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N94-25132

An Extended Laser Flash Technique for Thermal Diffusivity Measurement of High-Temperature Materials

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Abstract

Knowledge of thermal diffusivity data for high-temperature materials (solids and liquids) is very important in analyzing a number of processes, among them solidification, crystal growth, and welding. However, reliable thermal diffusivity versus temperature data, particularly those for high-temperature liquids, are still far from complete. The main measurement difficulties are due to the presence of convection and the requirement for a container. Fortunately, the availability of levitation techniques has made it possible to solve the containment problem. Based on the feasibility of the levitation technology, a new laser flash technique which is applicable to both levitated liquid and solid samples is being developed. At this point, the analysis for solid samples is near completion and highlights of the technique are presented here.

The levitated solid sample which is assumed to be a sphere is subjected to a very short burst of high power radiant energy. The temperature of the irradiated surface area is elevated and a transient heat transfer process takes place within the sample. This containerless process is a two-dimensional unsteady heat conduction problem. Due to the nonlinearity of the radiative plus convective boundary condition, an analytic solution cannot be obtained. Two options are available at this point. Firstly, the radiation boundary condition can be linearized, which then accommodates a closed-form analytic solution. Comparison of the analytic curves for the temperature rise at different points to the experimentally-measured values will then provide the thermal diffusivity values. Secondly, one may set up an inverse conduction problem whereby experimentally obtained surface temperature history is used as the boundary conditions. The thermal diffusivity can then be elevated by minimizing the difference between the real heat flux boundary condition (radiation plus convection) and the measurements.

Status of an experimental study directed at measuring the thermal diffusivity of high-temperature solid samples of pure Nickel and Inconel 718 superalloys are presented. Preliminary measurements showing surface temperature histories are discussed.

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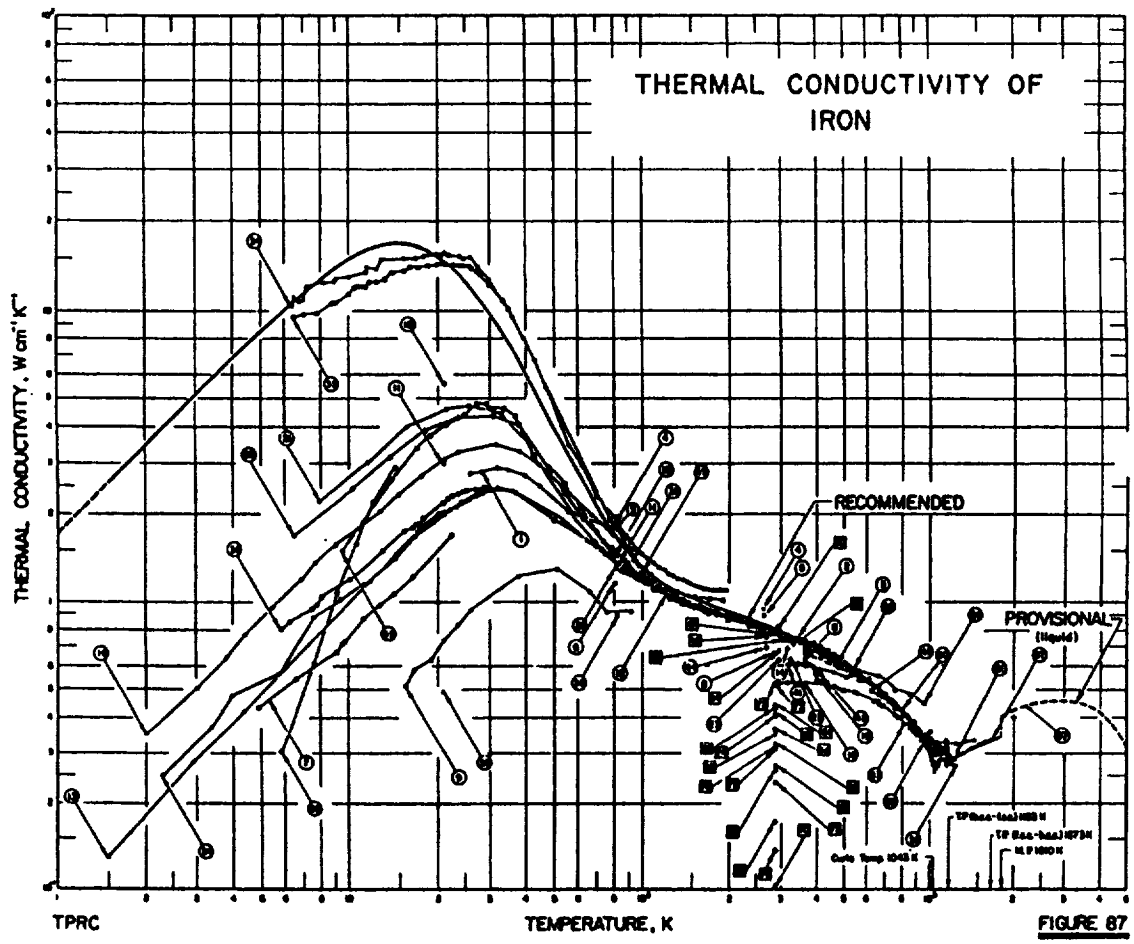
Acknowledgment: The work reported here is supported by the NASA Grant NAGGW-1192.


