 18 formed hydrate (4 at 2-3°C, 8 at 6-7°C, and 6 at 10-13°C). Of those that formed hydrate, all but two were subjected to controlled depressurization studies to simulate rise of the bubble through the oceanic water column. A summary of the individual bubbles observed at the various conditions of temperature, pressure, and dissolved gas content is contained in Table 2. Detailed analysis of the data collected is underway.
Table 2: Summary of Individual C1C2C3 Bubbles in the HWTF in 35 Salinity Artificial Seawater

<table>
<thead>
<tr>
<th>Depth, m (psia)</th>
<th>( X_{C1C2C3} \rightarrow 0 \text{ to } 0.00046 )</th>
<th>( 0 \text{ to } 0.00062 )</th>
<th>( 0 \text{ to } 0.00086 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>200-750</td>
<td>1 w/H</td>
<td>2 w/H</td>
<td>4 w/H</td>
</tr>
<tr>
<td>1000</td>
<td>6</td>
<td>9</td>
<td>22</td>
</tr>
<tr>
<td>2000-2300</td>
<td>4</td>
<td>8</td>
<td>14</td>
</tr>
<tr>
<td>3000</td>
<td>5</td>
<td>13</td>
<td>15</td>
</tr>
</tbody>
</table>

- \( X_{C1C2C3} \): Mole fraction of the C1C2C3 gas mixture dissolved in the water in the HWTF.
- w/H: Number of the total bubbles that formed hydrate.
- 103 individual bubbles were released into the HWTF.
- 18 formed hydrate. 16 underwent some degree of depressurization
- *First 15 bubbles at 2-3°C, 16th bubble at 5°C.
- †First 24 bubbles at 6-7°C, temperature varied from 6 to 12°C for last 8 bubbles.
- ‡First 33 bubbles at 10°C, next 8 bubbles at 11 to 14°C, last 14 bubbles at 10°C.
- §Hydrate formed in cup during injection, lowered pressure to decompose and release. The pressure was then increased to 1000-m simulated depth.

**Bubble Features Identified that have Utility for Video-Based Hydrate Detection**

Based on the 103 bubbles studied with the C1C2C3 gas mixture (see Table 2 above) and the previous 110 bubbles formed from methane, visual features of these bubbles that could be useful for determining the presence of hydrates in a gas-containing deepwater leak or eruption were consistently observed. When a hydrate shell forms on a bubble at appropriate conditions of \( T, P \), and dissolved gas concentration, the following were observed:

- The shell formation process changes the shape of the bubble from ellipsoidal to more oblate.
- The rise velocity of the bubble typically decreases by ~5 to 10%.
- The hydrodynamics of the bubble change from a rotating motion to a more wobbling and rocking motion.
- The reflectivity of the bubble increases.
- The dissolution rate of the bubble decreases by about an order of magnitude.

These are all potentially useful characteristics for development of a video-based hydrate recognition tool. Data analysis is continuing to refine the differences between hydrate-free bubbles and those that are hydrate covered or convert completely to hydrate.

An example illustrating some of these changes is shown in Figure 13.
Figure 13: Comparison of a methane bubble with and without a hydrate shell. The bubble in 13a has no hydrate shell; whereas, the bubble in 13b is covered with a hydrate shell. The differences in reflectivity and shape are apparent. A 1-mm scale bar is shown in both images.

**Manuscript Prepared and Submitted on the Dynamics of Gas Hydrate Morphology on a Methane Bubble**

A manuscript entitled, “Dynamic Morphology of Gas Hydrate on the Surface of a Buoyant Methane Bubble in Water,” has been completed and has been submitted to *Nature* for consideration for publication. This journal is one of the top two general scientific journals in the world. The manuscript contains first-ever, high fidelity, visualizations of the complex, surface mechanisms governing methane bubble hydrate formation and dissociation while rising through a laboratory-simulated deepwater column. This revealed a temporal progression of depth-dependent mechanisms involving synergistic feedbacks between bubble hydrodynamics, hydrate morphology, and surface-coverage characteristics. These unprecedented observations have important implications for modeling deep-sea methane transport to the upper ocean and atmosphere and for predicting the impact from seabed gas/oil eruptions. This potentially high-impact paper was made possible by the investment made in state-of-the-are high-speed, high-definition equipment at the beginning of this project several years ago.

