MACROFLASH BOIL-OFF CALORIMETRY INSTRUMENT FOR THE MEASUREMENT OF HEAT TRANSMISSION THROUGH MATERIALS

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HIGHLIGHTS

• Macroflash: flat plate, comparative boiloff calorimeter providing thermal data for materials from thermal insulation to structural composites to ceramics

• Measurement principle is boiloff calorimetry covering under real-world conditions
  ➢ Steady-state liquid nitrogen vaporization provides a direct calculation of heat flow rate

• Practical way to measure heat transmission through materials under steady-state conditions at below-ambient temperatures and under different compressive loads

• Provides test data at both large $\Delta T$ and/or small $\Delta T$; ranging from 77 K (LN$_2$) to 403 K

• Test specimens may be isotropic or non-isotropic; homogeneous or non-homogeneous
DESIGN OBJECTIVES

• Calibration standard: Cold Boundary Temperature (CBT) of 77 K; Warm Boundary Temperature (WBT) of 293 K
  • Temperature range: from 77 K (LN$_2$) to 403 K

• Directly calculate: heat flow rate (Q)
  • Heat flux (q) and effective thermal conductivity ($k_e$)

• Test specimen dimensions: 76-mm diameter by 6.4-mm thickness (calibration standard)
  • Any thickness from 1 mm to 10 mm can be tested for comparative data

• Specimen material/design type: non-isotropic, layered, asymmetrical, inhomogeneous types are all acceptable; solids or powders; foams, aerogels, wood, metals, ceramics, glass, composites

• Compression loading of test specimen: 0, 2, or 5 psi [standard settings]
GENERATION 2
MACROFLASH TEST INSTRUMENT

Measurement of heat transmission through materials by boiloff calorimetry

J. FESMIRE 7/23/2019
HARDWARE / SOFTWARE

- Macroflash unit
- Weight scale
- Mass flow meter (option to weight scale)
- Heater controller
- Gaseous nitrogen (GN\textsubscript{2}) purge supply
- National Instruments LabView with thermocouple module
- Custom software interface with input prompts, database codes, graphical summary, and final report with all calculations
- 10-liter LN\textsubscript{2} dewar and 350-ml pouring cup
METHODOLOGY

• Principle of heat flow measurement: boiloff calorimetry
  ➢ Flat plate, comparative (ASTM C1774, Annex A4)

• Test specimen: single material or specialized combination of different materials

• Materials: monolithic, composites, blanket, layered blanket, MLI, or bulk-fill type

• Thickness: critical measurement for $k_e$ calculation
  ➢ Fit-up for good thermal contact is also crucial for rigid materials

• LN$_2$ Fill to 220 g (or more) for initial cooldown and stabilization

• Steady-state condition: between 50 g and 100 g LN$_2$
THERMAL CONDUCTIVITY ($\lambda$) AND MEAN TEMPERATURE ($T_m$)

- **Bonus!** Optional intermediate temperature sensors for calculating thermal conductivity ($\lambda$) versus temperature ($T_m$)

- Multiple data points can be obtained from a single test
  - Any two temperatures between 78 K and 403 K can be set up

- **However!** Steady-state heat transmission: heat flows according to the $\Delta T$, not as a function of $T_m$

Interlayer temperature sensors installed for a flat disk test specimen

Macroflash in operation during cooldown
PREPARATION

Test Specimen:
- 76.2-mm by 6.35-mm thick
- Flatness is important
- Material type
- Manufacturer
- Compressive strength (MPa)

Primary Inputs:
- Thickness & diameter (mm)
- Mass (g)
- WBT set point
- Interlayer temperatures (if used)

Test Parameters:
- Loading (0, 2, or 5 psi)
- Thermal greases (yes/no)
- Gaseous nitrogen (GN₂) purge (yes/no)
- Basis by weight scale or flow meter
- Flow meter constants (if used)
- Calibration range (LO, FULL, or HI)
**PROCEDURE**

**Primary Inputs:**
- Start: Cooldown and stabilization with >220 g LN$_2$
- Test Run: Steady-state measurement from >100 g to <50 g LN$_2$
- Repeat 3 times

