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Application of the Line-Source Technique for Vacuum Thermal Conductivity Measurements

LAWRENCE R. GREENWOOD*

NASA Langley Research Center, Hampton, Va.

AND

ROBERT A. COMPARIN†

Virginia Polytechnic Institute, Blacksburg, Va.

THE space environment can alter the thermal and mechanical properties of engineering materials.¹ Properties must be measured in the environment of interest (i.e., in situ) to obtain valid results.² The necessity of making in situ measurements of thermal conductivity in a variety of environments (e.g., vacuum, planetary atmospheres) led to an evaluation of available techniques for this application. The conventional techniques, such as the guarded hot plate and comparator, are not well suited for environmental studies because the large contact areas between a heater plate and the sample could alter the interaction with the surrounding environment. The line-source technique appears to be well suited for making in situ measurements in a variety of environments, because the measuring apparatus is inside the sample, and all surfaces are free to interact with the surrounding environments. There are little data available, however, on the accuracy of this technique, or on its use in vacuum. This Note presents the results of a recent study

wherein 1) the accuracy of the line-source technique was established for application to an ablative heat-shield material, and 2) the technique was applied to make vacuum thermal conductivity measurements.

Line-Source Technique

A heater wire and thermocouple are placed inside the sample as shown in Fig. 1. Application of the equation for heat conduction with an instantaneous line-source leads to

$$K = [q/4\pi(\theta_2 - \theta_1)] \ln(t_2 - t_0)/(t_1 - t_0) \quad (1)$$

where K = thermal conductivity, q = heat input, θ = temperature, t = time after initiation of heat generation, and t_0 = correction factor.

The assumptions necessary to solve the basic differential equation of conduction to obtain Eq. (1) include: 1) a semi-infinite heat sink (i.e., no change in the sample surface temperature), 2) no heat loss at the sample ends, 3) a heat source that is vanishingly small, and 4) an instantaneous heat input at time zero. The integration of the basic differential equation results in a series solution, and to obtain Eq. (1) the assumption is made that $r^2/4\alpha t$ is very small, where r is the radial distance from the center line of the sample to the point where the temperature changes are measured, and α is the thermal diffusivity. A detailed discussion of the assumptions involved is given in Ref. 3.

In the experimental application of Eq. (1) to measure thermal conductivity, the assumed conditions can only be approximated, and departures from these initial assumptions result in errors. When $\log t$ vs temperature (θ) is plotted for a line-source measurement, Eq. (1) indicates that the resulting curve should be linear. However, since the assumed conditions can only be approximated, a plot of the experimental data will deviate from a straight line. Errors due to assumption (1) can be made negligible by a proper combination of sample size and test time.

Van der Held and Van Drunen³ have shown that the error due to assumptions can be eliminated by including the t_0 correction factor, a constant which must be determined experimentally for each sample. A number of researchers have proposed methods of determining t_0 , but all methods are difficult to apply and introduce uncertainties into the thermal conductivity calculations.

Since the value of t_0 is independent of time, it is apparent that the influence of t_0 on K becomes less as the test time in-

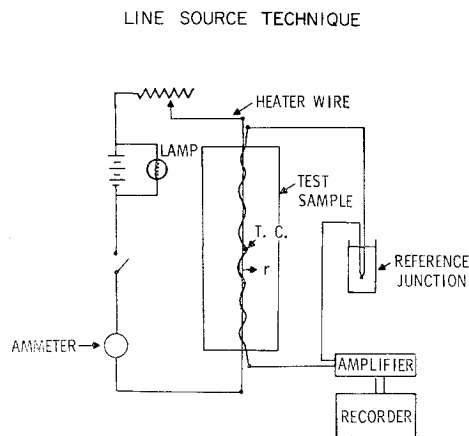


Fig. 1 Line-source technique for measuring thermal conductivity.

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* Head, Space Vacuum Laboratory Section. Associate AIAA.

† Professor, Mechanical Engineering Department. Member AIAA.

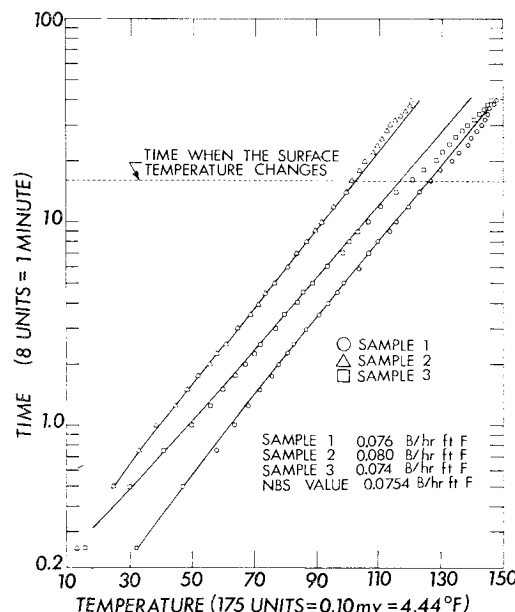


Fig. 2 Temperature variation with time at atmospheric pressure.

creases; at long test times, the influence of t_0 will be negligible, and the $\log t$ vs θ trace will become linear. In practice, the test time is limited to the time during which the surface temperature θ_s remains constant [see assumption (1)]. It is possible for the influence of t_0 to become negligibly small before θ_s , the surface temperature, begins to change, and K can then be calculated neglecting the t_0 correction. To determine when t_0 can be neglected in practice, it is necessary to monitor θ_s and the linearity of $\log t$ vs θ simultaneously.

