CALORIMETRIC MEASUREMENTS OF
LASER ENERGY AND POWER –
1975 SUPPLEMENT

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Abstract

The use of calorimeters for measuring laser output energies and powers is reviewed, primarily for the period 1972-1975 since preparation of an earlier review.

Introduction

References 1-113 of this report were discussed in an earlier review. The present review covers primarily papers that have been published since. It will also include some earlier papers that were unavailable or were overlooked at that time.

Due to space limitations, the published version of the earlier review included less general discussion of the principles of calorimetry than did the preliminary version. It did, however, include several additional references; three of these are to books and a review article on calorimetric principles, and seven are to articles specifically on laser calorimetry. Discussion of these latter seven is repeated in the sections below. Seven references in the earlier version were eliminated in the process of abridgment to the later form.

Some papers are not readily available or are unavailable in English, French, or German translation; in such cases, and some others, the abstract is cited with the reference. In many of these cases the abstract provides little information about the apparatus.

Calorimeter Design

DISK CALORIMETERS

Jacob et al. described twin calorimeters consisting of two anodized aluminum plates, 3.2 mm thick. A differential thermopile having junctions spaced 25.4 mm apart in a square grid pattern on the back of each plate senses the temperature difference. The
sensitivity was calculated from the mass and specific heat of the aluminum and the characteristics of the thermopile materials, and was also checked by comparison with a commercial power meter. The laser beam impinges directly on the aluminum oxide surface; the specular reflectivity of this was measured and found to vary from 1-3% in the 8.5-11-μm region. The maximum acceptable energy density was calculated for short pulses as a function of pulse duration, the assumed limit being that which would give a temperature of 933 K (660°C) (melting point of aluminum) at the rear of the 2.5-μm aluminum oxide layer; this is about 3 J·cm⁻² for 1-μs pulses.

Geist, Schmidt, and Case described a broadband twin radiometer having a receiving cavity approximately as shown in Fig. 1. The inside of the cavity is coated with black paint, so most of the radiation is absorbed on the low, nearly flat cone at the end of the cylindrical cavity; the cylindrical wall absorbs most of the radiation reflected at the first surface. The system thus approximates most closely a disk calorimeter, although it has some relation to the cone and hollow-sphere types. The base of the cavity includes a calibrating heater, and the temperature is sensed with an electrodeposited radial thermopile. The instrument was compared with the NBS C-series laser calorimeters using a 0.676-μm cw laser beam; agreement was within the experimental uncertainty, ~1.5%.

West et al. described a related configuration derived from the earlier cone-and-tube instruments. One version, Fig. 2, has an absorbing disk mounted at an angle to the beam and attached to a tube; both are black-painted. Mounting the disk at an angle to the beam ensures that any specular reflections from the metal, at holes in the paint, would strike the tube. Three of these and one of the older type are maintained at the NBS as reference standards for calibration of other instruments. Another version, also related to that of Neill, has a shallow polished conical mirror at the end of the cylinder to reflect and spread the incident radiation onto the cylindrical wall, Fig. 3. Both of these versions use a double-walled calorimetric body with a copper ring connecting the two walls. This construction serves to reduce systematic errors in comparing electrical heating calibrations and laser beam inputs because the surface temperature pattern on the outer wall becomes largely independent of the location of the source of the heat on the inner wall.

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Fig. 1. Cup-shaped calorimeter.127
Nowicki\textsuperscript{130} developed a blackened-disk calorimeter utilizing the temperature coefficient of the permittivity of a ferroelectric ceramic for the temperature sensing. Two plates of BaTiO$_3$ are sandwiched between two metal capacitor plates, one of which is blackened. The capacitor is connected into an ac bridge, the output of which is used to regulate the current in a heater between the two ceramic plates so as to maintain the capacitor at a constant elevated temperature. The reduction of the power required in the heater when radiation impinges on the blackened plate is assumed to be equal to the radiation power.