**Test Parameters:**
- WBT temp control

**Specimen Inputs:**
- Material type
- Manufacturer
- Compressive strength (MPa)
RESULTS

Primary Outputs:
- $k_e$ calibrated
- Mdot (mass flow rate)
- $q$ (heat flow rate)
- $Q$ (heat flux)
- WBT and CBT

Test Parameters:
- Std dev
- Interlayer temps (optional)

Specimen Outputs:
- Structural-Thermal Figure-of-Merit ($F_{ST}$)
- Bulk density
## Reporting of Results

### Test Specimen:
- Test Series No.
- Material Code
- Material
- Test Date
- No. of test runs

### Data Tabulation:
- Mdot
- Q
- q
- $K_{th}$-comp
- Std Dev
- $K_{b}$ (calibrated)
- Comp Strength
- $F_{st}$
- WBT & CBT (293 K & 78 K)

### Example of Macroflash Test Data Summary

<table>
<thead>
<tr>
<th>Test Series</th>
<th>Matl Code</th>
<th>Test Specimen Description</th>
<th>Manufacturer</th>
<th>Test Runs</th>
<th>Density (g/cm³)</th>
<th>Load psi</th>
<th>Grease Y or N</th>
<th>Q W</th>
<th>q W/m²</th>
<th>Std Dev %</th>
<th>$k_v$ Cal (mW/m-K, L, S, H)</th>
<th>Comp. Strength (MPa)</th>
<th>$F_{st}$ (K-m/s-g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Z1-155</td>
<td>A</td>
<td>Ultra Low Density (ULD) Aerogel composite</td>
<td>Aspen Aerogels</td>
<td>7</td>
<td>0.118</td>
<td>0</td>
<td>N</td>
<td>4.651</td>
<td>1020</td>
<td>1.0</td>
<td>18.6 L</td>
<td>1.260</td>
<td>0.1</td>
</tr>
<tr>
<td>Z1-149</td>
<td>A</td>
<td>Cryogel x201 #3 (1 Layer)</td>
<td>Aspen Aerogels</td>
<td>5</td>
<td>0.166</td>
<td>0</td>
<td>N</td>
<td>5.970</td>
<td>1309</td>
<td>0.6</td>
<td>12.4 L</td>
<td>0.8</td>
<td>255</td>
</tr>
<tr>
<td>Z1-118</td>
<td>A</td>
<td>X-aerogel Yellow Disk GRC (ref. Z315)</td>
<td>GRC</td>
<td>4</td>
<td>0.150</td>
<td>5</td>
<td>N</td>
<td>6.760</td>
<td>1482</td>
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<td>25.0 L</td>
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<tr>
<td>Z1-115</td>
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<td>X-aerogel FXP Tan Disk #1 (Flexcon)</td>
<td>Blushift</td>
<td>3</td>
<td>0.148</td>
<td>5</td>
<td>N</td>
<td>7.299</td>
<td>1601</td>
<td>0.5</td>
<td>28.1 L</td>
<td>1.6</td>
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<tr>
<td>Z1-094a</td>
<td>A</td>
<td>Primaloft Thin Aerogel</td>
<td>Primaloft</td>
<td>3</td>
<td>0.247</td>
<td>2</td>
<td>N</td>
<td>4.933</td>
<td>1082</td>
<td>0.3</td>
<td>12.8 L</td>
<td>0.1</td>
<td>157</td>
</tr>
<tr>
<td>Z1-158b</td>
<td>B</td>
<td>Glass Bubbles, nominal tap density</td>
<td>3M</td>
<td>5</td>
<td>0.073</td>
<td>0</td>
<td>N</td>
<td>7.070</td>
<td>1550</td>
<td>0.1</td>
<td>29.7 L</td>
<td>0.1</td>
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<tr>
<td>Z1-021</td>
<td>B</td>
<td>Perlite Powder</td>
<td>5</td>
<td>0.080</td>
<td>0</td>
<td>N</td>
<td>7.622</td>
<td>1671</td>
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<td>799</td>
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<tr>
