Ceramic Castable Cement Tanks and Piping for Molten Salt

PI: Asegun Henry, Massachusetts Institute of Technology
Co-PI: Kenneth Sandhage, Purdue University
Co-PI: Kenneth McGowan & Bob Cullen, Westmoreland Advanced Materials

Technology Addressed
Tanks and Piping for 750°C Molten Chloride Salts

Innovative Aspect
The usage of ceramic castable cements to make inexpensive tanks and piping. Castable cements simply require the addition of water to form an emulsion, which can then be poured into a mold of any desired shape and then cured.

Impact
• Cheaper materials that are compatible with molten salts allow for a less expensive infrastructure and enable achievement of the SunShot goals for the cost of storage

Background and Proposed Work
• Currently envisioned materials (H230 and 740H) for the infrastructure are ~ 4X more expensive than SS316. Castable cements are much less expensive.
• Demonstrate a lab scale tank and flanged pipe section, to prove that flowing salt doesn’t corrode
• Optimize the castable chemistry and microstructure to minimize penetration
• Test a cast flanged pipe section for leakage under pressure
• Develop a cost model for the tank and pipes

Key Milestones & Deliverables

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What is the problem at 750°C?

Conventional welded metal is too expensive because of corrosion.

SOLUTION = Use ceramics

Castable cements from WAM

Add water, mold into shape, cure in place

Very inexpensive $5,950/m³

New internal insulation architecture

Redundant leak protection mechanisms

Can also use for piping

Multiply by 4X for Ni alloys $\rightarrow$ > $20$/kWh-t for the tank alone

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Table 1. Component costs for direct, two-tank molten salt TES system (base case).

<table>
<thead>
<tr>
<th>Component</th>
<th>Materials ($/kWh_t)</th>
<th>Installation ($/kWh_t)</th>
<th>Total Cost ($/kWh_t)</th>
</tr>
</thead>
<tbody>
<tr>
<td>High-temperature tank—stainless steel</td>
<td>5.20</td>
<td>1.84</td>
<td>7.04</td>
</tr>
<tr>
<td>Low-temperatures tank—carbon steel</td>
<td>1.30</td>
<td>1.84</td>
<td>3.14</td>
</tr>
<tr>
<td>Tank supports, foundations, and site work</td>
<td>1.10</td>
<td>1.55</td>
<td>2.65</td>
</tr>
<tr>
<td>Storage medium</td>
<td>11.74</td>
<td>0.36</td>
<td>12.10</td>
</tr>
<tr>
<td>Electrical and instrumentation</td>
<td>0.47</td>
<td>0.43</td>
<td>0.90</td>
</tr>
<tr>
<td>Piping, valves, and fittings</td>
<td>0.20</td>
<td>0.18</td>
<td>0.39</td>
</tr>
<tr>
<td>Totals</td>
<td>20.01</td>
<td>6.22</td>
<td>26.22</td>
</tr>
</tbody>
</table>

G. Glatzmaier, Report No. NREL/TP-5500-53066

Molten Salt (MgCl₂-KCl)

High Density Inner Liner (e.g., WAM-BLG)

Insulating Porous Second Layer (e.g., WAM-ALII type)

Carbon Steel Outer Containment

If a crack forms the salt will penetrate and form a freeze plane

---

Image of a molten salt tank, showing its construction and parts. The tank is large and circular, with various components labeled for insulation and containment.
INNOVATION

- A different design & class of materials
- Lower cost material that is easy to fabricate
  - Add water & mold
  - Ability to tune/engineer the composition/microstructure
- Redundant leak protection
- Also use as pipes, with cast flanges

![Diagram of Molten Salt (MgCl₂-KCl) High Density Inner Liner (e.g., WAM-BLG) Insulating Porous Second Layer (e.g., WAM-ALII type) Thermal Insulation Carbon Steel Outer Containment. If a crack forms the salt will penetrate and form a freeze plane.]

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- Lower cost material that is easy to fabricate
  - Add water & mold
  - Ability to tune/engineer the composition/microstructure
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- Also use as pipes, with cast flanges
Ceramic Castable Cement Tanks and Piping for Molten Salt

What are the potential issues?

