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THERMAL CONDUCTIVITY MEASUREMENT OF
FUSED SILICA GLASS

by

D. Goldberg, R. P. Von Herzen
and J. Sclater

August 1980

TECHNICAL REPORT

*Supported by the National Science Foundation
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Department of Geology & Geophysics

ABSTRACT

The reproducibility of thermal conductivity measurements on fused silica glass by various investigators using different techniques suggests its suitability as a standard for such measurements. Our laboratory measurements with the needle probe technique on chip samples of silica glass saturated with water gave a value of 3.287 ± 0.154 (S.D.) mcal/cm s °C ($n = 21$) at 25 °C, which is within about 1% of the previously determined values and the value given by the manufacturer for this material. The good agreement indicates that the flat-plate steady-state and needle probe transient methods give the same value for this material, and that the water-saturated chip technique is an accurate method to measure thermal conductivity of isotropic samples.

INTRODUCTION

The comparison and calibration of different needle probes by measurement of a consistent conductivity standard provided the motivation for this study. The needle probe method described by Von Herzen and Maxwell (1959) is a transient technique for measuring the thermal conductivity of sediments. The technique approximates an infinite line heat source which creates temperature change proportional to the logarithm of time in the surrounding sediment medium. The heat source is generated through a single resistance loop of known characteristics and is placed in a hollow needle with a thermistor to measure temperature at the middle of its length.

When a constant current through the wire applies heat to the sample, the monitored thermistor gives the change in temperature as a function of time theoretically expressed as (Jaeger, 1958):

$$T(t) = \frac{q}{4\pi K} \ln (4\alpha t / Ba^2) \quad (\text{Eq. 1})$$

where

$T(t)$ = temperature as a function of time

t = time

q = heat per unit length per unit time

K = thermal conductivity of the sample

α = thermal diffusivity of the sample

a = probe radius

$B = 1.7811$

Jaeger's equation solved for K over a distinct time interval $(t_2 - t_1)$ after the heat has been applied gives:

$$K = \frac{q}{4\pi(\Delta T)} \ln \frac{t_2}{t_1} \quad (\text{Eq. 2})$$

where

K = thermal conductivity

ΔT = change in temperature over the time interval $(t_2 - t_1)$

q = heat input

$$= \frac{(\text{heater voltage})^2 (0.2389)(2)}{(\text{heater resistance})(\text{heater length})}$$

note: the factor of 2 in the numerator results from the two lengths of resistance wire in the needle as a result of its loop construction.

After the heat has been applied for about 10 seconds, the temperature/time relation approaches a logarithmic asymptote which is valid for two (2) minutes, when the radial boundaries of the 2 cm diameter sample vials begin to affect the temperature. The mean slope of the plotted temperatures versus logarithm of time allows the thermal conductivity, K , to be calculated using the constants from equation 2.

Thermal conductivity of a hard-rock sample can be measured using the needle probe by taking measurements on fragments of that sample (Horai and Baldrige, 1972). The sample is pulverized and saturated with water, packed in a cylindrical container, and the needle probe is inserted into the mixture along the axis. The conductivity of the saturated mixture is modelled as packed spheres of rock in a water matrix, and is determined by the conductivity of its solid and fluid components and the porosity of the mixture (Woodside and Messmer, 1961):

$$K_m = K_r (1-\phi) + K_w \phi \quad (\text{Eq. 3})$$

where,

K_m = thermal conductivity of the mixture of
saturated rock chips and water

K_r = thermal conductivity of the solid rock

K_w = thermal conductivity of water

ϕ = porosity or volume fraction of water in the
mixture

Woodside and Messmer's relation solved for K_r gives:

$$K_r = (K_m K_w^{-\phi})^{1/(1-\phi)} \quad (\text{Eq. 4})$$

This formula enables the conductivity of a solid rock to be calculated from the porosity, the known conductivity of water (Clark, 1966), and a measured value of the conductivity of its water saturated fragments.