<td>Z1-039</td>
<td>B</td>
<td>JSC Mars 1A regolith</td>
<td>JSC</td>
<td>4</td>
<td>0.783</td>
<td>2</td>
<td>N</td>
<td>15.411</td>
<td>3379</td>
<td>0.1</td>
<td>125 S</td>
<td>0.1</td>
<td>210</td>
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<tr>
<td>Z1-188a</td>
<td>C</td>
<td>Ultem 2300 #1 (after sanding flat)</td>
<td>Sabic</td>
<td>7</td>
<td>1.507</td>
<td>5</td>
<td>Y</td>
<td>25.591</td>
<td>5612</td>
<td>0.2</td>
<td>225 S</td>
<td>211</td>
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<tr>
<td>Z1-162e</td>
<td>C</td>
<td>G-10 CR composite (cal)</td>
<td>Sabic</td>
<td>7</td>
<td>1.939</td>
<td>5</td>
<td>Y</td>
<td>47.959</td>
<td>10516</td>
<td>0.4</td>
<td>464 S</td>
<td>448</td>
<td>498</td>
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<tr>
<td>Z1-017</td>
<td>C</td>
<td>G-10CR (warp direction)</td>
<td>South Dakota</td>
<td>7</td>
<td>1.929</td>
<td>5</td>
<td>Y</td>
<td>47.850</td>
<td>10492</td>
<td>0.2</td>
<td>624 S</td>
<td>448</td>
<td>372</td>
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<tr>
<td>Z1-130</td>
<td>E</td>
<td>DGEF-BDDS #8 Epoxy Resin</td>
<td>South Dakota</td>
<td>7</td>
<td>1.256</td>
<td>5</td>
<td>Y</td>
<td>20.117</td>
<td>4411</td>
<td>0.8</td>
<td>187 S</td>
<td>105</td>
<td>448</td>
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<tr>
<td>Z1-095</td>
<td>E</td>
<td>Epon862-04 (ref. Z235)</td>
<td>South Dakota</td>
<td>6</td>
<td>1.260</td>
<td>5</td>
<td>Y</td>
<td>22.666</td>
<td>4970</td>
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<td>212 S</td>
<td>105</td>
<td>393</td>
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<tr>
<td>Z1-157a</td>
<td>F</td>
<td>FoamGlass #3</td>
<td>Pittsburgh Corning</td>
<td>6</td>
<td>0.118</td>
<td>5</td>
<td>N</td>
<td>7.455</td>
<td>1635</td>
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<td>32.3 L</td>
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<td>210</td>
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<tr>
<td>Z1-153</td>
<td>F</td>
<td>Polyimide Foam Solimide AC-550</td>
<td>Evonik</td>
<td>5</td>
<td>0.008</td>
<td>0</td>
<td>N</td>
<td>8.119</td>
<td>1780</td>
<td>0.4</td>
<td>36.6 L</td>
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<td>Z1-150a</td>
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<td>SOFI BX-265 #2</td>
<td>Evonik</td>
<td>3</td>
<td>0.037</td>
<td>2</td>
<td>N</td>
<td>5.916</td>
<td>1297</td>
<td>0.2</td>
<td>22.6 L</td>
<td>0.4</td>
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<tr>
<td>Z1-129</td>
<td>F</td>
<td>Rohacell WF-300 stack #3/#4</td>
<td>Evonik</td>
<td>4</td>
<td>0.319</td>
<td>2</td>
<td>N</td>
<td>8.592</td>
<td>1884</td>
<td>0.3</td>
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<td>Z1-125</td>
<td>F</td>
<td>Divinycell Foam H45 #5</td>
<td>Evonik</td>
<td>4</td>
<td>0.051</td>
<td>2</td>
<td>N</td>
<td>6.024</td>