**Theoretical Model Developed for Predicting Phase Behavior and Solubility**

Members of this project team have completed a thermodynamic model capable of calculating gas-water-hydrate and water-hydrate thermodynamic phase stabilities for light hydrocarbons. This model outputs the solubility of hydrocarbon in the aqueous phase in the presence of hydrates, which is necessary to calculate the dissolution rate for each species leaving a hydrate-covered gas bubble as it rises through the ocean column. This model is based on prior models that are well established in the hydrate literature and used throughout academia and industry to accurately predict hydrate thermodynamics. The Langmuir constants, important parameters in these models, have been calculated from ab initio intermolecular potentials for this work. By incorporating these molecular-scale computational chemistry calculations, both the phase equilibria of the two phase water-hydrate system and aqueous phase hydrocarbon solubility can be generated; the latter being a necessary input into ocean bubble models. The accuracy of the model has been confirmed by high-pressure thermodynamic measurements at NETL for hydrate formation from methane in the aqueous phase. Experiments studying the thermodynamics of the C1C2C3 hydrocarbon gas mixture used in the HWTF bubble experiments are continuing. A manuscript describing the thermodynamic model and validation experiments is in preparation.
3.0 Conclusion

The NETL-RUA Complementary Offshore Research portfolio is a suite of seven projects focused on reducing the likelihood and/or mitigating the impact of deleterious environmental events associated with deepwater and ultra-deepwater offshore drilling and production. Collectively, these projects support the broader objective of collecting data and developing and executing models targeting the reduction of risks and environmental impacts associated with offshore oil and gas development and production activities.

UDW energy resources research through NETL ORD is focused on developing a scientific base for predicting and quantifying potential risks associated with exploration and production in extreme offshore environments. This includes projects targeting three core areas:

- **Improving Science-Base for Materials and Wellbore Integrity:** Studying performance and integrity of key offshore materials for which data in extreme environments are limited, including metallic tubulars and borehole cements.
- **Reducing Risks and Impacts Associated with Extreme Offshore Systems:** Developing critical data for predicting in situ conditions required for assessing risk, borehole/drilling design, loss of control conditions in deepwater and ultra-deepwater settings.
- **Improving Safety through Rapid Detection and In Situ Characterization:** Tools and techniques to monitor and quickly detect potential hazards for extreme offshore hydrocarbon.

The above research areas require a combination of laboratory-based experimental and characterization studies, integration and interpretation of field-based datasets, and development and application of numerical simulators. The work is performed by an interdisciplinary team comprised of Federal, contractor, and academic scientists and engineers. For more information and access to a bibliography of publications, presentations, and online access to data and products associated with this portfolio please see https://edx.netl.doe.gov/udw/.

Deepwater and UDW energy resources research through NETL ORD is focused on developing a scientific base for predicting and quantifying potential risks associated with exploration and production in extreme offshore environments. In FY13 notable accomplishments from the UDW Portfolio included:

*Improved Science-Base for Materials and Wellbore Integrity: Studying performance and integrity of key offshore materials for which data in extreme environments are limited, including metallic tubulars and borehole cements.*

Many important findings in this topic area were discovered in FY13, but some of the more significant ones included the following:

- For oil-grade alloy 718, corrosion-fatigue crack growth results showed no obvious effect of 3.5 wt% NaCl solution on the crack growth rates. However, crack growth rate increased in a 21 wt% NaCl for all specimens compared with tests in air and a 3.5 wt% NaCl solution. Crack growth rate differences were more pronounced in the lower ΔK regions where the effects of stress and strain on the crack tip are less dominant. Moreover, aging treatments led to lower corrosion-fatigue crack growth rates of oil-grade alloy 718 in all tested conditions including air.

- FCGR of all grades of line pipe steels increases with decreasing frequency and reaches a plateau for the X52, X65, and X80 grades. This suggests that FCGR propagation in all of the line pipe steels is associated with a hydrogen driven mechanism. No plateau was observed for X100 and 4137M grades, and was likely due to $K_{issc}$ behavior.
The plateau FCGR appears to be independent of the yield strength at the $\Delta K=1000\ N\text{mm}^{-3/2}$ in the mildly sour environment. At a higher value of $\Delta K, 1211\ N\text{mm}^{-3/2}$, FCGR decreases with increasing yield strength.

The effect of yield strength at the higher $\Delta K$ appears to be associated with the shorter diffusion distances in the higher strength steels, coupled with the higher hydrostatic stresses which enhance the concentration of hydrogen.

For atmospheric foam cements, experiments conducted in FY13 indicate a relationship between physical properties of the cement and the quality of the foam. A draft TRS correlating the foam quality with the mechanical properties (permeability, porosity, and strength) is complete and undergoing review by the team. Ultrasonic velocity measurements have started and are ongoing.

Initiated a novel computational modeling effort that will support improved understanding of the spatial distribution of gas bubbles in foamed cement systems and thus have implications for understanding and predicting the integrity of the foam cement barrier in the wellbore environment.


First group to identify and characterize a viable fluid that exhibits the requirements of a desired deepwater viscosity standard.

First group to report cyclic hydrocarbon liquid-solid phase transition solidification data.

First group to extend viscosity model of pure hydrocarbons to HPHT conditions.

Completed development of the beta version of BLOSOM which is a comprehensive oil spill occurrence model predicting the behavior and fate of spill behavior from the well head to the shore for a variety of hydrocarbon mixtures.

BLOSOM was invited to participate in an ongoing API oil spill model comparison effort which is assisting with the shakedown and testing of this new simulation tool.