Sakurai, Mitsuhashi, and Honda\textsuperscript{131} described apparently the same calorimeter as in an earlier paper.\textsuperscript{33} Zakurenko et al.\textsuperscript{125} described a somewhat similar disk calorimeter with automatically controlled Peltier cooling, using a semiconductor thermopile.

Gardener and Harting\textsuperscript{121} described a disk calorimeter, for beams of fast atoms, using an arrangement with a radial thermopile and radial...
heat flow somewhat similar to those of Jennings et al.\textsuperscript{1} and Mefferd et al.\textsuperscript{35}

CONE CALORIMETERS

Preston\textsuperscript{132} studied the spectral response of cone calorimeters by comparison with a black radiation thermopile, using filtered bands of broadband radiation peaked at 0.65, 0.85, and 1.2 \( \mu \text{m} \). The results for the absorption efficiency of the cone were in good agreement with those calculated on the assumptions that the reflections are perfectly specular and that the effective average reflectivity of nickel for rays incident at 12 angles from 7.5° to 90° is equal to that for normal incidence.

Boutinou and Sauneuf\textsuperscript{133} described twin graphite cone calorimeters. The junctions of the iron-constantan thermopile are attached at equal intervals along a helix on the cone or at the center of segments of equal area. Calibration is performed by discharging a capacitor across electrodes around the opening and at the apex of the cone. The response to laser pulses was found to be linear for energy densities up to 0.5 J \( \cdot \text{cm}^{-2} \) for pulses 30 ns wide, and less for shorter pulses. The response as a function of beam diameter was studied by placing a diverging lens in the beam and changing the position of the calorimeter. From comparisons of similar calorimeters in a split-beam system it was concluded that the precision is limited to ~20\%, and the design is unsatisfactory for diameters greater than 50 mm.

A brief preliminary description was given of calorimeters of larger diameter using a flat graphite disk with deep circular grooves giving some cone effect.

Neill\textsuperscript{123} described a calorimeter consisting of an aluminum cylinder blackened on the interior and containing a polished aluminum cone having a base diameter equal to that of the cylinder. Reflection from the exterior surface of the cone reduced the flux intensity at the interior of the cylinder below the threshold for damage to the blackened surface.

Stricker and Rom\textsuperscript{124} described a calorimeter having a curved glass cone in which a thin spiral strip of platinum on the interior functioned as a resistance thermometer; the response time was a few milliseconds.

HOLLOW-SPHERE CALORIMETERS

Smith et al.\textsuperscript{134} described a large variation of a hollow-sphere calorimeter for powers from 0.3-100 kW at wavelengths of 1-11 \( \mu \text{m} \). The aperture diameter is 10 cm. The beam first strikes a cylindrical convex mirror and then a sandblasted plate, to diffuse the beam and reduce the power density. The remainder of the cavity wall consists of black-painted panels. Water is pumped through channels in
the wall and the temperature rise is measured for 100-s irradiation periods. Electrical heaters in the water stream are used for calibration.

Zeiders and Hella described a hollow-sphere calorimeter for high-power beams. The beam enters through a relatively large aperture and strikes a curved mirror of cylindrical symmetry designed to distribute the energy uniformly throughout the black-painted interior of the sphere. The temperature rise is measured by resistance thermometry, using wire wound into a groove in the wall. Calibration by the method of mixtures is suggested: swirling a known amount of hot water in the sphere and measuring the temperature decrease of the water and resistance increase of the wire. This method would be quick and easy, and adequate for moderate accuracy.

Dombi et al. described a calorimeter consisting of a 10-mm cube of thin gold foil with a 2-mm hole, equivalent to a hollow sphere. Büchi and Pfeiffer described a hollow-sphere calorimeter with distributed thermistors for the temperature measurement.

BOLOMETER CALORIMETERS

Koren et al. described a bolometer for laser pulses consisting of a thin black film of V$_2$O$_5$ evaporated onto a 10-mm-diam sapphire plate 8 µm thick. The resistance change is measured with an oscilloscope. Peak resistance change occurs in a few hundred microseconds, and the time constant for cooling is about 5 ms. The noise level for small pulses is about ±3 µJ.