<td>1321</td>
<td>0.1</td>
<td>23.9 L</td>
<td>0.6</td>
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<tr>
<td>Z1-018</td>
<td>F</td>
<td>Certifoam-25 Extruded Polystyrene</td>
<td>Diversifoam</td>
<td>4</td>
<td>0.032</td>
<td>2</td>
<td>N</td>
<td>5.620</td>
<td>1232</td>
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<td>22.0 L</td>
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<td>392</td>
</tr>
<tr>
<td>Z1-177</td>
<td>G</td>
<td>Plain Glass Sample #3</td>
<td>Cabot</td>
<td>7</td>
<td>2.475</td>
<td>5</td>
<td>N</td>
<td>57.696</td>
<td>12652</td>
<td>0.5</td>
<td>872 S</td>
<td>50</td>
<td>23</td>
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<tr>
<td>Z1-160</td>
<td>L</td>
<td>Cabot Corp. Thermal Wrap</td>
<td>Cabot</td>
<td>5</td>
<td>0.074</td>
<td>2</td>
<td>N</td>
<td>5.604</td>
<td>1229</td>
<td>0.2</td>
<td>16.3 L</td>
<td>0.1</td>
<td>157</td>
</tr>
<tr>
<td>Z1-032</td>
<td>L</td>
<td>Reflectix RB3 Single Bubble</td>
<td>Reflectix</td>
<td>5</td>
<td>0.066</td>
<td>0</td>
<td>N</td>
<td>8.026</td>
<td>1760</td>
<td>0.1</td>
<td>33.3 L</td>
<td>0.1</td>
<td>157</td>
</tr>
<tr>
<td>Z1-145</td>
<td>P</td>
<td>Aeroplastic Versify Sample-A 22-stack</td>
<td>KSC</td>
<td>5</td>
<td>0.896</td>
<td>5</td>
<td>N</td>
<td>18.883</td>
<td>4141</td>
<td>0.2</td>
<td>153 S</td>
<td>0.1</td>
<td>157</td>
</tr>
<tr>
<td>Z1-015</td>
<td>P</td>
<td>Ultem PEI 11 (w/ grease)</td>
<td>Sabic</td>
<td>7</td>
<td>1.027</td>
<td>5</td>
<td>Y</td>
<td>18.889</td>
<td>4142</td>
<td>0.4</td>
<td>157 S</td>
<td>151</td>
<td>935</td>
</tr>
<tr>
<td>Z1-002</td>
<td>P</td>
<td>Teflon TFE</td>
<td>DuPont</td>
<td>7</td>
<td>2.120</td>
<td>5</td>
<td>Y</td>
<td>28.375</td>
<td>6222</td>
<td>0.5</td>
<td>253 S</td>
<td>24</td>
<td>45</td>
</tr>
<tr>
<td>Z1-151</td>
<td>W</td>
<td>Balsa (in-plane) 7.3mm</td>
<td>South Dakota</td>
<td>5</td>
<td>0.166</td>
<td>5</td>
<td>N</td>
<td>8.264</td>
<td>1812</td>
<td>0.6</td>
<td>46.0 L</td>
<td>7</td>
<td>916</td>
</tr>
<tr>
<td>Z1-020</td>
<td>W</td>
<td>Balsa Wood (with the grain; warp direction)</td>
<td>4</td>
<td>0.160</td>
<td>5</td>
<td>N</td>
<td>15.548</td>
<td>3409</td>
<td>0.4</td>
<td>133 S</td>
<td>7</td>
<td>328</td>
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<tr>
<td>Z1-014</td>
<td>W</td>
<td>Pine Wood #1</td>
<td>South Dakota</td>
<td>4</td>
<td>0.515</td>
<td>5</td>
<td>N</td>
<td>15.695</td>
<td>3442</td>
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<td>128 S</td>
<td>7</td>
<td>106</td>
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<tr>
<td>Z1-182</td>
<td>X</td>
<td>Ceramic resin post-fire (U Milch)</td>
<td>Formlabs</td>
<td>7</td>
<td>1.412</td>
<td>5</td>
<td>Y</td>
<td>79.335</td>
<td>17397</td>
<td>0.7</td>
<td>370 S</td>
<td>72</td>
<td>138</td>
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</tbody>
</table>
CALCULATION & ANALYSIS

