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

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Abstract. The Macroflash is a flat plate boiloff calorimeter that provides effective thermal conductivity ($k_e$) data for a wide range of materials from thermal insulation to structural composites to ceramics. The apparatus and method provide a practical, standardized way to measure heat transmission through materials under steady-state conditions at below-ambient temperatures and under different compressive loads. Another unique feature of this device is that it can provide test data at both large and small temperature differences. Using liquid nitrogen as a method to directly measure the heat flow rate, the device is applicable to testing under an ambient pressure environment at a wide range of temperatures, from 77 K to 403 K. Test specimens may be isotropic or non-isotropic; homogeneous or non-homogeneous. The Macroflash is currently calibrated in the range from approximately 10 mW/m-K to 1,000 mW/m-K using well-characterized materials. Reference data for hundreds of test specimens including foams, powders, aerogels, plastics, composites, carbon composites, wood, glass, ceramic, metal, and multi-layered composites have been compiled from Macroflash testing. The Macroflash apparatus is described and its operation, instrumentation, and control system discussed. The calibration approach is detailed as well as analysis of key data sets of standard materials.

1. Introduction

The advent of advanced polymers, composites, and novel material systems along with growing industrial needs in below-ambient temperature applications have brought about the Macroflash development. Accurate thermal performance information, including effective thermal conductivity data, are needed under relevant end-use conditions. The Macroflash is a practical tool for basic testing of common materials or research evaluation of advanced materials/systems.

The Macroflash is a flat plate boiloff calorimeter that provides thermal performance data for a wide range of materials from thermal insulation to structural composites. Boiloff calorimetry is the measurement principle for determining the effective thermal conductivity ($k_e$) and heat flux ($q$) of a test specimen under a wide range of conditions. The Macroflash is a comparative instrument that is calibrated in part from test data compiled using the Cryostat-500 apparatus, an absolute thermal performance instrument that is also for flat-plate specimens [2]. One of the greatest advantages of using liquid nitrogen (LN$_2$) boiloff
calorimetry is its ultimate simplicity and provision of a direct energy measurement. The liquid provides a stable cold boundary temperature and serves as a sort of power meter. The approach also lends itself to testing under representative conditions (i.e., those that reflect the actual-use conditions) afforded by the very large temperature difference established by the liquid nitrogen.

The Macroflash apparatus and method were developed to provide a practical, standardized way to measure heat transmission through materials and systems under steady-state conditions at below-ambient temperatures. A unique feature of this device is that it can test at both small and large temperature differences. Using liquid nitrogen as a method to directly measure the heat flow rate, the device is applicable to testing under an ambient pressure environment at a wide range of temperatures, from 77 K to 403 K. Tests with the Macroflash follow the guidelines of standard ASTM C1774 (Annex A4), providing a cost-effective, field-representative methodology to test any material for below-ambient temperature applications to moderately elevated temperature conditions [1]. Materials include solids, foams, powders, composites, and multi-layered systems and may be isotropic or non-isotropic; homogeneous or non-homogeneous. From engineered 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. The basic characteristics of two different flat-plate boiloff calorimeters are given in table 1.

| Instrument | Type     | Test Specimen Size        | ASTM Test Standard | Environment              | Heat Flux (W/m²) |
|------------|----------|---------------------------|--------------------|--------------------------|-----------------|
| Cryostat-500 | Absolute | 203 mm diameter, up to 40 mm thick | C1774, Annex A3 | Full range vacuum, 77 K–403 K | 0.4–400 |
| Macroflash | Comparative | 76 mm diameter, up to 10 mm thick | C1774, Annex A4 | Ambient pressure, 77 K–403 K | 80–1000 |

2. Test apparatus design and setup

The Macroflash can be used to test solids, foams, or powders and the materials may be isotropic or non-isotropic; homogeneous or non-homogeneous. Test specimens are typically 75-mm in diameter and 6-mm in thickness. The cold boundary temperature (CBT) is maintained by LN₂ at 77 K while a heater assembly maintains a steady-state warm boundary temperature (WBT) from ambient up to 403 K. The steady boiloff of LN₂ provides a direct measure of the rate of heat being transmitted through the test specimen. Nitrogen or other gas is supplied to the instrument to establish a stable, moisture-free, ambient pressure environment. The CBT can be adjusted to any temperature between 77 K and approximately 300 K by the interposition of a known thermal resistance layer between the cold mass and the test specimen or by the use of intermediate temperature sensors.