Wolinski and Badziak and Muzii described other bolometric devices for laser energy measurements. Mitsuhashi and Sakurai reviewed laser calorimetry and developed a "twisted-double-wire" calorimeter - possibly a bolometer type - for pulsed ruby laser radiation. Kortum and Mueller reviewed recent literature on bolometers and thermoelectric detectors, for laser and other measurements.

Lushchikov et al. described a power meter for infrared lasers in which the laser beam is attenuated, chopped, and compared with the chopped beam from a black-body source, using a bolometer as the detector.

Farmer described a commercial version of the double cone bolometer, similar to that of Schmidt and Greenhow and Schmidt.

VOLUME-ABSORPTION CALORIMETERS

Watt described calorimeters for short, high-intensity, 1.06-µm pulses. The beam enters a cylindrical cavity through a coaxial entry tube. The cavity is coated with a diffusing white paint. Suspended near the front of the cavity is a ring of heat-absorbing glass whose inside diameter is greater than that of the entry tube; thus
it is not struck directly by the incident beam but eventually absorbs most of the radiation scattered about in the cavity. The hot junctions of a thermopile are cemented to the ring. The calorimeter contains no electrical heater, but is calibrated by a split-beam comparison with a cone calorimeter using short intervals (0.1-1 s) of a cw beam.

Boulanger et al.\textsuperscript{145} described a twin volume-absorbing calorimeter for 10.6-\textmu m beams. The absorbing material is Plexiglas or Makrolon, 0.5 or 1 mm thick, cemented to copper plates. An iron-constantan thermopile is used to measure the temperature difference between the working and reference plates. Calibration is performed by calculation from the masses and specific heats of the components or by use of an electrical heater on the copper plate.

Gunn\textsuperscript{146} described twin calorimeters built in diameters from 13-330 mm. Two anodized aluminum disks are suspended in a massive aluminum housing; on each are uniformly distributed 12 or 21 junctions of a chromel-constantan wire thermopile. Calibrating heaters are located on the rims of the disks. The absorbing material is cemented to the face of the disk; various colored glasses were tested at 1.06 \textmu m\textsuperscript{146} and 0.46-0.69 \textmu m.\textsuperscript{147} Three different glasses withstood energy densities up to about 60 J·cm\textsuperscript{-2} for 200-ps, 1.06-\textmu m pulses. The noise level of the calorimeters is typically 10 \textmu J per square centimeter of absorber area. A more economically constructed version using commercial semiconductor thermoelectric modules has also been described.\textsuperscript{148}

Calorimeters of the same type but with different absorbing materials — magnesium oxide, lithium fluoride, and various plastics — were also studied for use from 9.24-10.76 \textmu m with both cw and short pulse beams.\textsuperscript{98} Stirred-liquid calorimeters were also built and used with copper sulfate solution\textsuperscript{146} for 1.06-\textmu m beams and various organic liquids for 9.27-10.59 \textmu m.\textsuperscript{98} The solid-absorber calorimeters were also compared with a previously-described tubular calorimeter\textsuperscript{70} using short intervals of cw beams at 1.06 \textmu m\textsuperscript{147} and 9.27-10.59 \textmu m.\textsuperscript{98}

Karlova and Kuz'min\textsuperscript{149} briefly described a calorimeter using magnesium fluoride as a volume absorber for high-power 10-\textmu m pulses.

Johnson\textsuperscript{150} and Reichelt et al.\textsuperscript{151} described energy meters for 10.6-\textmu m pulses in which the energy is totally absorbed in a gas, sulfur hexafluoride. Offenberger et al.\textsuperscript{152} described another in which pulsed beams are partially absorbed in a dilute mixture of propylene and helium. In all of these the pressure increase rather than the temperature increase is measured. The type is capable of ready extension to large beam diameters and large pulse energies. Related to these energy meters is the use of various
absorbing gases as attenuators for 10-µm beams.¹⁵³

Briones¹¹⁹ described calorimeters that operate by measuring the volume or pressure increase of an absorbing liquid.

