Hydrate-Bearing Clayey Sediments: Morphology, Physical Properties, Production and Engineering/Geological Implications

Project Period (10/1/2012 to 9/30/2016)

Submitted by:
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ACCOMPLISHMENTS

Context – Goals. Fine grained sediments host more than 90% of the global gas hydrate accumulations. Yet, hydrate formation in clayey sediments is least understood and characterized. This research focuses on hydrate bearing clayey sediments. The goals of this research are (1) to gain a fundamental understanding of hydrate formation and ensuing morphology, (2) to develop laboratory techniques to emulate “natural” formations, (3) to assess and develop analytical tools to predict physical properties, (4) to evaluate engineering and geological implications, and (5) to advance gas production alternatives to recover methane from these sediments.

Accomplished
The main accomplishments for this period include:

- Formation of CO₂ hydrate in fine-grained sediment
  - Transformation from ice/water to hydrate in hydrophobic silica
- Quantified mass, and advanced thermal analysis of hydrate formation in fine-grained sediment
- Crystal formation experiments in porous media

Plan - Next reporting period
Physical understanding of hydrate formation in fine grained sediments and small pores. Evaluate the difference between gas pressure, liquid pressure and crystal pressure, and the relevance to hydrate stability. Advance Numerical model studies of physical properties of hydrate bearing sediments. Well production simulation with numerical methods.

Research in Progress
The following pages capture the slides presented at the meeting for the end of year 3, which include specific information about this quarter.
Hydrate-Bearing Clayey Sediments
Morphology, Physical Properties, Production and Engineering/Geological Implications

Transition to Phase 4 / Budget Period 4

DOE - National Energy Technology Laboratory
Agreement: DE-FE0009897

J. Carlos Santamarina
Georgia Institute of Technology
(on leave at KAUST)

Goals – Objectives - Background

Natural HBF – Fine Grained (Analogues)
Underlying Physics
Devices
Hydrate Formation in the Lab
“Reservoir” Simulation
Physical Properties
Gas Production

Next – Team – Schedule
Goals and Objectives

Background

(Additional examples: see 2014 End of Year Report)

Observation: Fine grained sediments
  • host more than 90% of the global gas hydrate accumulations

State-of Knowledge: Hydrate formation in clayey sediments
  • least understood
  • poorly characterized

Objectives:
  • in-depth understanding ofhydrate bearing fine-grained sediments
  • new gas production paradigm

(FITL, Fall 2006)
Goals and Objectives

*The proposed research*
- focus: hydrate bearing clayey sediments
- fundamental understanding of hydrate formation
- hydrate lens topology
- laboratory techniques to emulate "natural" formations
- analytical tools to predict physical properties
- engineering and geological implications
- gas production alternatives

Project Tasks

*Focus:* hydrate bearing clayey sediments

*Tasks:*
- fundamental understanding of hydrate formation in fine-grained sed.
- laboratory emulation with real methane hydrate
- assessment and prediction of physical properties
- evaluation of engineering and geological implications
- possible paradigm shift in gas production from fine-grained sed.
Task 2 - Formation, distribution, topology

Guiding Questions:
• nucleation and grow in fine-grained sediments?
• continue feeding lens growth?
• underlying hydro-chemo-mechanical effects?
• sediment characteristics that control evolving hydrate topology?
• emulation in the laboratory?

Laboratory challenges:
CH₄ in hydrate = 1:6 >> CH₄ in water = 1:700
Hydrate formation transport-limited in water saturated sediments
Low advective transport in clayey sediments (diffusive transport?)

Task 3 - Physical properties

Guiding Questions.
Hydro-thermo-electro-mechanical properties of fine-grained sediments with segregated hydrate?
(relevance to simulators)

SubTask 3a: Analytical estimations (two-component systems)
• upper and lower bounds
• physical models

SubTask 3b: Numerical Extension (interacting lenses)

SubTask 3c: Experimental measurements
• Form hydrate-bearing clays and measure salient physical properties
• Small strain stiffness (V_p and V_s), strength
• Thermal, hydraulic and electrical conductivity
Task 4 - Gas Production

**Guiding Questions**
- What are viable production strategies?
- Gas migration: from lenses towards to the production well?
- What is the role of elastic deformation of layer boundaries?
- Production strategy to keep fractures open during recovery?
- Can discontinuities become "highways" for gas flow?
- Production strategies to minimize volume contraction?