THERMAL CONDUCTIVITY OF
IRON
RECOMMENDED
PROVISIONAL
(liquid)
THERMAL CONDUCTIVITY, W cm⁻¹ K⁻¹
TEMPERATURE, K
TPRC
FIGURE 87

6.6. EXPERIMENTAL DATA FOR THE VISCOSITIES OF PURE LIQUID METALS

It is very difficult to state definitely the accuracy of viscosity measurements for liquid metals. Errors of ± 1 to ± 20 per cent would seem to be a fair estimate with the exception of a few metals. There are not many well-established data for liquid metal viscosities. Viscosity data for pure liquid metals are listed in Table 6.3. Data for others currently available are given in Table 6.6.

As shown in Fig. 6.26, experimental viscosity values for liquid aluminium exhibit very large discrepancies, and the values calculated from eqn (6.40)

taken from Iida, T. and Guthrie, R. I. L. ***The Physical Properties of Liquid Metals,*** Oxford Science Publications, 1988.

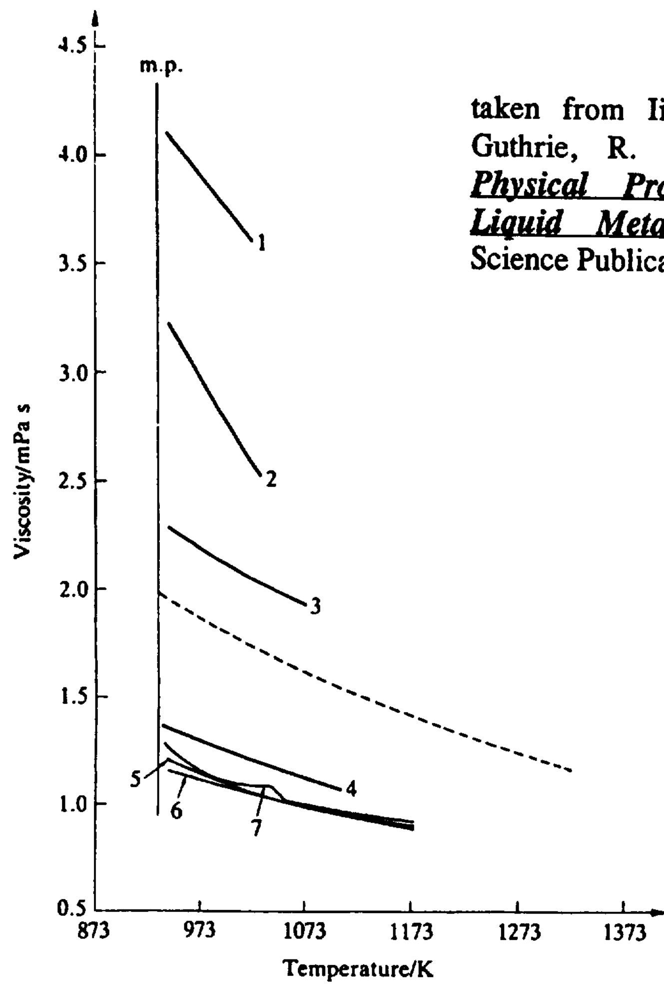


FIG. 6.26. Viscosity of liquid aluminium as a function of temperature, as determined by several workers: (1) Jones and Bartlett (1952–3); (2) Yao and Kondic (1952–3); (3) Yao (1956); (4) Rothwell (1961–2); (5) Gebhardt and Detering (1959); (6) Gebhardt, Becker, and Dorner (1954); (7) Lihl, Nachtigall, and Schwaiger (1968); ----- predicted from eqn (6.40).

Pulse Diffusivity Method

The thermal diffusivity, α, can be calculated as

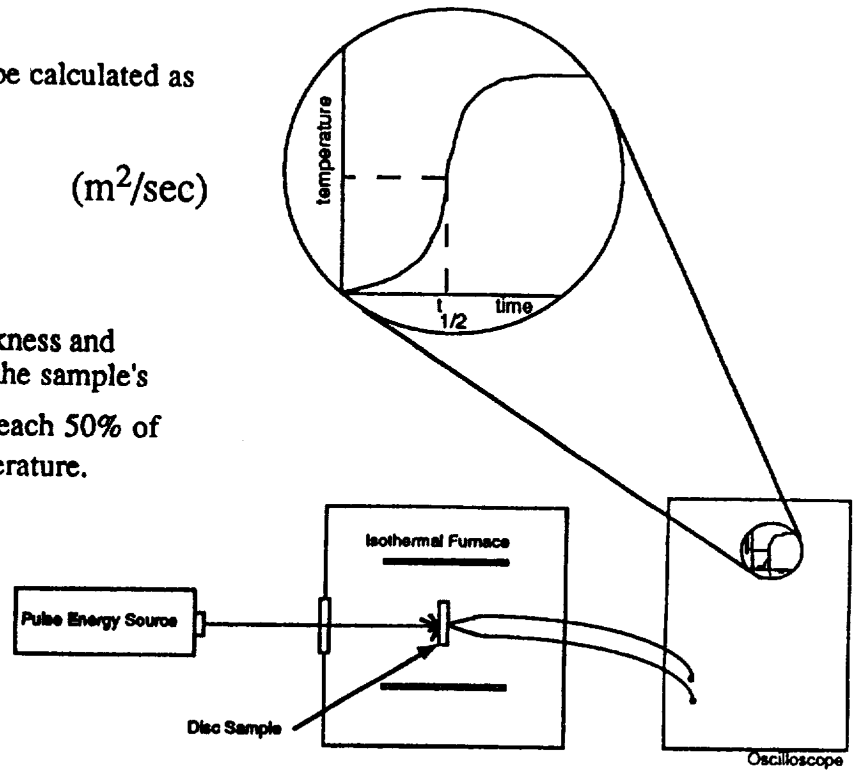
$$\alpha = 0.1388 \frac{L^2}{t_{1/2}} \qquad (m^2/sec)$$