Experimental Procedure

The accuracy of the line-source technique was established by comparisons with guarded-hot-plate measurements made by the National Bureau of Standards (NBS) according to the Standard Method of Test ASTM C177. The mean sample temperature during the test was 70.1°F with a 40.0°F/in. temperature gradient through the sample.

The line-source is initiated by closing a switch (Fig. 1). The voltage and current are measured and the temperature-time response is monitored using the recorder.

The material studied was NASA E4Al, a filled silicone elastomer with a density of 42.5 lb/ft³. Slabs, 8 × 8 × 1 in., were prepared and sent to the NBS for the guarded-hot-plate measurements. Then the samples were returned to our laboratory and cut into 1 × 1 × 4 in. samples for line-source measurements. Additional line-source samples were prepared from a separate batch of heat-shield materials and used for the vacuum measurements.

Vacuum thermal conductivity measurements were made in a bell jar at 10⁻⁵ torr for a 9-day vacuum exposure time at 70°F. Three samples were tested simultaneously. In addition, control samples were tested at constant relative humidity (55%) and atmospheric pressure. Daily measurements were made on these control samples for comparison with vacuum-stored samples.

Discussion

The NBS measured K to be 0.0754 Btu/hr-ft °F (the average K of the 8 × 8 × 1 in. sample). Since the t_0 correction could influence the line-source accuracy to a large extent, tests were performed to determine the maximum test times (thus minimizing t_0) and yet not violate the assumption concerning no change in surface temperatures [see assumption (1)]. The intent was to go to long test times and thus eliminate the necessity of determining t_0 . By placing thermocouples on the surface of the samples during line-source measurements, it was found that it took about 2 min before θ_s changed. This would indicate that measurements should be taken prior to 2 min so as to avoid violating the surface temperature boundary condition.

Typical line-source experimental data are shown in Fig. 2; the curves for $\log t$ vs θ are nearly linear, indicating that $t_0 \approx 0$. The values of K calculated from the linear portions of the curves in Fig. 2 for the three samples are 0.076, 0.080, and 0.074 Btu/hr-ft °F. The average value measured using the line-source technique is within 2% of the NBS value. The maximum deviation for any one of the line-source samples was less than 7% of the NBS value.

Figure 3 shows K vs storage time for the vacuum and ambient control samples; each data point is the average value from the three samples tested at the same conditions. For the vacuum samples, K decreased about 15% after 1-day exposure and remained approximately 15% lower than the ambient samples for the 9-day duration of the test. At the end of the 9 days, the bell jar was vented to the atmosphere where the relative humidity was about 30% and K for the re-exposed samples was measured daily for the next 3 days. After 1-day re-exposure, K reverted to approximately its original value and remained at this value for the remainder of the tests.

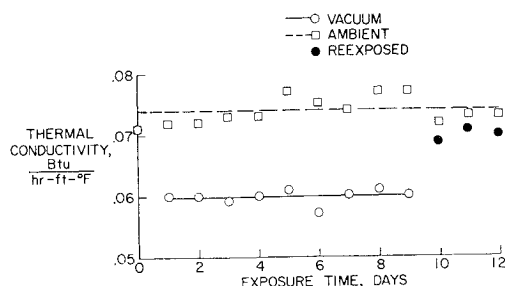


Fig. 3 Effects of vacuum on the thermal conductivity of heat-shield material.

Conclusions

The line-source technique measured the average thermal conductivity of a heat shield material to within 2% of the guarded-hot-plate value, with a maximum deviation for any one sample of less than 7%. The technique appears to be well suited for use in vacuum. For the E4Al material, a vacuum-induced decrease in thermal conductivity of 15% was observed after 24 hr in the vacuum environment.

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A System for Removing Contaminants from Spacecraft Optical Systems

W. C. GIBSON*

Lockheed Electronics, Houston, Texas

AND

J. L. MODISSETTE†

Houston Baptist College, Houston, Texas

BEGINNING early in the Mercury Program and continuing through the orbit of Apollo IX, manned spacecraft of the United States have encountered the problem of window filming to varying degrees. It was initially thought that the film might be caused by materials blowing back from the nose cone during the launch phase. Postflight chemical analysis of the contaminant, performed by Manned Spacecraft Center (MSC) personnel, revealed the residue to be composed mainly of silicones. As the manned spaceflight program continued it became increasingly evident that the RTV silicon impregnated gasket material used in securing the window to the spacecraft was responsible for the problem. The reader is referred to NASA document TND-4916

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* Associate Engineers, Houston Aerospace Systems Division, Optical Experiments Department. Associate AIAA.

† Chairman, Division of Science and Mathematics; formerly Acting Chief, Astronomy Branch, NASA manned Spacecraft Center. Associate Fellow AIAA.