• Total uncertainty in $k_e$ is calculated to be 4.8 % for the Macroflash
• Overall repeatability for most test series is demonstrated to be within 0.5%
• Direct calculation of heat flow rate ($Q$) by boiloff calorimetry

$Q = \text{mdot} \times h_{fg}$

$k_e = \frac{Qx}{A_e T} = \frac{4Qx}{d_e^2 T}$

$q = \frac{Q}{A_e}$

Where:
mdot = mass flow rate (g/s)
h$_{fg}$ = heat of vaporization (J/g)
$k_e$ = effective thermal conductivity (mW/m-K)
x = thickness (mm)
$\Delta T$ = temperature difference
d$_e$ = effective diameter
$A_e$ = effective area
CALIBRATION APPROACH

- Produce and compare thermal conductivity test data of reference materials under similar conditions:
  - With data from Cryostat-100 or Cryostat-500 (absolute instruments)
  - With literature data for similar materials
- Compare with “next best” related cryogenic data (the hard reality, in most cases)
- Connect back to ambient test data (and perform these tests also!)

1) Test Specimen: material, density, size, shape, surface finish, etc.
2) Test Method: type of apparatus, principal of heat flow calculation, steady state or transient, etc.
3) Test Conditions: WBT, CBT, ΔT, calculated $T_{in}$, thermal paste, compression load, gas environment, etc.
4) Test Results: type of thermal conductivity, large ΔT or small ΔT, $T_{in}$, method of calculating thermal conductivity ($\lambda$, $\lambda_e$, k-value, k_p, k_e, etc.)
CALIBRATION

- Macroflash has three ranges of calibration: low end (LO), full range (FULL), and high end (HI)
- The default is FULL but the user can select LO (<50 mW/m-K) or HI (>500 mW/m-K)

LO END Calibration Materials

<table>
<thead>
<tr>
<th>Reference Material</th>
<th>Density</th>
<th>Macroflash Test Data</th>
<th>Absolute Value</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerogel Blanket (Cryogel)</td>
<td>177</td>
<td>comparative-Ke</td>
<td>k_e</td>
<td>1</td>
</tr>
<tr>
<td>Polysio Spray Foam (SOFI BX-265)</td>
<td>36</td>
<td>mW/m-K</td>
<td>39.6</td>
<td>2</td>
</tr>
<tr>
<td>Divinycell Foam H45</td>
<td>50</td>
<td>mW/m-K</td>
<td>40.3</td>
<td>3</td>
</tr>
<tr>
<td>Cellular Glass (in plane)</td>
<td>118</td>
<td>mW/m-K</td>
<td>48.2</td>
<td>4</td>
</tr>
<tr>
<td>Balsa wood (in plane)</td>
<td>166</td>
<td>mW/m-K</td>
<td>60.1</td>
<td>5</td>
</tr>
</tbody>
</table>

CALIBRATION (LO RANGE)

MacroFlash-Z1 Effective Thermal Conductivity ($k_e$) Calibration - Low End

\[ y = 1.0773x + 19.672 \]
\[ R^2 = 0.9992 \]

- For materials with absolute $k_e < 50 \text{ mW/m-K}$
- Calibration Reference Materials:
  - Cryogel aerogel blanket
  - SOFI BX-265 foam
  - Divinycell foam
  - Foamglas
  - Balsa wood

- Macroflash Z1 Data, LOW END, Rev.1
- Linear (Macroflash Z1 Data, LOW END, Rev.1)
CALIBRATION (FULL RANGE)

MacroFlash-Z1 Effective Thermal Conductivity ($k_e$) Calibration - Full Range

$y = 1.6235x - 37.661$

$R^2 = 0.9988$

For materials with absolute $k_e > 50$ mW/m-K

Calibration Reference Materials:
- Cryogel aerogel blanket
- SOFI BX-265 foam
- Pine wood
- PTFE
- G10CR

MacroFlash Data vs. Comparative $k_{e-comp}$ (mW/m-K)