PARTIAL-ABSORPTION CALORIMETERS

Valitov and Kalinin¹⁵⁴ described a power-measuring calorimeter using two mirrors; this may be similar to that of Rasmussen.⁹⁹

Reflectance and Absorptance Measurements

Calorimetric methods may be applied to the determination of the reflection and reradiation losses from materials used as absorbers in laser calorimeters. Jacob et al.¹²⁶ mounted a specimen of anodized aluminum absorber above a disk calorimeter and surrounded the specimen with a hemispherical mirror to direct radiation reflected from the specimen onto the disk. A small hole in the mirror admitted a laser beam to the specimen. The system gave an upper limit of 5% for the reflectance at 10.6 µm.

Boulanger et al.¹⁴⁵ measured the reflection loss from their plastic-plate calorimeters by tilting the calorimeter at an angle of 5° to the incident beam and placing a mirror so as to return the specularly-reflected component to the calorimeter absorber.

Gunn¹⁴⁶ described a reflectance calorimeter consisting of a blackened metal cup, fitted with a thermopile and calibrating heater, arranged to intercept nearly all of the specularly-reflected and diffusely-reflected radiation as well as excess thermal radiation from the heated surface of specimens of absorbing material. A hole in the cup admits the laser beam at an angle of 15° to the specimen, which is mounted on an aluminum disk fitted with its own thermopile and calibrating heater to measure the absorbed energy. Separate measurements of the specular reflectance were also described.

West and Schmidt¹²⁸ have described in greater detail a previously-described¹³ procedure for using an auxiliary calorimeter to measure the losses from blackened tube-cone and tube-disk calorimeters.

Calorimetric methods have also been used for measuring the absorptivity of mirror materials.⁴¹,¹⁵⁶-¹⁶⁷

Nadezhkin et al.¹⁵⁵ described an instrument for measurement of the energy of high-power pulses in which the radiation is received on a low-absorption glass plate; this may be similar to the calorimeter of Edwards.¹¹

The gas-absorption energy meter of Offenberger et al.¹⁵² (preceeding section) is also intended to absorb only a fraction of the energy in a transmitted beam.
Test and Comparison Procedures

Many papers have dealt with electrical calibration of calorimeters and with techniques for comparing calorimeters and other power- and energy-measuring instruments in laser beams; a few key points will be mentioned here. In general, a split-beam arrangement must be used; a single-beam system is usable only if the laser stability is better than the precision required of the comparison. The two calorimeters under comparison may be interchanged between the two beams, or they may be interchanged in one beam while a third calorimeter is used as a monitor in the other beam. The calorimeters should be far enough from the laser to reduce the effect of laser-flashlamp radiation to an acceptable level. Irises and entry tubes may be used to reduce the effects of variable extraneous radiation and air currents. The splitter and any windows in the system should be wedges, in order to avoid interference effects from parallel surfaces. The angle of incidence of the beam at the splitter should be small if the angle of polarization of the laser beam varies. A lens may be used in one or both beams and the position of the calorimeter varied to test the effects of variation of power-energy density. Irises and central stops may be used to ensure that the beam lies well within the aperture of the calorimeter; diffraction effects must be taken into account.

O'Neil et al. have discussed samplers, attenuators, and methodology for diagnostics of high energy pulsed CO₂ lasers. Franzen studied Fresnel beam splitters for the same.

Miscellany

Zakurenko et al. used an ice calorimeter for laser energy measurements. In this system, the energy received by the calorimeter causes melting of ice, which is measured as a volume decrease in an ice-water mixture. The abstract does not indicate the type of receiver used within the calorimeter to absorb the laser energy; but the virtues of ice calorimeters include ability to measure slow processes and lack of dependence of the response on the location of the deposition of heat, so an absorbing liquid, such as an aqueous solution, or a conical or other cavity could be used.