**Description**
- Target: *new gas production paradigm*
- From key differences with oil production
  - interconnected hydrate network
  - hydrate-to-fluid expansion
  - gas-driven openings
  - gas migration in layered stratigraphy
  - available heat

Task 5 - Implications

**Guiding Question:**
*How does segregated hydrate in fine grained sediments affect engineering tasks (besides production) and geological processes?*

SubTask 5a – Seafloor infrastructure settlement
SubTask 5b – Stability (borehole and slopes)
SubTask 5c – Unique implications to carbon cycle
Natural HBS - *Fine Grained* Analogues

(additional examples: see 2014 End of Year Report)
Gulf of Mexico, US

smectite-dominant clay

Photos: GEOMAR

Hydrate Ridge, US

clay sediments (smectite, illite, chlorite, and kaolinite)

Photos: GEOMAR
Crystallization in Fine Grained Sediments

Emerald

Pyrite sun


Crystallization in Fine Grained Sediments

**Gypsum lenses**

Underlying Physics

(additional information: see 2014 End of Year Report)
Crystal Growth in Sediments: Mechanics

\[ \gamma_{\text{ice-soil}} > \gamma_{\text{ice-water}} + \gamma_{\text{soil-water}} \quad (\gamma: \text{Interfacial energy}) \]
Invasion-Driven Fracture
Tip Conditions

\[ \Delta P = \frac{\rho \sigma_s S_\delta}{e} \]

\( e = 2.078 \)

\( e = 2.231 \)

\( e_0 = 2.105 \)

Sediment in compression EVERYwhere

Crack/Lens Propagation

Experimental

Numerical (MCC)

Displacement & normal to crack plane

Displacement & // crack alignment
Pressure Diffusion

T = 10^{-4} \quad T = 5 \times 10^{-4} \quad T = 0.12

Pressure Diffusion

Invasion vs. Localization

INVASION
Fluid invasion
Crystal growth in pores
Hyd.: patchy saturation

\[ F_c = \frac{2\pi \sigma_{LV}}{\sigma' d} \]

LOCALIZATION
Lenses
Fractures
Hyd.: lenses

course grained soils
high effective stress

fine grained soils
low effective stress
Hydrate Topology

\[ \psi = \frac{10 \gamma_{\text{water}}}{d_{10} \sigma} = 10^{-3} \]

\[ \psi = \frac{10 \gamma_{\text{water}}}{d_{10} \sigma} = 1 \]

Effective stress \( \sigma' \) [MPa]

Characteristic particle size \( d \) [\( \mu \text{m} \)]

Pore-filling

Lenses/Veins

Mt. Elbert

NGHP

Blake Ridge

Effective stress \( \sigma' \) [MPa]

Nodules/Chunks

Devices

(additional information: see 2014 End of Year Report)
Chamber (X-ray transparent)

X-ray CT Scanner
X-ray CT Scanner

Calibration: specimen and protocol

- Tilt – angle tilt of rotary stage relative to detector
- SDD – source-to-detector distance
- SOD – source-to-object distance

Hydrate Formation in the Lab

(additional information: see 2014 End of Year Report)
**Task 2**

Laboratory protocol to form hydrate bearing clayey sediments

*Dispersed nucleation followed by increased in effective stress and aging*
- dispersed nucleation (partial water saturation, ice-seeding, and ground hydrate premixing)
- aging (e.g., thermal cycling) + stress: segregation into lenses?

*Saturated blocky sediment + hydrate formation along discontinuities*
- gas-driven fractures
- shear bands
- sediment slicing with intersecting planes
- pre-flushing freezing air $\Rightarrow$ gas flow + pressurization

---

**Challenges in Fine-grained Sediments**

- **Fine-grained**
- **Small pores**
  - Low conductivity
  - Difficult nucleation
  - Long time-scale
  - Morphology
  - Extra driving force
Method 1: Spontaneous nucleation

Henry's law:

\[ M_{p,T} = P_{\text{applied}} k_H^0 \cdot \exp \left[ -\frac{\Delta H}{R} \left( \frac{1}{T} - \frac{1}{298.15 \text{K}} \right) \right] \]

- Concentration
- Enthalpy of the solution
- Henry's law constant
- Universal gas constant

\[ \Delta H = -14130 \text{ J/mol} \]

\[ k_H^0 = 1.3 \times 10^3 \text{ M/atm at 298.15 K} \]

\[ R = 8.314 \text{ J/(mol·K)} \]