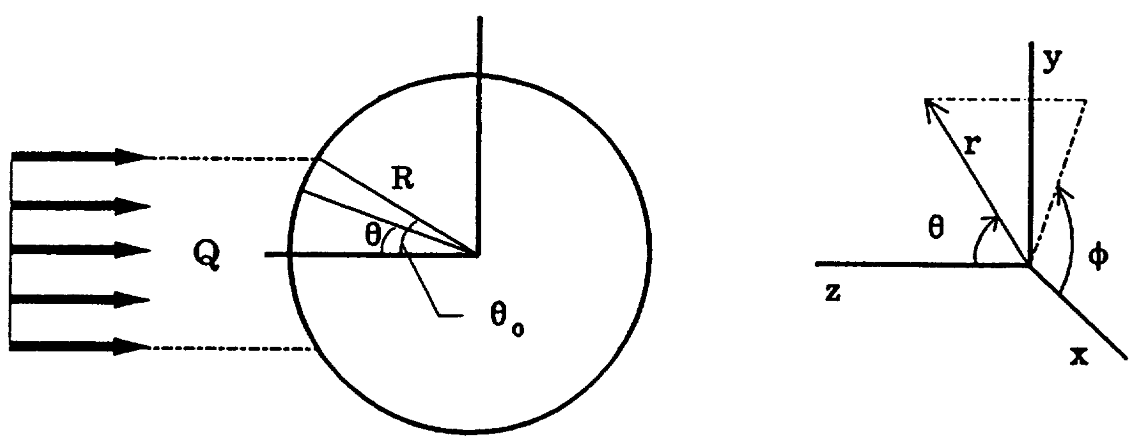
where

L is the sample thickness and
$t_{1/2}$ is the time for the sample's rear surface to reach 50% of maximum temperature.

Note:

$$\alpha = \frac{k}{\rho c_p}$$





(a) (b)

FEATURES OF THE FLASH TECHNIQUE

Original technique:

☞ Proposed by Parker *et al.*[1] in 1961.
☞ According to the Science Citation Index, more than 300 scientific papers have referred to Parker *et al.*[1]

The features of this technique were reviewed by Taylor and Maglic[2] in 1984:

☞ A literature survey in the early seventies showed that about 75% of the thermal diffusivity data reported within that period were obtained using the flash method.
☞ Variety of materials including metals, alloys, ceramics, composites, etc.
☞ Thermal diffusivity values (10^{-7} to 10^{-3} m^2/s) have been reported in the temperature range of 100 to 3300 K.
☞ Small disk-shaped specimen (6 ~ 16 mm in diameter and less than 4 mm in thickness)
☞ Half-time transients within 0.04 to 0.25 seconds

Variations of the flash method up to now account for:

Radiation Losses	Multi-Layer Specimen
Finite Pulse Time	Large Sample Effects

1. W. J. Parker, R. J. Jenkins, C. P. Butler, and G. L. Abbott, "Flash Method of Determining Thermal Diffusivity, Heat Capacity, and Thermal Conductivity," J. Appl. Phys., 1961, Vol. 32, pp. 1679-1684.
2. R. E. Taylor and K. D. Maglic, "Pulse Method for Thermal Diffusivity Measurement," Compendium of Thermophysical Property Measurement Methods, Vol. 1, edited by K. D. Maglic, Plenum Publishing Corporation, 1984, pp. 305-336.

THEORETICAL ANALYSIS
Governing Equation

Assuming that the spherical sample is homogeneous and the thermophysical properties are independent of temperature for the given temperature range of interest, the extended flash technique can be modeled as a two-dimensional (azimuthal symmetry) unsteady heat conduction within the sphere.

$$\frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2\frac{\partial \Theta}{\partial r}\right) + \frac{1}{r^2}\frac{\partial}{\partial \mu}\left[(1-\mu^2)\frac{\partial \Theta}{\partial \mu}\right] = \frac{1}{\alpha}\frac{\partial \Theta}{\partial t}$$

where:

r	radial coordinate within the sphere, m
t	time, s
T	temperature, K
T_{sur}	temperature of the surroundings, K
α	thermal diffusivity, m^2/s
Θ	temperature difference, defined as $(T - T_{sur})$, °C
μ	transformed θ coordinate, defined as $\cos\theta$

THEORETICAL ANALYSIS
Assumptions Related to the Initial Conditions

The following assumptions are made:

1. The intensity distribution of the radiation energy flux of the incident beam is known. In cases where a laser is utilized, a Gaussian distribution is assumed:

$$Q(r_b) = Q_M \exp(-2r_b^2/w_b^2)$$

where:

- Q_M peak energy flux at the center of the beam, J/m^2
- r_b radial distance from the center of the beam, m
- w_b diameter of the laser beam, m

2. The energy radiation is absorbed instantaneously and uniformly within a thin layer with the thickness g at the irradiated front surface ($r = R$, $0 \leq \theta \leq \theta_0$, $0 \leq \phi \leq 2\pi$). This layer of material is very thin compared to the radius of the sample.