• Reaction with salt
• Convective enhancement
• Salt penetration into pores
• Tank wall cracking
• CTE mismatches between layers
• Seal leakage at pipe flanges
• Cost
Ceramic Castable Cement Tanks and Piping for Molten Salt

What are the project objectives?

(1) Demonstrate minimal/acceptable salt penetration
(2) Demonstrate corrosion resistance to flowing salt
(3) Demonstrate pipe sections with flanged interfaces and no leakage
(4) Demonstrate $\leq$ $15$/kWh-t via a comprehensive cost model

Prototype System Test Rig
What are we proposing to do?

• Reaction with salt
  --- *Testing modified compositions with small crucibles*

• Salt penetration into pores
  --- *Testing full tanks and pipe sections in prototype loop (salt level in tank - static)*

• Convective enhancement
  --- *Testing full tanks and pipe sections in prototype loop (salt level in tank - flowing)*

• Tank wall cracking
  --- *Thermal cycles during prototype loop tests*

• CTE mismatches between layers
  --- *Measure CTE + 3D Modeling*

• Seal leakage at pipe flanges
  --- *Testing pipe section interfaces under applied pressure*

• Cost
  --- *3D modeling + Cost model (materials + labor etc.)*
## Ceramic Castable Cement Tanks and Piping for Molten Salt

### TIMELINE

| TASKS                                       | Month: 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 |
|---------------------------------------------|----------|---|---|---|---|---|---|---|---|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| **TASK 1: Test a tank and flanged pipe section** |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.1 – Finalize the tank and system design |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.2 – Cast the tank and pipe section   |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.3 – System construction              |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.4 – Preliminary testing, debugging and 24 hr test |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.5 – Extended test at ≥ 750°C for ≥ 100 hrs |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 1.6 – Leak test sealed pipe section ≥ 750°C |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| **TASK 2.0: Optimizing the microstructure/chemistry** |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 2.1 – Fabrication of test crucibles   |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 2.2 – Pretest characterization         |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 2.3 – 100 hr tests at 750°C            |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 2.4 – Post mortem characterization and analysis |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| **TASK 3: Property measurements**          |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 3.1 - Sample fabrication              |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 3.2 – Measure CTE from 25-900°C       |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 3.3 – Measure thermal diffusivity from 25-900°C |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 3.4 – Profilometry measurements       |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| **TASK 4: Full scale tank design and technoeconomics** |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 4.1 – Initial design for thermal considerations |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 4.2 – Revised tank design for thermomechanical considerations |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
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| Task 4.4 – Tank cost model                 |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
| Task 4.5 – Pipe cost model                 |          |   |   |   |   |   |   |   |   |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |    |
TASK 1: PROTOTYPE TEST LOOP-

<table>
<thead>
<tr>
<th>TASKS</th>
<th>Month:</th>
<th>Q1</th>
<th>Q2</th>
<th>Q3</th>
<th>Q4</th>
<th>Q5</th>
<th>Q6</th>
<th>Q7</th>
<th>Q8</th>
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Intermediate reservoir

2 Gallon Tank

Pipe Test Section

W Pump

Motor

Scale
Ceramic Castable Cement Tanks and Piping for Molten Salt

TASK 2: CEMENT CHEMISTRY/MICROSTRUCTURE OPTIMIZATION

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**TASK 2.0: Optimizing the microstructure/chemistry**

- **Task 2.1 – Fabrication of test crucibles**
- **Task 2.2 – Pretest characterization**
- **Task 2.3 – 100 hr tests at 750°C**
- **Task 2.4 – Post mortem characterization and analysis**

An example of failure with another salt

![Example of failure](image)
Ceramic Castable Cement Tanks and Piping for Molten Salt

**TASK 3: PROPERTY MEASUREMENTS**

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Netzsch TMA 402 F1 Hyperion
Steel Furnace Up to 1100C

Netzsch LFA 467
Furnace Up to 1250C

Handheld Profilometer
10 micron resolution
Ceramic Castable Cement Tanks and Piping for Molten Salt

TASK 4: FULL SCALE DESIGN & COST MODELS

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CTE match → Tanks grow together