<table>
<thead>
<tr>
<th>Methane concentration [mol/kg]</th>
<th>Without hydrate (C_{in})</th>
<th>Salt Water (con. of NaCl)</th>
<th>Pure water</th>
<th>Salt water (con. of NaCl)</th>
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</thead>
<tbody>
<tr>
<td>Pure water</td>
<td>0.11 (273K,3MPa)</td>
<td>0.00177 (1m)</td>
<td>0.065 (274K,3.5MPa)</td>
<td>0.05184 (273K,10MPa)</td>
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<td>Salt Water (con. of NaCl)</td>
<td>0.0974 (273K,6MPa)</td>
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<tr>
<td>Pure water</td>
<td>0.12 (276K,6.6MPa)</td>
<td>0.067 (273K,0.1MPa)</td>
<td>0.066 (274K,6.5MPa)</td>
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<tr>
<td>Salt Water (con. of NaCl)</td>
<td>0.13 (275K,10MPa)</td>
<td>0.067 (273K,0.1MPa)</td>
<td>0.09689 (273K,10MPa)</td>
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</tr>
<tr>
<td>Pure water</td>
<td>0.00247 (273K,0.1MPa)</td>
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</tbody>
</table>

Initial methane concentration: \( C_p = 0.14 \text{ mol/kg (P=12MPa and T=288K)} \)

CH4 solubility – hydrate present: \( C_{in} = 0.063 \text{ mol/kg} \)

CH4 concentration in hydrate: \( C_h = 8.06 \text{ mol/kg} \)

Lense: \( L = 4 \text{ mm} \) every \( L = 1 \text{ m} \)
Method 2: Avoid Diffusion $\rightarrow$ THF Hydrate

Ice starts pre-melting at the surface when $T = -33 \, ^\circ C$

The structure does not fully solidify until 0K (Li, and Somorjai, 2007)

Multiple Methods: Ice $\rightarrow$ Hydrate

Ice starts pre-melting at the surface when $T = -33 \, ^\circ C$

The structure does not fully solidify until 0K (Li, and Somorjai, 2007)
Method 3: Water sat + Freeze + I→H

![Graph showing pressure and temperature changes during the method](image)

Method 4: Gas sat + Stability PT + Water

![Graph showing pressure and temperature changes during the method](image)

*Store gas into diatom inner pores
Stimulated by diatoms found in NanKai Trough*
Method 5: Gas sat + Water + Stability PT

Store gas into diatom inner pores
Stimulated by diatoms found in NanKai Trough
Method 6: Water sat + Ice + I → H
Method 7: Dry + Ice + Stability PT

Strategy: Supply gas through dry sediment

before

after

Kaolin

Method 8: Hydrophobic + Ice Lens + I → H

large lense
Method 8: Hydrophobic + Ice Lens + I→H

![Diagram showing pressure vs. temperature with phase boundaries and scan points]

Scan 1 → Scan 2 → Scan 4 → Scan 6

Method 8: Hydrophobic + Ice Lens + I→H

![Images of small lenses and scans]

small lens

small lense
Method 9: Water sat + Stability + CO$_2$ inject

**Diatom-sand layers**

Method 9: Water sat + Stability + CO$_2$ inject

**Kaolinite**

Gas driven fracture induced hydrate formation
Gas driven fracture induced hydrate formation

Kaolinite
After dissociation

Kaolinite

Method 10: Gas-injection into Clay Slurry
“Reservoir” Simulation

**Mass: Measurement vs. Simulation**

- Temperature
- Pressure
- Volume of voids $V$
- $\rho_{\text{CO}_2} = f(P,T)$
- Mass of gas phase $\text{CO}_2$
  $M_{\text{CO}_2}(t) = f(V, \rho_{\text{CO}_2})$
- Initial mass of $\text{H}_2\text{O}$
- Hydrate formation/dissociation rate, leakage
Thermal Analysis

\[ \rho_{CO2} = f(P, T) \]
No leakage, constant

**Theoretical real gas Tr Temperature**

**Heat redistribution in sediment**

**Theoretical real sediment Tr Temperature**

**Thermocouple Lag**

**Heat diffusion to the environment**

**Predicted sediment Tr Temperature measurement**

**Measured Tr Temperature**

**Measurement vs. Simulator**

---

**Thermal Analysis - Thermocouple Response**

\[
\frac{dQ}{dt} = \pi r_i^2 \rho_i c_i \frac{d(T_i(t))}{dt} \quad q[t] = \frac{dQ}{dt} = h2\pi r_i l (T_i(t) - T_w(t))
\]

\[
q^i = h2\pi r_i l \left[ \left( T_{wc}^i - T_i^i \right) + \left( T_{wc}^{i+1} - T_i^{i+1} \right) \right] / 2
\]

\[
T_{i+1} = T_i^i + \frac{q^i \Delta t}{\pi r_i^2 \rho_i c_i}
\]

\[
T_{i+1} = T_i^i + \alpha (T_{wc}^i - T_i^i + T_w^{i+1}) / (1 + \alpha)
\]