3. The duration of the radiation burst is negligible compared to the rise time to half-maximum temperature characteristics being sought.

THEORETICAL ANALYSIS
Initial Conditions

The initial condition can be written as:

$$\Theta(r^*, \mu, 0) = \frac{Q\,\mu}{\rho c_p g} \qquad \left(\left(1-\frac{g}{R}\right) \leq r^* \leq 1,\ \mu_0 \leq \mu \leq 1\right)$$

$$\Theta(r^*, \mu, 0) = 0 \qquad \textit{rest of the sphere.}$$

where the assumption $T_i = T_{sur}$ is employed. The angle θ_0 represents the relative size of the sample and beam diameters.

NOTE: More realistic initial conditions are currently being studied.

THEORETICAL ANALYSIS
Boundary Conditions

The appropriate boundary condition accounting for both radiative and convective heat losses is:

$$-k\,T_n = \epsilon_{eff}\,\sigma\,F\,(T^4 - T_\infty^4) + h\,(T - T_\infty).$$

Upon linearization, it can be expressed as:

$$-k\frac{\partial\Theta}{\partial r}(R,\mu,t) = (4\epsilon_{eff}\sigma F T_{sur}^3 + h)\;\Theta(R,\mu,t)$$

where:

k	thermal conductivity, W/(mK)
ϵ_{eff}	effective hemispherical emissivity of the sample
F	radiation view factor
σ	Stefan-Boltzmann constant
h	convection heat transfer coefficient.

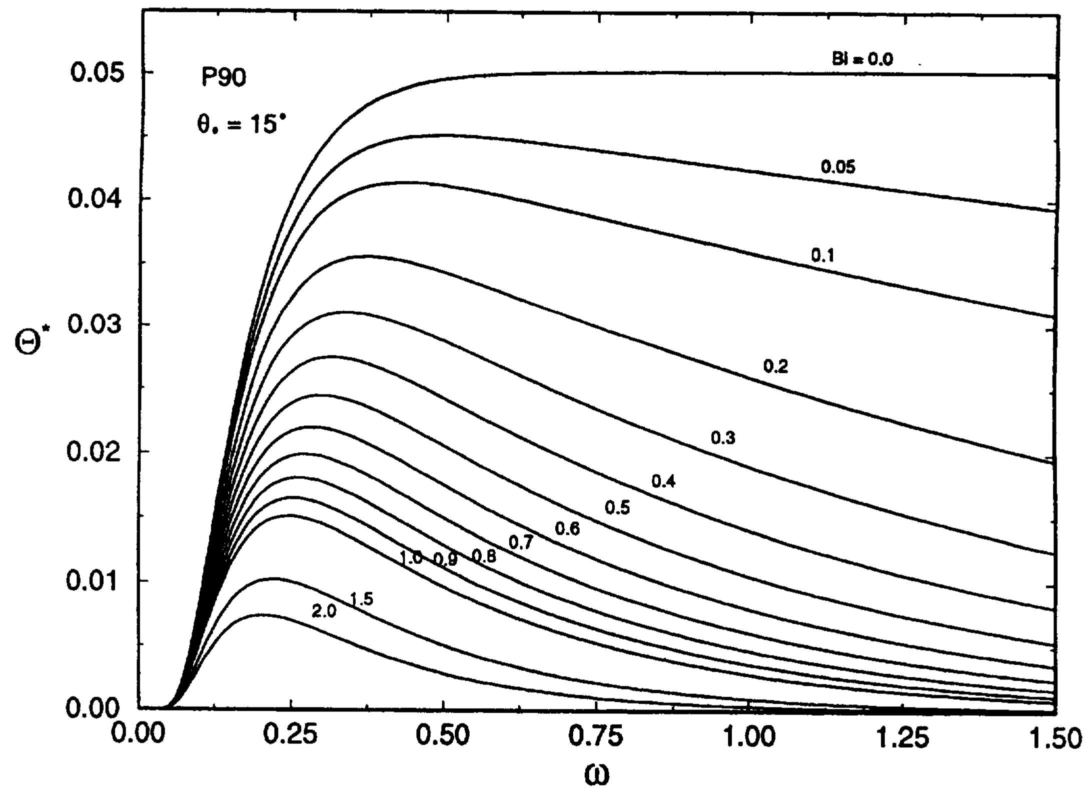
Alternately, by defining $r^* = r/R$, we can write:

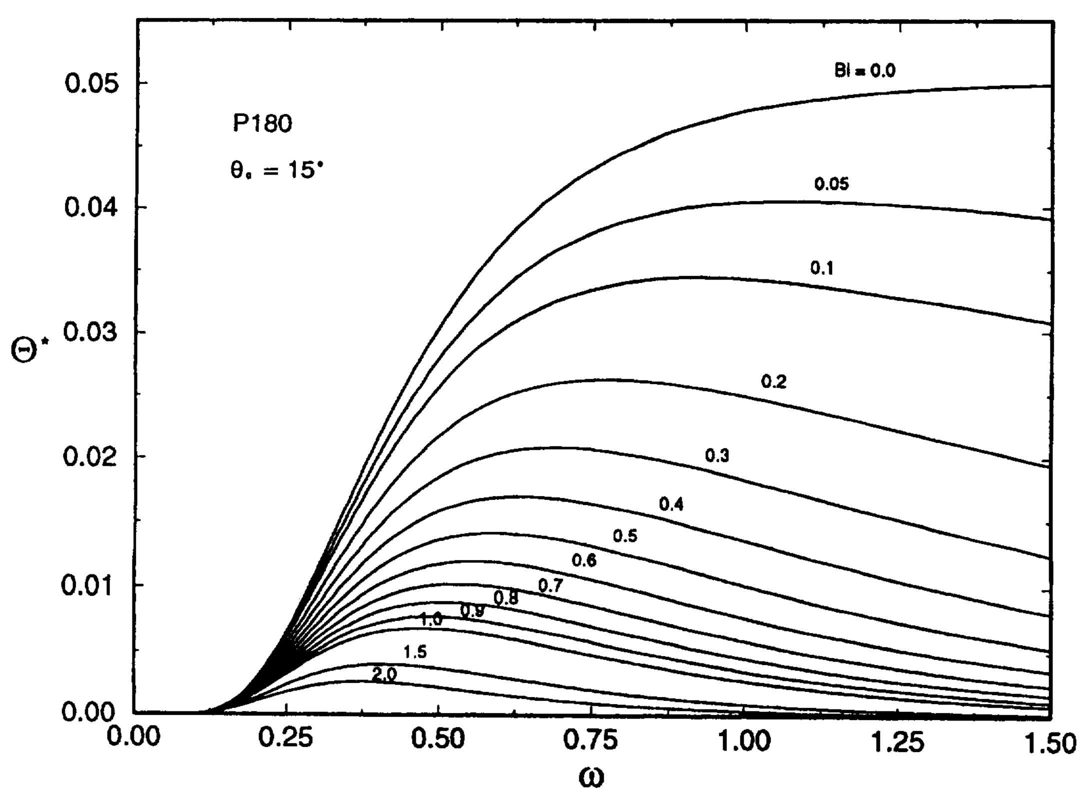
$$-\frac{\partial\Theta}{\partial r^*}(1,\mu,t) = Bi\;\Theta(1,\mu,t)$$

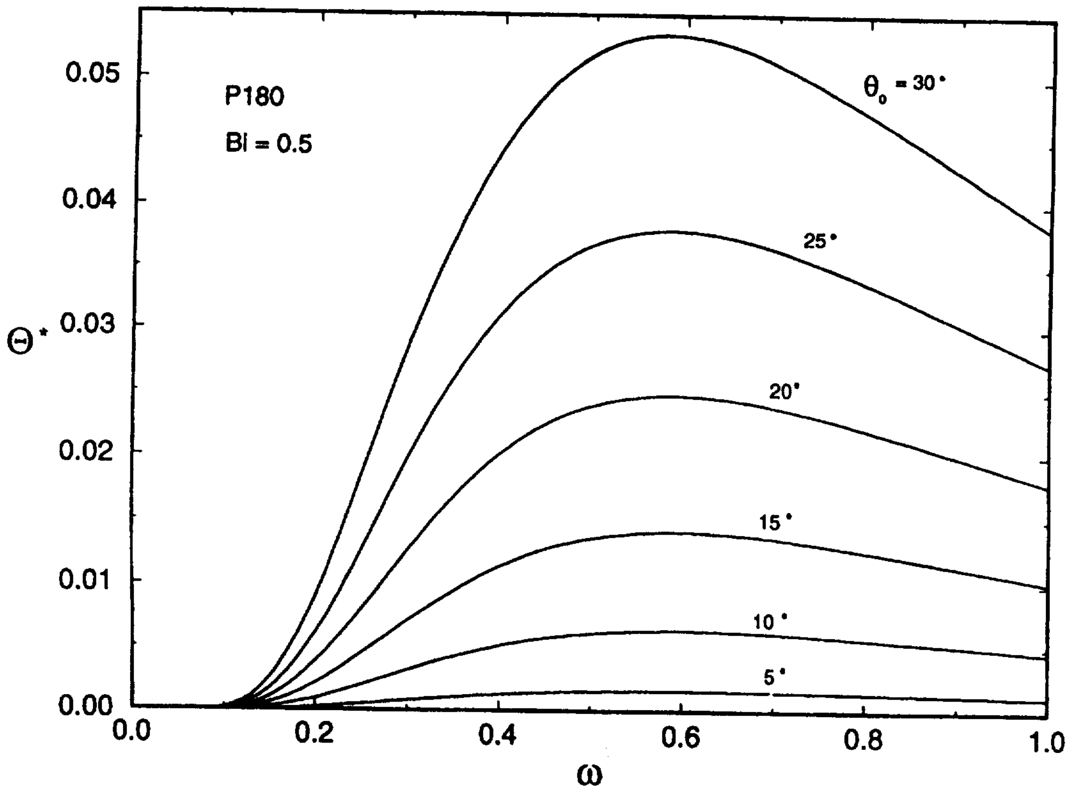
The Biot number (Bi) which is defined as:

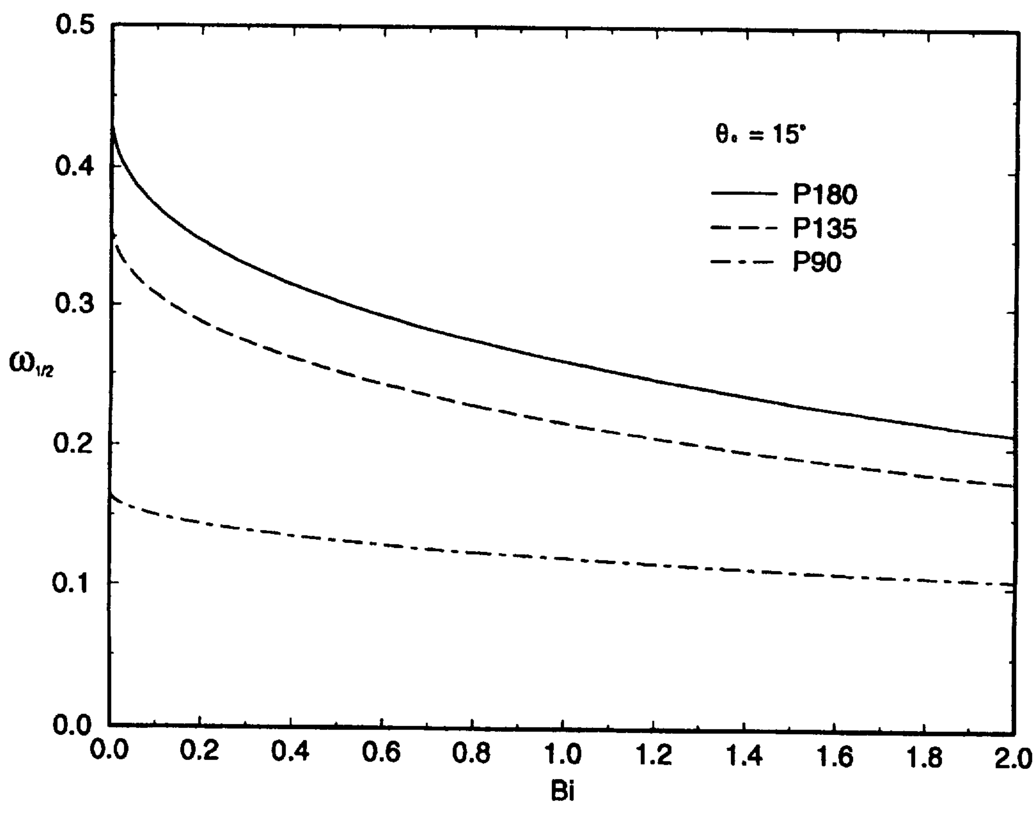
$$Bi = \frac{(4\epsilon_{eff}\sigma F T_{sur}^3 + h)\,R}{k}$$

is a measure of both convective and radiative surface heat losses. In the case of no heat loss, the Biot number equals zero.


P90
θ, = 15°
Bi = 0.0
0.05
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.5
2.0
Θ*
ω


P180
θ, = 15°
Bi = 0.0
0.05
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.5
2.0
Θ*
ω


0.05
0.04
0.03
0.02
0.01
0.00
0.0
0.2
0.4
0.6
0.8
1.0
P180
Bi = 0.5
θ₀ = 30°
25°
20°
15°
10°
5°
Θ*
ω


0.5
0.4
0.3
0.2
0.1
0.0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
θ₀ = 15°
P180
P135
P90
ω1/2
Bi

THERMAL DIFFUSIVITY DETERMINATION

A simple one-step method to determine the thermal diffusivity which is independent of the heat loss parameter (Bi) is proposed.

- ☞ Record the temperature rise histories, at least at two points on the surface of the sphere simultaneously.
- ☞ Determine the rise time to the half-maximum temperature $t_{1/2}$ from the experimental temperature rise curves.
- ☞ For a given energy distribution and incidence angle, the relationships between $\omega_{1/2}$ and Bi at the two points are expressed as:

$$[\omega_{1/2}]_1 = a_1 \, e^{-b_1\sqrt{Bi}} \qquad at\ point\ P_1$$

$$[\omega_{1/2}]_2 = a_2 \, e^{-b_2\sqrt{Bi}} \qquad at\ point\ P_2$$

Because the Biot number should be the same in both equations, it can be eliminated. Finally:

$$\alpha = \left[\frac{a_1^{b_2}}{a_2^{b_1}} \, \frac{[t_{1/2}]_2^{b_1}}{[t_{1/2}]_1^{b_2}} \right]^{\frac{1}{b_2-b_1}} R^2 = A \, \frac{[t_{1/2}]_2^{B}}{[t_{1/2}]_1^{1+B}} \, R^2$$

where the parameters A and B are as follows:

$$A = \left[\frac{a_1^{b_2}}{a_2^{b_1}} \right]^{\frac{1}{b_2-b_1}} \qquad B = \frac{b_1}{b_2-b_1}$$