PROCEDURE

The application of the needle probe technique on a fused silica glass standard, chosen for its consistent physical properties, requires a pulverized, saturated sample whose conductivity is reduced to zero porosity by Woodside and Messmer's relation.

The glass sample is crushed with a mortar and pestle into a powder having a 0.5 mm maximum grain diameter. The sample is divided into standard 2 cm diameter plastic vials which are weighed. The powder is saturated with water in a moderate vacuum (20 torr) and agitated to eliminate air bubbles, and any excess water is decanted off of the saturated sample. The sample is weighed again. The difference between the dry and saturated weights gives the volume of interstitial water.

The weight of the dry sample divided by its density gives its volume. If the density of the sample is not known, as is the usual case for sediment samples, the volume of the

saturated sample can be calculated by weighing a volume of water equal to the volume of the saturated sample and subtracting the volume of interstitial water. In appendix B, this method is used to check the given density of the standard and to estimate error.

The porosity, ϕ , of the saturated sample is determined by the volume fraction of water in the volume of the mixture:

$$\phi = \frac{V_w}{V_w + V_s} \quad (\text{Eq. 5})$$

where

V_w = calculated volume of water

V_s = calculated volume of the sample

In appendix A, the porosity is calculated from equation 5 and used to determine solid sample conductivity in equation 4.

To measure thermal conductivity using the WHOI-MIT needle probe apparatus (see figures 1 and 2), the saturated sample should first equilibrate to room temperature. A KWH-series needle is inserted into the sample and connected with the "K-box" on function switch 3. Two decade resistance boxes connect to cal 1 and cal 2, function switches 1 and 2, and the heater power is applied by function switch 4. A voltmeter and a chart recorder are also plugged into the K-box.

The temperature of the sample is measured by the thermistor on function 3 as a voltage on the voltmeter. Decade box 1, cal 1, is set to match this voltage reading, and the determined resistance is converted to an ambient

temperature by computed calibration sheets for each KWH-series needle. A temperature 2°C above the measured ambient is chosen, and decade box 1, cal 1, is set to the corresponding resistance. A temperature 7°C above the measured ambient is chosen, and decade box 2, cal 2, is set to the corresponding resistance. The chart recorder is calibrated to the upper and lower temperatures as its zero and full scale bounds, respectively, by the offset and gain controls on the apparatus. The linear recorder scale is maintained by a linearizing bridge circuit in the K-box.

The chart recorder, set at a rate of 4 inches per minute, and the heater power are turned on. The trace on the chart record between 10 seconds and 2.0 minutes will be a logarithmic function of temperature versus time. The temperature values at 0.25 minute intervals on the chart record are plotted on 2 cycle, 70 division semi-log paper, and a linear, best-fit extrapolation to the edges gives the 0.1 minute (t_1) and 10.0 minute (t_2) temperatures. A value for ΔT can be calculated and used in equation 2 to find conductivity, K (see figures 3 and 4).

FUSED SILICA GLASS STANDARD

The thermal conductivity of fused silica glass has been documented by numerous experiments using steady-state methods. A compilation of these values is shown in figure 5. An extrapolation of each data group to 25°C by least

squares regression gives a mean value of $3.093 \text{ mcal/cm s } ^\circ\text{C}$ with a standard deviation of $0.232 \text{ mcal/cm s } ^\circ\text{C}$. The significant scatter may be due to different samples or apparatus used, but each shows a similar linear increase with temperature, indicating similar temperature coefficients.

Other measurements of the conductivity of fused silica at room temperature have been in close agreement. Sass et al (1971) measured 17 fused silica samples using a chip steady-state technique at $3.29 \pm .02 \text{ mcal/cm s } ^\circ\text{C}$, and a solid disc steady-state measurement at $3.26 \pm .02 \text{ mcal/cm s } ^\circ\text{C}$. Ratcliffe (1959), using hot plate and cold bath steady-state methods, determined a value of $3.28 \pm .02 \text{ mcal/cm s } ^\circ\text{C}$. Birch and Clark (1940) measured conductivity of fused silica at $3.26 \pm .03 \text{ mcal/cm s } ^\circ\text{C}$ with the steady-state method. The conductivity of fused silica glass is well-constrained by these measurements and is a good standard for needle probe calibration. The standard error intervals determined for these values are purely statistical, not systematic, and are not necessarily representative of the total error for each study.