Mo/W/Inconel Bolts
Grafoil gasket
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Resources ($)
<table>
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<tr>
<th></th>
<th>Total Project</th>
<th>DOE Funds</th>
<th>Cost Share</th>
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</thead>
<tbody>
<tr>
<td>$2,214,831</td>
<td>$1,771,798</td>
<td>$443,033</td>
<td></td>
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Key Milestones & Deliverables

Year 1
• Demonstrate the system is fully constructed and working with a molten salt and a flow rate between 0.1-5 gpm
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• Demonstrate that the castable cement tank and pipe section do not leak and do not exhibit excessive penetration when exposed to flowing salt
• Demonstrate that the cost of the tank and piping meets the SunShot goals
Ceramic Castable Cement Tanks and Piping for Molten Salt

Why ceramic lined pipes won’t work

\[ \alpha \cdot \Delta T \cdot L_0 \]

\[ 20e^{-6K^{-1}} \times 725K \times 6m = 0.087m \]

\[ 5e^{-6K^{-1}} \times 725K \times 6m = 0.022m \]

2.5” 750°C 25°C
Why tank thermal cycles are not very significant

\[ R_{tot} = R_{conv} + R_{cond} + R_{conv} \]

\[ \frac{d}{dt} \left( m \cdot C_p \cdot T \right) = \frac{(T_{salt} - T_\infty)}{R_{tot}} \]

\[ T = T_\infty + \left( T_0 - T_\infty \right) \exp(-t / \tau) \]

\[ \tau = m \cdot C_p \cdot R_{tot} \]

The time constant is large because the mass is large.

\[ \tau \sim 50 \text{ days} \]
Ceramic Castable Cement Tanks and Piping for Molten Salt

Addressing Crack Formation and Propagation

Particle Packing
Choice of Material
  Perm. Lin. Change
Water Content
  Surfactant Choice
Dryout Schedule
Compression
Use of Large Aggregate or Fiber
  Work of Fracture
Ceramic Castable Cement Tanks and Piping for Molten Salt

Abrasion Resistance

ASTM C-704
Ceramic Castable Cement Tanks and Piping for Molten Salt

Alkali and Alkaline Earth Resistance

Metallics Grain Boundary Attack
Grain Size Change (temp)

Ceramics Grain Boundary Attack

Method of Use of Calcium Hexa Aluminate Refractory Linings and/or Chemical Barriers in High Alkali or Alkaline Environments
CANADA 0047205-000043 DCA 9/19/2007 2,663,798 1/19/2016 2,663,798 ISSUED

With ORNL
Ceramic Castable Cement Tanks and Piping for Molten Salt

- **Dry-Out**
  - Water Must be Removed Prior to Molten Metal Contact

\[ \text{H}_2\text{O \ bp = 100}^\circ\text{C} \]

\[ 3\text{CaAl}_2\text{O}_4\cdot6\text{H}_2\text{O} \] decomposes 240-370°C

Highly insulating Micro porous board systems like Excelfrax products from Unifrax are water soluble.

These must be protected with a foil lining and interface temp needs to be below the melting point of the foil.
Ceramic Castable Cement Tanks and Piping for Molten Salt

Rate of Temperature Increase for Dry Out

Lining Thickness

- <3 Inches
- 3 to 9 Inches
- 9 to 15 Inches
- > 15 Inches

Temperature (F)

Hour

0 200 400 600 800 1000 1200 1400 1600

25 Deg / Hr
20 Deg / Hr
15 Deg / Hr
10 Deg / Hr

0 20 40 60 80 100 120 140 160
Ceramic Castable Cement Tanks and Piping for Molten Salt

Steel Shell, 1-3 cm thick

Outside Furnace

Estimated Heat Flux Improvement 20%

Freeze Plane Placement

Normal Temperature Profile

Expected Temperature Profile

Al Solidus Temp

Solidification Temperature

Working lining, typically 30-40 cm thick

Inside Furnace ~ 694°C

Normal Position in Backup Insulation

Position with Insulating and Inert Refractory
• Thermal Induced Stress in the Y-Direction
X-ray diffraction patterns obtained from solidified products generated upon exposure to ambient air at 750°C for 50 h of: a) a 32 mol% MgCl₂/68 mol% KCl molten salt, and b) 53 mol% CaCl₂/47 mol% NaCl molten salt.