The Macroflash is currently calibrated in the range from approximately 10 mW/m-K to 1000 mW/m-K using well-characterized materials. Reference data for hundreds of test specimens including foams, powders, aerogels, plastics, composites, carbon composites, wood, glass, ceramic, metal, and multi-layered composites have been compiled. The primary reference instrument for flat-plate boiloff calorimeter testing is Cryostat-500. The technology is built on the prior technology of flat-plate calorimeters developed by the Cryogenics Test Laboratory at NASA Kennedy Space Center (KSC) [3].

The Macroflash houses a cold mass test chamber centered directly over the test specimen, and wrapped by a custom designed multilayer aerogel blanket system to ensure the thermal isolation and stability necessary for accurate steady-state boiloff measurements. Test specimens with thicknesses from approximately 1 mm to 10 mm can be tested. For rigid-type materials, flatness is critical for ensuring good thermal contact on the cold side of the specimen. Compression loading is selected for 0 kPa, 14 kPa, or 34 kPa (0 psi, 2 psi, or 5 psi), with or without thermal grease, and the system can be readily adapted for other loads as required.

The Macroflash includes a heater controller connected to the heater plate assembly for control of the warm boundary temperature (WBT). All instruments are connected to a customized software interface using
a National Instruments LabVIEW® data acquisition system for recording and monitoring. The mass flow rate of the boiloff can be measured by a weight scale or mass flow meter. A simplified schematic for the Macroflash instrument is given in figure 1.

![Macroflash schematic](image-url)

**Figure 1.** Macroflash: simplified schematic (not to scale). The cold mass assembly is 76-mm diameter by 250-mm tall. A test specimen is 76-mm diameter by 6-mm in thickness.

### 3. Testing methodology

The test measurement principle is LN₂ boiloff calorimetry where the mass flow rate of nitrogen gas is directly related to the rate of heat energy transmitted through the material [4]. The test conditions are representative of actual-use cryogenic applications; unless otherwise specified, standard is a WBT of 293 K and a CBT of 78 K.

All tests are performed at an ambient pressure gaseous nitrogen (GN₂) condition for consistency. Under steady-state flow conditions, the rate of heat flow through the test specimen is constant at all points through the thickness of the specimen. The boiloff mass flow rate, measured by a weight scale or mass flow meter, allows direct calculation of heat flux and effective thermal conductivity as given in equations 1-3.

\[
Q = \dot{m} h_{fg}
\]

\[
q = \frac{Q}{A_e}
\]

\[
k_e = \frac{q x}{(A_e \Delta T)}
\]

The heat flow rate (Q) is the product of boiloff mass flow rate (\(\dot{m}\)) and enthalpy of vaporization of liquid nitrogen (\(h_{fg}\)). Knowing the boundary temperatures on either side of the specimen in a set test environment, the heat flux (q) can be calculated. Knowing the specimen's effective heat transfer area (\(A_e\)), as well as the thickness of the specimen (x) and the temperature difference (\(\Delta T\)), the effective thermal conductivity (\(k_e\)) is easily calculated.
Based on using a mass flow meter with a full-scale accuracy of 1%, an uncertainty of approximately 5% has been calculated for the flat plate boiloff calorimeter apparatus [2]. For the basic weight scale used (Ohaus Explorer EX10201 with 10.2 kg capacity and 0.1 g readability) the accuracy is comparable. The condensed water vapor and ice is handled by design and procedure. The design of the unit with a series of insulated vent pathways including a top insulation cap allows the boiloff vapor to warm to near ambient temperature to minimize the condensate and frost. The standard procedure includes a topping off to 220 g or more of LN₂ and a cold soak phase from this level down to 100 g where the test measurement phase begins. The test phase is then repeated a minimum of three times to verify consistent results. The methodology provides a repeatability of <1%. The precision and bias for a given test are pending on future round-robin testing of identical test specimens at different laboratories.