Macroflash Z1 Data, FULL RANGE, Rev.1

Linear (Macroflash Z1 Data, FULL RANGE, Rev.1)
TEST DATA

• Macroflash instruments have tested 500+ material test specimens representing thousands of test runs
  • Materials include composite panels, foams, ceramics, glasses, aerogels, layered composites, hybrid composites, and many others (insulators to conductors)
  • Extensive library and database of both “new” and “standard” materials

• Combined properties: Thermal Conductivity + Density + Strength

Structural-Thermal Figure-of-Merit ($F_{ST}$)

where:

$\sigma$ = compressive strength [MPa]

$k_e$ = effective thermal conductivity [mW/mK]

$\rho$ = bulk density [kg/m$^3$]

$$F_{ST} = \frac{\sigma}{\rho k_e} \times 10^6 \left[ \frac{K \cdot m \cdot s}{g} \right]$$
Thermophysical data for structural-thermal materials used in cryogenic systems

<table>
<thead>
<tr>
<th>Material</th>
<th>$\sigma$</th>
<th>$\rho$</th>
<th>$K_e$</th>
<th>$F_{ST}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>G-10 (transverse direction)</td>
<td>448</td>
<td>1,939</td>
<td>467</td>
<td>495</td>
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<tr>
<td>Ultem® 2300 Glass Filled PEI</td>
<td>221</td>
<td>1,500</td>
<td>212</td>
<td>695</td>
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<tr>
<td>Ultem® 9185 PEI (3-D printed)</td>
<td>100</td>
<td>1,199</td>
<td>145</td>
<td>575</td>
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<tr>
<td>Teflon™ PTFE</td>
<td>24.1</td>
<td>2,120</td>
<td>253</td>
<td>45</td>
</tr>
<tr>
<td>Rohacell® WF-300 PMI foam (14 kPa)</td>
<td>17.8</td>
<td>324</td>
<td>42.1</td>
<td>1,305</td>
</tr>
<tr>
<td>Balsa Wood (transverse direction)</td>
<td>7.0</td>
<td>166</td>
<td>45.9</td>
<td>919</td>
</tr>
<tr>
<td>AeroZero® polyimide aerogel</td>
<td>1.6</td>
<td>150</td>
<td>28.1</td>
<td>380</td>
</tr>
<tr>
<td>Foamglas® Cellular Glass Foam</td>
<td>0.8</td>
<td>118</td>
<td>32.3</td>
<td>210</td>
</tr>
<tr>
<td>Divinycell® H45 PVC Foam (14 kPa)</td>
<td>0.6</td>
<td>50</td>
<td>23.8</td>
<td>504</td>
</tr>
<tr>
<td>Spray Foam Polyiso BX-265 (14 kPa)</td>
<td>0.4</td>
<td>37</td>
<td>22.6</td>
<td>483</td>
</tr>
</tbody>
</table>

$\dagger$ At ambient temperature  *Boundary temperatures 293 K / 78 K; compressive load 5 psi or as noted.
CONCLUSION

- The easy to use Macroflash instrument provides effective thermal conductivity ($k_e$) data for a wide range of materials from thermal insulation to structural composites to ceramics.

- The Macroflash follows the guidelines of standard ASTM C1774 (Annex A4), providing a cost-effective, real-world methodology to test any material for below-ambient temperature applications to moderately elevated temperature conditions.

- Materials include solids, foams, powders, composites, and multi-layered systems and may be isotropic or non-isotropic; homogeneous or non-homogeneous; layered or engineered systems.

- From materials development, to research testing, to quality control in manufacturing, the standardized device provides utility for the fields of energy, transportation, aerospace, construction, medical, and environment.
CONCLUSION

Macroflash boiloff calorimetry instrument for the measurement of heat transmission through materials

- Calibration in work for HI range (>1,000 mW/m·K) materials
- Macroflash enables the testing of complex materials such as layered composites and other highly non-isotropic materials/systems
- Advancements in polymers, ceramics, carbon composites are opportunity for thermal characterization and materials science support
- Additive manufacturing technologies, for both metals and polymers, are opportunity to provide useful thermal data for engineered designs
THANK YOU

for your attention

Questions?

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