Where:

\[
\alpha = \frac{h \Delta t}{\pi r_i \rho_i c_i}
\]
Thermal Analysis-Thermocouple Lag

Thermocouple response during step temperature change in sediment

Thermocouple response during gas injection in air

Thermal Analysis – 3D FEM
**Physical properties**

(Additional information: see 2014 End of Year Report)

### Properties - Needs

<table>
<thead>
<tr>
<th>Category</th>
<th>Needs</th>
</tr>
</thead>
</table>
| **Mechanical** | - Borehole stability  
|             | - Seafloor subsidence  
|             | - Slope stability / Submarine landslides                             |
| **Thermal**  | - Reservoir modeling                                                |
|             | - Production enhancement                                            |
| **Hydraulic** | - Hydraulic fracturing                                              |
|             | - Water production                                                  |
| **Electrical** | - Saturation estimations                                           |
|             | - Fracture tomography                                               |
Physical Properties

Numerical Simulations
- Idealized topologies
- Physics inspired
- Logging
- Pressure core

Effective Media Models

Bounds

$k_{\text{parallel}} = \sum x_i \cdot k_i$
$k_{\text{water}} = \frac{\sum x_i}{\sum k_i}$
Cryogenic Suction Induced Consolidation

Gas Production

(additional information: see 2014 End of Year Report)
Gas Production

Prevailing Paradigm:
• Based on oil production

Consequently: Clayey sediments are not considered good prospects
• low permeability
• unacceptable high settlements if production by depressurization
• available technology driven by petroleum production
• lack of economically viable production concepts

Gas production from hydrate-bearing – All tested in sands
• depressurization
• heating
• inhibitors (including CO₂-CH₄ replacement)

Keys for a Paradigm Shift?

Key #1: Interconnected hydrate lenses

Korean cores - Park et al., 2009
**Key #2: Volume expansion**

\[
\beta = \frac{V_g + V_w}{V} = \frac{z\chi RT_g}{P_m + \frac{2\Gamma}{R_m} \cos \theta} + \frac{18 \chi \rho_n}{16 + 18 \chi \rho_w}
\]

**Key #3: Gas driven fractures**
Key #4: Migration in layered sediments

- Pressure Panel
- Filter
- 90 cm

Pressure vs. Time graph:
- X: Stop air injection
- O: Start air injection
- AEV: 1.3 [kPa]
- Hydraulic effect: 0.3 [kPa]

Key #5: Heat Source

- Initial condition with hydrate
- Case 1 (no ice formation)
- Case 2 (Ice formation)

- $S_h + S_w = 1$
- $P = 3$ MPa
- $T_{eq} = 2.5^\circ C$

Graph showing ice fraction $F_{ic}$ vs. hydrate saturation $S_h$
But … #1: Limited Volume of Influence

\[ K_1 \left( \frac{k' \cdot 2 \cdot r^2}{k_{sw} \cdot h} \right) = K_2 \left( \frac{k' \cdot 2 \cdot r^2}{k_{sw} \cdot h} \right) \cdot \frac{\Delta h}{R_{sw}} \cdot (r^2 - r'^2) \]

Embodied energy EE [J]

\[ EE = \varepsilon \cdot n \cdot S_{sw} \cdot V \cdot E \cdot d_{sw} \]
But … #2: Sediment-Well Interaction

Goals – Objectives - Background

Natural HBF – Fine Grained (Analogues)
Underlying Physics
Devices
Hydrate Formation in the Lab
“Reservoir” Simulation
Physical Properties
Gas Production

Next – Team – Schedule
Coming up?