Several papers have dealt with pyroelectric detectors as applied to laser and other measurements.

Blevin and Brown in a paper on determination of the Stefan-Boltzmann constant include some useful information on black coatings.
Von Gutfeld\textsuperscript{182} discovered a transverse thermoelectric voltage effect in evaporated molybdenum and tungsten films irradiated by short laser pulses; the phenomenon may be useful for detectors.

Several earlier papers have included significant amounts of review material on calorimetric and other methods of laser power and energy measurement.\textsuperscript{23,25,27,28,183-185} Three papers\textsuperscript{1,37,58} referenced earlier\textsuperscript{114,115} have been reprinted.\textsuperscript{186}

The designation of laser calorimeters as disk, cone, hollow-sphere, bolometer, volume-absorption and partial-absorption types is rather arbitrary; there is considerable overlap, and many instruments do not fit cleanly into any category. Table 1 presents a summary of some of the more important characteristics of most of the laser calorimeters described in the references. The following numbered paragraphs define the abbreviations used in the corresponding columns in Table 1.

1. Perhaps the most fundamental division is between calorimeters that absorb radiation at a surface (sur) and those that absorb it throughout a volume of material (vol).

2. In the case of surface absorption, the absorbing surface may be a bright metal (met) or may be blackened with some coating such as black paint, anodizing on aluminum, or enamel on copper wire (blk). The (blk) absorptivity is usually higher but the damage threshold lower than in the (met). In the case of volume absorption, the absorber may be a solid (sol), liquid (liq), or gas (gas).

3. In the case of surface absorption, the radiation may strike the surface perpendicularly, or nearly so, with reflected energy being lost, as in a disk calorimeter (dia). Various configurations may be used to increase the absorptance. These include the cone (con) and hollow sphere (hol). A grooved disk (grv) gives some of the effect of a cone. A cup (cup) absorbs the radiation mainly on the blackened bottom, as in a disk calorimeter, but intercepts some of the scattered radiation on the cylindrical wall. An inverted cone or convex mirror (inv) of polished metal within the calorimeter may be used to spread high-intensity radiation onto a blackened cylindrical or spherical wall. A bolometer of randomly packed insulated wire, a "rat's nest" (rat), gives multiple reflections within the mass of wire. Disks, cones, or hollow spheres may be constructed of randomly or regularly arranged wire that serves both as the absorber and the thermometer or bolometer; the indentations of the resulting surface increase its absorptivity.

4. The calorimeter may be designed for total or near-total absorption of its laser beam (tot) or for partial absorption (par) with
most of the beam continuing on, available for other uses.

5. The measurement may be of the power \( \text{pow} \) of a cw beam, or of the energy \( \text{en} \) of one or more pulses or of a timed interval of a cw beam.

6. The calorimetric methodology may be isoperibolic \( \text{iso} \) in which the temperature rise of the calorimetric body is measured, with some correction for heat lost during the rise. The steady-state conduction method \( \text{sst} \) is most often used for power measurements but may also be used for energy measurements by integrating the temperature-time curve. A particular case of the steady-state method is the liquid flow \( \text{flo} \) method. In another isothermal method energy is measured by observing the amount of phase change \( \text{pha} \), solid to liquid or liquid to gas, that it causes.

7. The temperature measurement may employ a thermocouple \( \text{tc} \), multi-junction thermopile \( \text{tp} \), thermistor \( \text{tst} \), or metallic resistance thermometer \( \text{res} \). A particular case of the latter is the bolometer \( \text{bol} \). Or temperature change may be inferred by measurement of a change of volume \( \text{vch} \) or change of pressure \( \text{pch} \).

8. The calorimeter may incorporate a heater \( \text{htr} \) for calibration with electrical energy, or have no heater \( \text{noh} \). With appropriate circuitry, the resistor of any bolometer-type calorimeter can be made to serve as its own heater; it is sometimes unclear whether this has in fact been done.
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