Released the GOM Geocube which supports timely access to current information for the offshore GOM and encompasses the natural system and engineered components from the subsurface to the shore and now includes atmospheric information feeds as well. Geocube is publically available but is also being leveraged by the GOM IAM team for our risk evaluations. [https://edx.netl.doe.gov/gom-geocube/](https://edx.netl.doe.gov/gom-geocube/)

**Improving Safety through Rapid Detection and In Situ Characterization: Tools and techniques to monitor and quickly detect potential hazards for extreme offshore hydrocarbon.**

- Video Analysis of Submerged Natural Gas Leaks. For submerged gas leaks that are not choked, high speed video showed a distinct pulsing flow. This pulsation frequency is an indicator of the gas leak rate.

- Participated in Small-Scale Oil Leak Jet Experiments at OHMSETT. NETL was invited by the Department of Interior, Bureau of Safety and Environmental Enforcement (BSEE) to participate in small-scale oil leak experiments at the BSEE OHMSETT facility. This work is vital to the calibration and accuracy of future oil spill predictions.

- DOI/BSEE Awards $738K to Accelerate Development of the ROV Video Technique. Research under this project resulted in an additional award for a proposal based on this effort. This new BSEE award will allow for further validation of this plume imaging effort.

- Extensive set of experiments completed with structure II hydrate forming gas was produced based on the experimental work in this project.

The above research utilizes a strategic combination of laboratory-based experimental and characterization studies, integration and interpretation of field-based datasets, and development and application of numerical simulators. The work is performed by an interdisciplinary team comprised of Federal, contractor, and academic scientists and engineers. For more information and access to a bibliography of publications, presentations, and online access to data and products associated with this portfolio please see [https://edx.netl.doe.gov/udw/](https://edx.netl.doe.gov/udw/).
4.0 Technology Transfer

Presentations

*Improved Science-Base for Materials & Wellbore Integrity*


**Reducing Risks & Impacts Associated with Extreme Offshore Systems**


Publications

**Improved Science-Base for Materials & Wellbore Integrity**


**Reducing Risks & Impacts Associated with Extreme Offshore Systems**


**Improving Safety through Rapid Detection & In Situ Characterization**


Tools Developed and Released via NETL’s EDX (https://edx.netl.doe.gov)

In FY13, a dedicated website was developed for the UDW portfolio to communicate to the public the purpose, background information, and results of work conducted by the EPAct UDW Portfolio: https://edx.netl.doe.gov/udw.

NETL’s GOM Geocube focuses on the offshore Gulf of Mexico region. NETL's Geocubes are flexible, customizable web mapping application that concatenates key spatial datasets and information relevant to energy research needs for a given region.

gewELL was developed in collaboration with the UDW Portfolio. geowell is a map-based application that provides quick access to websites of primary sources of subsurface geologic and wellbore information for appropriate U.S. state, tribal and federal agencies.

Energy Data Exchange is a tool to support coordination and collaboration across research efforts that require a common set of information related to subsurface energy resources. The UDW Portfolio supports ongoing development of the system as a whole, as well as addition of key content that is provided through NETL’s EDX.

Dataset Released via NETL’s EDX (https://edx.netl.doe.gov)

EOS-PVT HTHP Pentane Density Data
Density data for n-pentane. Temperature Range: 325K to 520K Pressure Range: 1.8 MPa to 275 MPa

HTHP Decane Viscosity Data
Viscosity data for n-decane Pressure Range: 4.7 -238.8 MPa Temperature Range: 303-520K

HTHP Dioctyl Phthalate DOP Viscosity Data
Viscosity Data for Dioctyl Phthalate (DOP) Pressure range: 2.3 - 259.9 MPa Temperature range: 298 - 533K

HTHP Eicosane Viscosity Data
Viscosity Data for n-eicosane Pressure range: 2.0 - 243.1 MPa Temperature range: 323 - 520K

HTHP Hexadecane Viscosity Data
Viscosity data for n-hexadecane Pressure range: 3.3 - 226.6 MPa Temperature range: 303 - 520K

HTHP Krytox GPL Oil Viscosity
Viscosity Data for Krytox GPL 102 (Perfluoropolyether Oil) Pressure range: 8.0 - 245.1 MPa Temperature range: 311 - 533K

HTHP Octadecane Viscosity Data
Viscosity Data for n-octadecane Pressure range: 6.4 - 243.1 MPa Temperature range: 323 - 520K
5.0 References


The National Energy Technology Laboratory (NETL) conducts cutting-edge energy research and technology development and analyzes energy systems and international energy issues for the U.S. Department of Energy. The NETL Regional University Alliance (NETL-RUA) is an applied research collaboration that combines NETL’s energy research expertise with the broad capabilities of five nationally recognized, regional universities: Carnegie Mellon University (CMU), The Pennsylvania State University (Penn State), University of Pittsburgh (Pitt), Virginia Tech, and West Virginia University (WVU), and the engineering and construction expertise of an industry partner (URS). NETL-RUA leverages its expertise with current fossil energy sources to discover and develop sustainable energy systems of the future, introduce new technology, and boost economic development and national security.
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