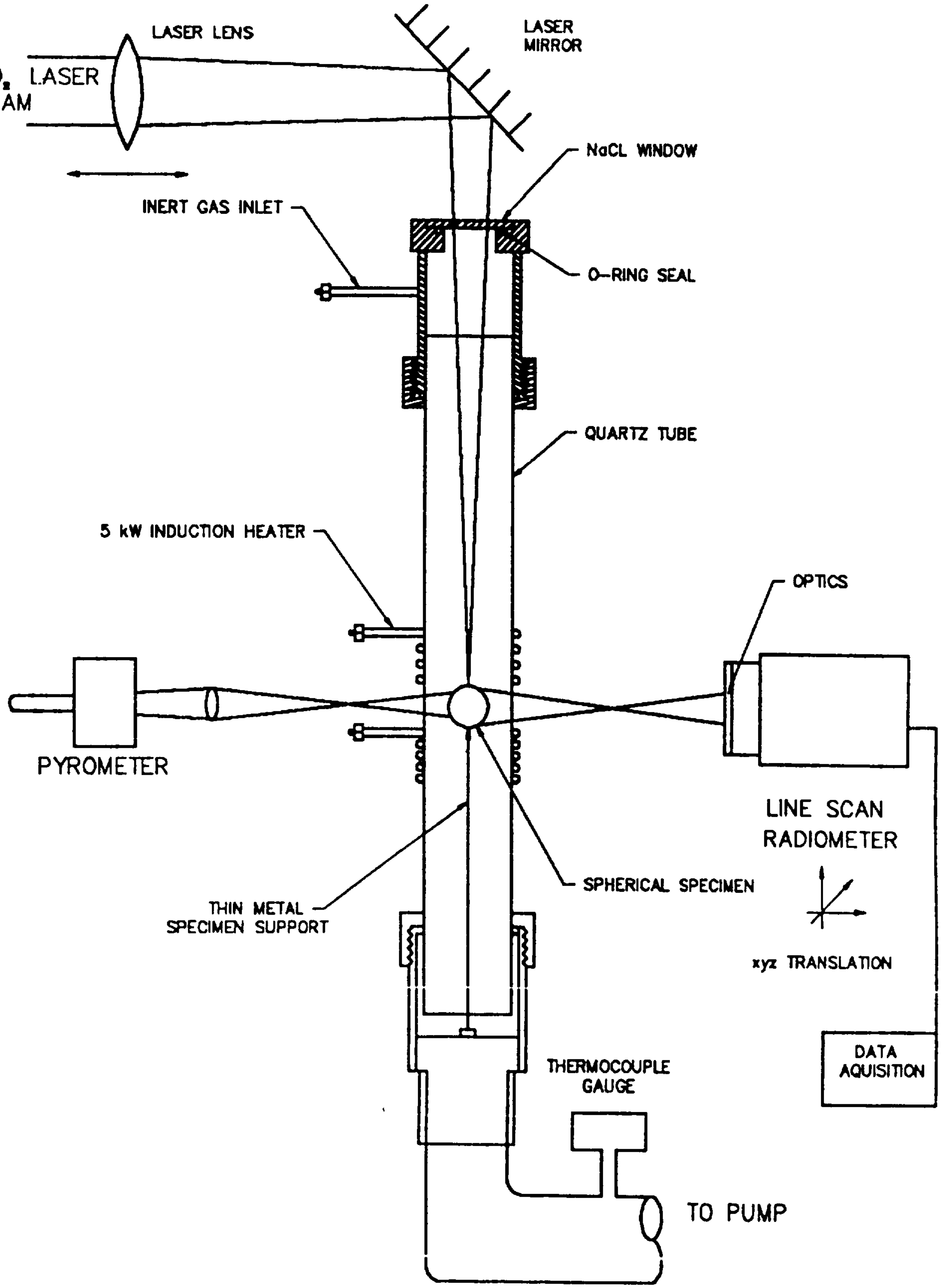
The Values of A and B
(P_1 = 90° and P_2 = 180°)

Gaussian Energy Flux Distribution

θ_0	5°	10°	15°	20°	25°	30°
A	0.03960	0.03751	0.03416	0.02966	0.02383	0.01742
B	1.53493	1.57327	1.63901	1.73608	1.89390	2.11846

Uniform Energy Flux Distribution

θ_0	5°	10°	15°	20°	25°	30°
A	0.039505	0.03725	0.033603	0.028711	0.0222435	0.015702
B	1.537113	1.578354	1.649957	1.758798	1.936426	2.190517


LASER LENS
LASER MIRROR
CO₂ LASER BEAM
NaCL WINDOW
INERT GAS INLET
O-RING SEAL
QUARTZ TUBE
5 kW INDUCTION HEATER
OPTICS
PYROMETER
LINE SCAN RADIOMETER
SPHERICAL SPECIMEN
THIN METAL SPECIMEN SUPPORT
xyz TRANSLATION
DATA AQUISITION
THERMOCOUPLE GAUGE
TO PUMP