A fused silica sample of known thermal conductivity was chosen as the standard for needle probe calibration. Corning (Corning Glass Co., Corning, N.Y.) fused silica no. 7940 was obtained for its specified physical properties and high purity. The thermal conductivity of this glass was given by the manufacturer as $3.29 \text{ mcal/cm s } ^\circ\text{C}$, and the density as 2.202 g/cm^3 .

DATA AND DISCUSSION

Thermal conductivity of fused silica standard was measured using four KWH-series needles, nos. 4, 5, 9, and 12, by the technique described above. The calculated conductivities are corrected using a porosity value $\phi = 0.3419$, as determined in appendix A, from equation 4. The conductivity measurements are shown in appendix C and are plotted in figure 6.

The mean conductivity of all measurements ($n = 21$) on three silica samples is 3.287 ± 0.154 (S.D.) mcal/cm s $^{\circ}\text{C}$. The mean of the values determined by each needle, the right-hand column in appendix C, varies from 3.248 ± 0.192 to 3.338 ± 0.159 mcal/cm s $^{\circ}\text{C}$. The mean of these four values is 3.294 ± 0.152 ($\pm 4.6\%$). The conductivity values measured by each needle gives a mean and deviation close to those determined by all measurements. This implies that the variation between needles is within the experimental error for any measurement and is probably not a significant source of error. Similarly, the mean conductivity value for each sample of silica glass is within the mean $\pm$ standard deviation of the other two samples, so that there is no significant difference between samples.

The standard deviation of all measurements, about 4.7% of the mean, is only a statistical result and ignores the systematic errors. This is the reproducibility of the technique using any needle, and is approximately the same as that determined by Von Herzen and Maxwell (1959). Since the error between the needles is negligible, the standard error of all 21 measurements, 0.03 mcal/cm s $^{\circ}\text{C}$, gives a 1% interval of confidence in the relative mean determined.

Errors that may cause systematic variations include curve fit variations, porosity variation and measurement accuracy, and thermistor calibration. The linear curve used in the analog plotting can be reproduced within a 2% error interval. The porosity may change with time due to the settling of sediment spheres in a water matrix. In addition, air saturation and measurement losses may affect the porosity, creating a small error noticeable by a difference in the weight of the sample before and after saturation. Lastly, a recent recalibration of the thermistors gave a change of 2% for a 5° drift in the measured temperature. These possible systematic errors do not affect the interval of reproducibility of 4.7%, nor the confidence of the relative mean determined, but could bias absolute value obtained.

The statistical mean has only a -0.091% difference from the given conductivity by Corning of 3.29 mcal/cm s °C, and the mean of the values determined separately by each needle a +0.12% difference. These values are also close to the conductivity of fused silica determined by earlier experimentors, as summarized above. The excellent agreement of these average values with previous values suggests that our data and the needle probe technique do not have any systematic biases greater than about 1%.

Appendix A: Porosity

V_S = Volume of silica
 W_S = Weight of silica
 V_W = Volume of interstitial water = W_W
 W_W = Weight of interstitial water
 W_W' = Weight of equivalent volume of water
 W_C = Weight of container
 $W_t = W_S + W_W + W_C$
 ρ_W = Density of water (Clark, 1966)
 ρ_S = Density of silica (Corning)

$$\rho = \frac{V_W}{V_W + V_S}$$

$$\begin{aligned}
 V_W &= W_W / \rho_W \\
 V_S &= W_S / \rho_S \\
 V_W + V_S &= W_W / \rho_W + W_S / \rho_S
 \end{aligned}$$