**Experimental**
Extend lens formation after injection
Formation in slurries: shallow accumulations

**Numerical**
Extend to real topologies

**Production**
Emphasis on shallow accumulations

Team:

Liang Lei (4th year)
Seth Mallett (3rd year)
NN (1st year)

Sheng Dai

Marco Terzariol (Production – GT/KAUST)
Junbong Jang (Production – GT/KAUST)
Hosung Shin (Well-sediment – Ulsan U.)
## Schedule

<table>
<thead>
<tr>
<th>Task / SubTask</th>
<th>YEAR 1</th>
<th>YEAR 2</th>
<th>YEAR 3</th>
<th>YEAR 4</th>
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<tbody>
<tr>
<td><strong>1.0 – PMP</strong></td>
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<tr>
<td><strong>2.0 – Formation &amp; morphology</strong></td>
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<td>2a: Literature review</td>
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<td>2a: Laboratory protocol</td>
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<td>2c: X-ray tomography</td>
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<td><strong>3.0 - Physical properties</strong></td>
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<td>3a: Analytical estimations</td>
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<td>3b: Numerical Extension</td>
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<td>3c: Measurements</td>
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<td><strong>4 - Gas Production</strong></td>
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<td>4a: Experimental Study</td>
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<td>4b: Modeling</td>
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<td><strong>5 – Implications</strong></td>
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<td>5a: Settlement</td>
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<td>5b: Stability</td>
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<td>5c: Implications C-cycle</td>
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## MILESTONE LOG

<table>
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<th>Milestone</th>
<th>Planed completion date</th>
<th>Actual completion date</th>
<th>Verification method</th>
<th>Comments</th>
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<tbody>
<tr>
<td>Literature review</td>
<td>5/2013</td>
<td>5/2013</td>
<td>Report</td>
<td>Completed first phase. Will continue throughout the project</td>
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<tr>
<td>Preliminary laboratory protocol</td>
<td>8/2013</td>
<td>8/2013</td>
<td>Report (with preliminary validation data)</td>
<td>this and previous reports</td>
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<tr>
<td>Cells for Micro-CT</td>
<td>8/2013</td>
<td>8/2013</td>
<td>Report (with first images)</td>
<td>this and previous reports</td>
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<tr>
<td>Compilation of CT images: segregated hydrate in clayey sediments</td>
<td>8/2014</td>
<td>In progress</td>
<td>Report (with images)</td>
<td>Observed in experiments. Gas production engineering is conducted analytically/numerically</td>
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<td>Preliminary experimental studies on gas production</td>
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<td>12/2014</td>
<td>Report (with images)</td>
<td>Observed in experiments. Gas production engineering is conducted analytically/numerically</td>
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<td>Analytical/numerical study of 2-media physical properties</td>
<td>5/2015</td>
<td>6/2015</td>
<td>Report (with analytical and numerical data)</td>
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<tr>
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<td>12/2015</td>
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<td>Report (with data)</td>
<td>Observed in experiments. Gas production engineering is conducted analytically/numerically</td>
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<tr>
<td>Early numerical results related to gas production</td>
<td>5/2016</td>
<td>In progress</td>
<td>Report</td>
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<tr>
<td>Comprehensive results (includes Implications)</td>
<td>9/2016</td>
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<td>Comprehensive Report</td>
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</table>

## PRODUCTS

- **Publications**: In progress
- **Presentations**: In progress
- **Website**: Publications and key presentations are included in [http://pmrl.ce.gatech.edu/](http://pmrl.ce.gatech.edu/) (for academic purposes only)
- **Technologies or techniques**: X-ray tomographer and X-ray transparent pressure vessel
- **Inventions, patent applications, and/or licenses**: None at this point.
- **Other products**: None at this point.
PARTICIPANTS & OTHER COLLABORATING ORGANIZATIONS

Research Team: The current team is shown next. We anticipate including external collaborators as the project advances.

- **PI:** J. Carlos Santamarina
- **Admin. support:** Rebecca Colter
- **PhD #1**
  - Liang Lei
- **PhD #2**
  - Seth Mallett
- **URA - Summer**
  - A. Garcia

IMPACT
While it is still too early to assess impact, we can already highlight preliminary success of exploring hydrate lenses morphology in real systems, and analogue studies using a high resolution tomographer.

CHANGES/PROBLEMS:
None at this point.

SPECIAL REPORTING REQUIREMENTS:
We are progressing towards all goals for this project.

BUDGETARY INFORMATION:
As of the end of this research period, expenditures are summarized in the following table. Note: in our academic cycle, higher expenditures typically take place during the summer quarter.
<table>
<thead>
<tr>
<th>Baseline Reporting Quarter DE-FE009897</th>
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<td>-7,512</td>
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