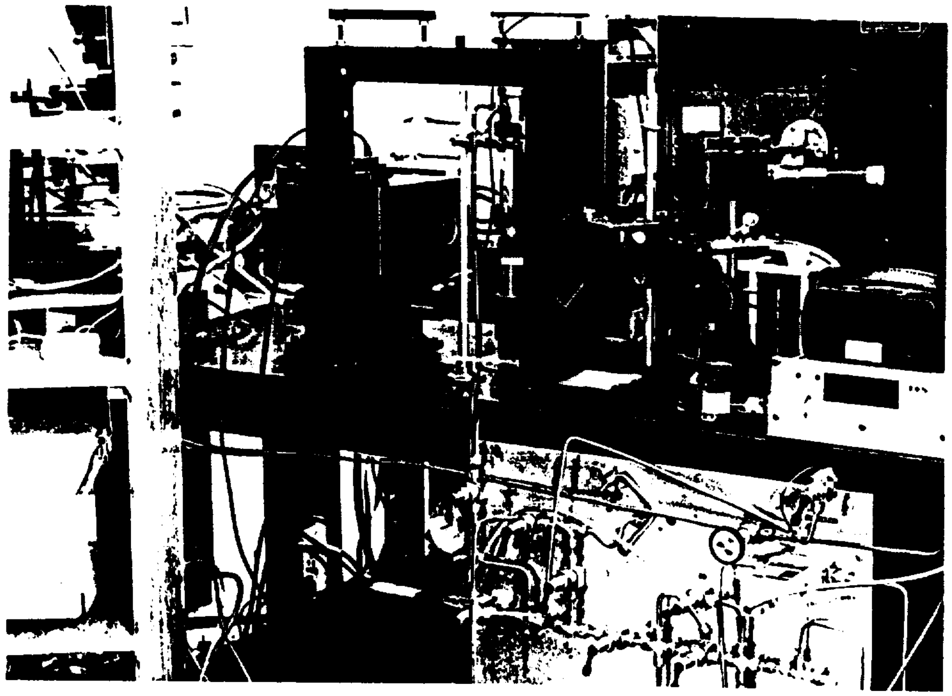


Figure 2. Photograph of experimental apparatus. Left to right: data acquisition computer, line-scan radiometer, hot specimen in vertical glass tube with laser window and gas supply, optical pyrometer, induction heater power supply, manometer and gas handling equipment.

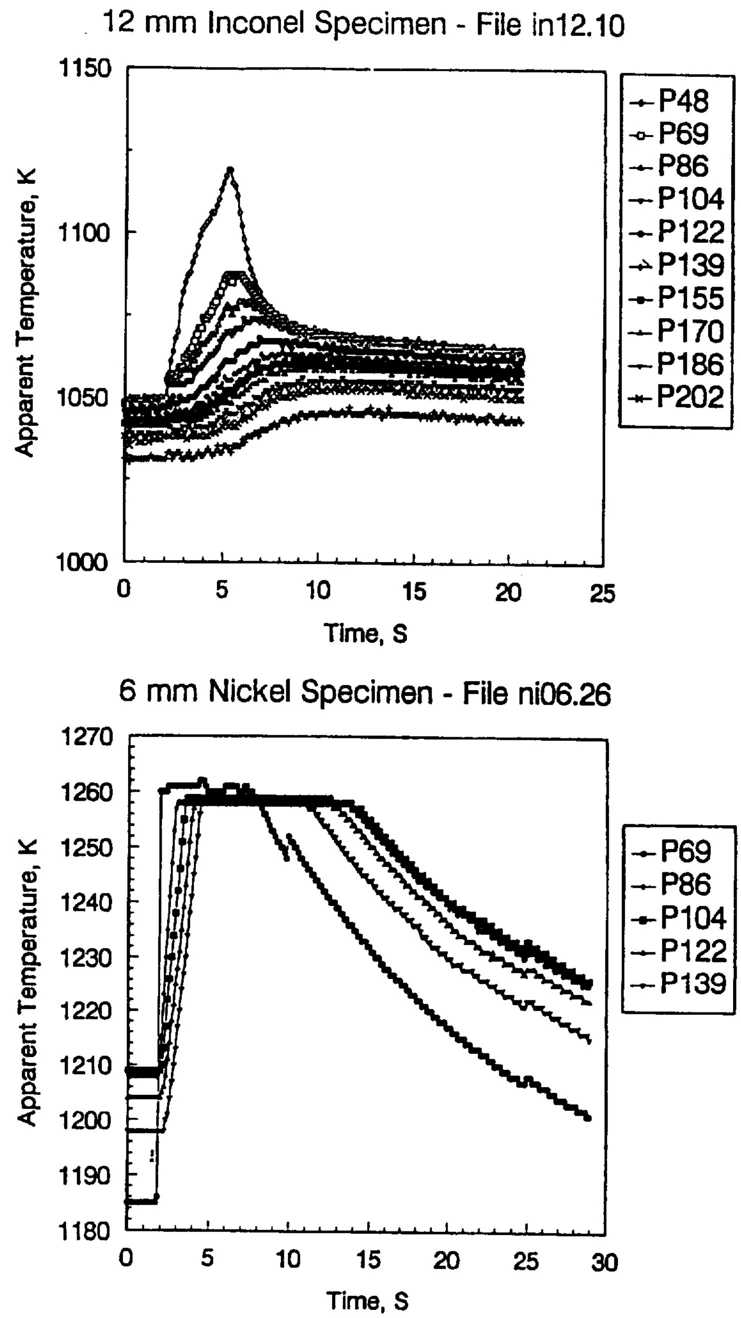


Figure 5. Temperature-time response for pulsed heating experiments on a 1.2 cm Inconel and a 0.6 cm diameter nickel sphere. The plots show the normalised temperature determined from the calibration pixels as a function.

CONCLUSIONS

- ☞ In order to determine the thermal diffusivity of high-temperature materials, an extended single-step containerless flash technique which is applicable to levitated spherical specimen is proposed.

- ☞ The thermal diffusivity is determined by knowing the sample diameter and recording the temperature rise at least at two different points on surface of the sphere simultaneously.

- ☞ The main advantage of this method is that the thermal diffusivity can be determined without the necessity of knowing heat losses at the surface. In addition, the limiting stringent specimen size requirements associated with similar methods is no longer applicable.

NEXT PHASE

- ☞ Extend the theoretical linearized analysis to other liquid droplet shapes (e.g. ellipsoid of revolution)
- ☞ Solve the "Inverse Problem" to determine thermal diffusivity
- ☞ Explore Marangoni (surface-tension-driven) convection effects

Session IV

Presentations of Opportunity