	sample 1	sample 2
W_C	20.45	98.61
$W_C + W_S$	64.81	185.60
$W_C + W_S + W_W$	75.26	206.04
W_W	10.45	20.44
W_S	44.36	87.00
ρ_W	0.997	0.997
ρ_S	2.202	2.202
V_W	10.481	20.502
V_S	20.415	39.510
$V_W + V_S$	36.626	60.012
$V_W / V_W + V_S$	0.3422	0.3416
$\phi (\%)$	34.22	34.16

$$\phi_{\text{mean}} = 34.19 \%$$

Appendix B: Density of Fused Silica

V_S = Volume of silica
 W_S = Weight of silica
 V_W = Volume of interstitial water = W_W
 W_W = Weight of interstitial water from appendix A
 $W_{W'}$ = Weight of equivalent volume of water
 W_C = Weight of container
 $W_t = W_S + W_W + W_C$

$$\begin{aligned}
 V_S &= W_{W'} - W_W - W_C \\
 V_S &= (39.58 \text{ g}) - (10.45 \text{ g}) - (9.32 \text{ g}) \\
 V_S &= 19.81 \text{ cm}^3
 \end{aligned}$$

$$\begin{aligned}
 W_S &= W_t - W_W - W_C \\
 W_S &= (63.20 \text{ g}) - (10.45 \text{ g}) - (9.32 \text{ g}) \\
 W_S &= 43.43 \text{ g}
 \end{aligned}$$

$$\begin{aligned}
 \rho &= W_S / V_S \\
 \rho &= (43.43 \text{ g}) / (19.81 \text{ cm}^3) \\
 \rho &= 2.192 \text{ g/cm}^3 \\
 \rho_{\text{Corning}} &= 2.202 \text{ g/cm}^3
 \end{aligned}$$

$$\text{Error} = -0.45\%$$

Appendix C: Thermal Conductivity of Fused Silica

KWH needle	Sample	K_m	$K_r(\rho = .3419)^*$	K_{mean}	σ
4	1	2.460	3.236	3.328	0.102
4	2	2.531	3.379		
4	2	2.467	3.250		
4	2	2.564	3.446		
5	2	2.370	3.058	3.264	0.153
5	3	2.541	3.399		
5	3	2.533	3.383		
5	2	2.508	3.333		
5	2	2.415	3.147	3.248	0.192
9	1	2.527	3.371		
9	1	2.417	3.151		
9	2	2.365	3.048		
9	3	2.471	3.258	3.338	0.159
9	2	2.621	3.563		
9	2	2.503	3.322		
9	1	2.351	3.021		
12	1	2.552	3.442	3.294	0.152
12	2	2.412	3.141		
12	2	2.449	3.214		
12	1	2.602	3.524		
12	2	2.527	3.371	3.29	+0.12%
	K_{mean}	2.485	3.287		
	σ	0.0767	0.154		
	$K_{Corning}$		3.29		
	Error		-0.091%		

* Thermal conductivity of water assumed 1.45 TCU (Clark, 1966, Table 21-14)

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Fig. 1. Conductivity Apparatus. A. K-box. B. Conductivity needle probe.
C. Chart recorder. D. Decade resistance boxes. E. Multimeter.

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Fig. 3. Typical chart record of conductivity measurement on fused silica. Temperatures are chosen at 0.25 minute intervals to be plotted as in fig. 4.

KW49 f3①

Fig. 4. Typical logarithmic plot of temperature versus the logarithm of time. The value of K determined is of the silica and water mixture. The divergence of the line near 2 minutes is due to the boundary effect of the container.

Fig. 5. Compilation of previous measurements of thermal conductivity of fused silica glass. The mean value is 3.093 mcal/cm s °C at 25°C.

Fig. 6. Needle probe measurement of thermal conductivity on fused silica glass. The mean value ($n = 21$) is 3.287 ± 0.154 (S.D.).

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