Thermal Diffusivity Measurements of Carbon Insulations

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In the laser flash technique for measuring thermal diffusivity a flash of radiant energy is deposited over one surface of a sample.[1] The diffusivity is calculated from the sample thickness and the time required for the rear-face temperature rise to reach a known percentage of its maximum value. The method has been used to make measurements on materials whose diffusivities range from 0.001 to 10 $cm^2 s^{-1}$ (a range of 10^4) and from 80 to 2500 K. The time-temperature history of the rear face can be made dimensionless so that the experimental rise curve can be compared to mathematical models and deviations from these models detected.[2] It is possible to correct for radiation heat losses.[2] It has been shown that measurements are possible on very heterogeneous dispersed composites and on layered samples.[2] In fact, it is the most versatile technique known for determining thermal diffusivity and hence thermal conductivity.

However, the technique has not been applied extensively to porous insulation materials. The reasons for this include problems associated with thin samples of soft materials with ill-defined surfaces, laser beam penetration into the sample interior, very large heat loss corrections and excessive temperature rise caused by the laser flash. The observed rise times depend upon the square of the thickness, so errors in the effective thickness are magnified. Thin samples are required due to the relatively long times for the heat to diffuse through the sample of insulation and the face that heat losses increase rapidly as the measuring time is lengthened. Because of the small mass of the insulation samples, normally used power settings result in temperature rises that are very large. The desired rear face temperature is one or two degrees. In order to achieve this, front face temperature rises are often about 80°C. However, for a thin carbon foam insulator subject to the same experimental conditions, the rear face temperature rise may be 40 to 80°C and the front face rise over 400°C. This in turn leads to excessive heat losses, enhanced radiation transmission of heat through the sample and non-linearity of the rear face temperature rise detector.

Thermocouples cannot be used directly to measure rear face temperature rise of fibrous or foam insulation materials. Apart from the problem of determining temperature transients of poor conductors using thermocouples with relatively high conductivity, there is the serious complication of attaching the very small diameter thermocouples to a representative mass. Therefore, i.r. detectors are used. These detectors are nonlinear over temperature ranges of tens to hundreds of degrees. However, they are satisfactory for following small temperature rises and generally can be considered to be linear over a one to two degree span. Since the actual temperature rise is not required in the determination, all that is necessary is that the detector response be linear over the observed rise.

In the present program, several carbon bonded carbon fiber samples (supplied by ORNL) were used. The bulk sample densities were 0.18 to 0.28 gm cm^{-3} or about one tenth that of graphites. The specific heat values closely followed those recommended for well-graphitized graphites and carbon/carbon materials. "Apparent" diffusivity values (α) are calculated from the relation $\lambda = k_x \ell^2 / t_x$ where ℓ is the sample thickness, t_x is the elapsed time required for the rear face temperature rise to reach x% of its maximum value and k_x is a constant, calculable from the Carslaw and Jaeger solution of one-dimensional heat flow in a semi-infinite material subject to a Delta heat input.[3] It was found that the diffusivity values for a given laser shot increased with increasing x. This is a normal event when radiation losses are present and there are several standard radiation loss procedures to correct the calculated diffusivity values. However, in the case of the carbon insulations, it was noted that the apparent diffusivity calculated at any x% rise increased as the laser power level increased. Also placing absorbers between the sample and laser resulted in larger calculated diffusivity values as the amount of energy absorbed was increased.

The primary reason for this behavior was traced to i.r. detector non-linearity. We found that the liquid-nitrogen cooled InSb detector we use was reasonably linear over a 5 degree rise but became very non-linear as the magnitude of the temperature rise increased beyond this. We were able to determine the non-linearity for a particular set of conditions and correct the rise curve. The corrected values were substantially (about 35%) greater than the values obtained without considering detector non-linearity but including heat losses. It was discovered that the heat loss corrections for the data corrected for non-linearity were significantly less than those calculated from the original data. It was further discovered that diffusivity values obtained when the laser power absorbed by the samples was decreased to the minimum consistent with obtaining measurable rear face rise curves were in good agreement with the values obtained by correcting for laser non-linearity. Correcting for non-linearity is a tedious operation (the corrections depend upon the bias and signal amplification used and therefore vary from run to run). Thus the importance of using an absolute minimum of power was demonstrated.

References

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