The software interface includes test specimen preparations and test condition entries, and automated data analysis, calculations, and reporting. Primary inputs include thickness & diameter (mm), mass (g), WBT set point (K), and interlayer temperatures (if used). Test parameters include compression loading (psi), thermal greases (yes/no), GN₂ purge (yes/no), basis by weight scale or flow meter, and calibration range (LO, FULL, or HI). Primary outputs include kₑ calibrated, \(\dot{m}\) (g/s), Q (W), q (W/m²), standard deviation (%), WBT (K), and CBT (K). Specimen outputs include the Structural-Thermal Figure-of-Merit (F₅₇) and bulk density (kg/m³).

Boiloff calorimetry provides the ability to test both simple uniform materials and complicated non-homogeneous, anisotropic, composite materials with equal ease [5]. The extent of the temperature difference is dependent on the cryogenic fluid and the heat source used, providing high sensitivity for the accurate measurement of highly thermally insulating materials or structural materials alike. In addition, optional intermediate temperature sensors can be used for calculating thermal conductivity (\(λ\)) as a function of mean temperature (\(Tₘ\)) over the wide range of temperatures provided in a single test [6].

Different materials and varied test objectives require an appropriate combination of apparatus and method. The type, thickness, density, flatness, compliance, and strength of the material are important considerations. The test specimens (examples shown in figure 2) are typically 76-mm diameter by 6.35-mm thickness and should be flat and smooth-faced or easily compressible to insure good thermal contact between the heater assembly and the cold mass.

**Figure 2.** Macroflash test specimens (76-mm diameter by 6.4-mm thickness, typical).

4. **Calibration and range of measurement**

Initial measurement and calculation of comparative kₑ values are calibrated to give the kₑ values to be reported. The calibration is based on a linear fit of measured material thermal conductivities to standard reference data for commercial materials with known properties. One challenge is the manufacturers’ reported values for thermal conductivity are not usually for the boundary temperatures of 293 K and 78 K. Most typically, these data are taken at ambient temperature (around 293 K) with only a small temperature difference imposed by the commercial test instruments generally used. The standard reference data therefore have to be derived by careful research and certain selection from the available peer-reviewed technical literature of the world over many decades [9-11]. Some of these reference data are taken from the absolute cryostat test instruments, Cryostat-100 and Cryostat-500, of the Cryogenics Test Laboratory at NASA/KSC [12-15].
Standard methods of test specimen preparation, testing, thermal performance calculations, data recording and data reporting are all essential. The key parameters include density, compression, specimen thickness, diameter, and whether or not thermal contact grease is used. For standard reference work, many factors must match up including test specimen type, method, condition, etc., as listed below:

1. Test specimen: material, density, size, shape, surface finish
2. Test method: type of apparatus, principle of heat flow calculation, steady-state or transient
3. Test conditions: WBT, CBT, ∆T, calculated Tm, thermal grease, compression load, environment
4. Test results: type of thermal conductivity (λ, ke, or other), large ∆T or small ∆t, use of Tm, and method of calculation

More than 10 years of research and testing of hundreds of test specimens has provided a library of data and a foundation for calibration. Three ranges of calibration are listed: LO (ke < 50 mW/m-K), FULL (ke > 50 mW/m-K) and HI (ke > 500 mW/m-K). The full range curve fit is the default and works well except for materials with a ke < 30 mW/m-K. The high range curve is a current work in progress.

The reference materials for the calibration curves are found in the literature as discussed. As the Macroflash is a comparative instrument, testing of known materials with known thermal conductivity data under the same conditions is the process of calibration. The known thermal conductivity data under like conditions must come from an absolute instrument, making these data rare and unavailable but for a few materials. The materials used for the preliminary Macroflash calibration are listed in table 2. The compression loading and use of grease (or not) are selected to match, as closely as possible, the manner in which the materials were tested in the literature data. The low-end calibration curve is given in figure 3 while the full range calibration is presented in figure 4. The baseline test conditions for calibration remain as follows: 293 K and 78 K boundary temperatures; ambient pressure gaseous nitrogen environment.

Table 2. Reference materials used in Macroflash calibration.

| Range         | Compression | Grease | Density* | Note                                      |
|---------------|-------------|--------|----------|-------------------------------------------|
| Low-end       |             |        |          |                                           |
| Cryogel®      | 0           | no     | 177      | Aerogel composite blanket [13]            |
| SOFI BX-265   | 2           | no     | 36       | Polyisocyanurate spray-on foam insulation (SOFI) [14] |
| Divinycell® H-45 | 2     | no     | 50       | Rigid foam insulation [15]                |
| Foamglas®     | 5           | no     | 118      | Cellular glass [11]                       |
| Balsa wood    | 5           | no     | 166      | In-plane [9]                              |
| Full-range    |             |        |          |                                           |
| CryogelTM      | 0           | no     | 177      | Aerogel composite blanket                 |
| SOFI BX-265   | 2           | no     | 36       | Polyisocyanurate spray-on foam insulation (SOFI) [14] |
| Pine wood     | 5           | no     | 515      | White pine (in plane) [10]                |
| PTFE          | 5           | yes    | 2,120    | Polytetrafluoroethylene [9]               |
| G10-CR        | 5           | yes    | 1,939    | Glass fiber reinforced composite (in plane) [9] |

† Note: 1 psi = 6.895 kPa  *
As tested
Figure 3. Macroflash calibration curve for effective thermal conductivity: low range (LO).

Figure 4. Macroflash calibration curve for effective thermal conductivity: full range (FULL).
5. Example test results and discussion

The test specimens cover a wide variety of materials from thermal insulators, composites, building materials, glasses, ceramics, and novel experimental materials such as syntactic micro-balloon nanocomposites [16-17]. Combining values for thermal, mechanical, and density properties allows for a rapidly obtained, quantitative screening parameter to identify potentially high performing structural thermal insulation candidates. Thermophysical data for a range of different structural-thermal materials used in cryogenic systems data are given in table 3. Also included are G10 composite, Teflon™, balsa wood, and polyisocyanurate spray foam for general reference and connection to the absolute data from literature [9, 11]. These $k_e$ data were produced using a Macroflash, per ASTM C1774 Annex A4, for boundary temperatures of 78 K (CBT) and 293 K (WBT) and under a compressive load of 34 kPa. The structural-thermal figure-of-merit ($F_{ST}$) is calculated as in equation (4).

$$F_{ST} = \frac{\sigma}{\rho k_e} \times 10^6 \left[ \frac{K \cdot m \cdot s}{g} \right]$$

(4)

Where $\rho$ is the bulk density in kg/m$^3$, $k_e$ is the effective thermal conductivity in mW/m-K (with the prescribed CBT and WBT), and $\sigma$ is the compressive strength in MPa (at ambient temperature).

| Material                                      | $^\dagger$σ MPa | $^*$ρ kg/m$^3$ | $^*$k$e$ mW/m-K | $F_{ST}$ K-m-s/g |
|----------------------------------------------|-----------------|----------------|----------------|-----------------|
| G-10 (transverse direction)                  | 448             | 1,939          | 467            | 495             |
| Ultem® 2300 Glass Filled PEI                 | 221             | 1500           | 212            | 695             |
| Teflon™ PTFE                                 | 24.1            | 2,120          | 253            | 45              |
| Rohacell® WF-300 PMI Foam (2 psi)             | 17.8            | 324            | 42.1           | 1,305           |
| Balsa Wood (transverse direction)             | 7.0             | 166            | 45.9           | 919             |
| AeroZero® polyimide aerogel                  | 1.6             | 150            | 28.1           | 380             |
| Foamglas® Cellular Glass Foam                | 0.8             | 118            | 32.3           | 210             |
| Divinycell® H45 PVC Foam (2 psi)              | 0.6             | 50             | 23.8           | 504             |
| Spray Foam Polysio BX-265 (2 psi)            | 0.4             | 37             | 22.6           | 483             |

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

6. Conclusion

The Macroflash instrument, an easy-to-use flat-plate boiloff calorimeter, has been developed to provide effective thermal conductivity ($k_e$) data for a wide range of materials from thermal insulation to structural composites. Following the guidelines of standard ASTM C1774 (Annex A4), the Macroflash provides a cost-effective, field-representative methodology to test any material for below-ambient temperature applications to moderately elevated temperature conditions. Non-isotropic, layered, asymmetrical, or inhomogeneous types of materials are all acceptable; solids or powders can be tested; and foams, aerogels, wood, metals, ceramics, glass, or composites are readily tested. From engineered materials development, to research testing, to quality control in manufacturing, this standardized device provides utility for the fields of energy, transportation, aerospace, construction, medical, and environment.

7. References

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