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other crystals (see Fig. 1). There is reason, therefore, to propose that the 0.22-ev center, when present as a compensated acceptor, is an efficient scatterer, particularly at low temperatures.

The marked absence of the high density of 0.2-ev trapping levels found so consistently in high-resistivity *n*-type GaAs grown without intentionally added impurity[1] (see also reference 19) suggests that these levels are associated with an imperfection which is not present in the final GaAs:Cu crystals.

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Flash Method of Determining Thermal Diffusivity, Heat Capacity, and Thermal Conductivity*

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A flash method of measuring the thermal diffusivity, heat capacity, and thermal conductivity is described for the first time. A high-intensity short-duration light pulse is absorbed in the front surface of a thermally insulated specimen a few millimeters thick coated with camphor black, and the resulting temperature history of the rear surface is measured by a thermocouple and recorded with an oscilloscope and camera. The thermal diffusivity is determined by the shape of the temperature versus time curve at the rear surface, the heat capacity by the maximum temperature indicated by the thermocouple, and the thermal conductivity by the product of the heat capacity, thermal diffusivity, and the density. These three thermal properties are determined for copper, silver, iron, nickel, aluminum, tin, zinc, and some alloys at 22°C and 135°C and compared with previously reported values.

INTRODUCTION

THERE has been a renewed interest in developing new methods of determining the thermal conductivity and the thermal diffusivity of materials in recent years. This is largely a result of the rapid advances of materials technology and the many new applications of materials at elevated temperatures. There are a number of presently existing steady-state and non-steady-state methods of measuring these parameters. However, there is some dissatisfaction with the length of time required to make reliable measurements, and in some cases, the large sample sizes required by these techniques impose intolerable limitations. The difficulty of extending these methods to high temperatures has proven to be a stumbling block in high-temperature technology.

The heat flow equation can be solved for a wide variety of boundary conditions, and these solutions can often generate values of the thermal properties. However, inability to satisfy the boundary conditions has led to difficulties in some of the classical techniques. Two of these difficulties are caused by surface heat losses and thermal contact resistance between the specimen and its associated heat sources and sinks. The problem of thermal contact resistance has been virtually eliminated in some recent thermal diffusivity determinations by thermally insulating the specimen and introducing the heat by an arc image furnace. A system of this type has been described by Butler and Inn[1] in which the thermal diffusivity is expressed in terms of the differences between the temperature versus time curves taken by thermocouples located at two points along a thermally insulated rod continuously irradiated at the front surface by a carbon arc. It has been suggested[2] that the Angstrom method, which utilizes a periodic front surface temperature variation for diffusivity measurements, can also be adapted to the arc image furnace. It is necessary to make these two types of determinations in a vacuum chamber in order to eliminate convective heat losses. However, above 1000°C the radiation losses create a problem of considerable magnitude.

The technique described in this report utilizes a flash tube to eliminate the problem of the thermal contact resistance, while the heat losses are minimized by making the measurements in a time short enough so that very little cooling can take place. Although this method has only been tested for metals in the vicinity of room temperature, there is no reason to believe that measure-

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[1] C. P. Butler and E. C. Y. Inn in *Thermodynamic and Transport Properties of Gases, Liquids and Solids* (American Society of Mechanical Engineers, 29 W. 39th St., New York, New York, 1959).

[2] A. Hirschman, W. L. Derksen, and T. I. Monahan, "A Proposed Method for Measuring Thermal Diffusivity at Elevated Temperatures," Armed Forces Special Weapons Project Report, AFSWP-1145, Material Laboratory, New York Naval Shipyard, April, 1959.

ments cannot be made for all types of solid materials in any temperature range simply by preheating or cooling the specimens.

THEORY OF THE METHOD

If the initial temperature distribution within a thermally insulated solid of uniform thickness L is $T(x,0)$, the temperature distribution at any later time t is given by Carslaw and Jaeger[3] as

$$T(x,t)=\frac{1}{L}\int_0^L T(x,0)dx+\frac{2}{L}\sum_{n=1}^{\infty}\exp\left(\frac{-n^2\pi^2\alpha t}{L^2}\right)\times\cos\frac{n\pi x}{L}\int_0^L T(x,0)\cos\frac{n\pi x}{L}dx, \quad (1)$$

where α is the thermal diffusivity in cm²/sec. If a pulse of radiant energy Q cal/cm² is instantaneously and uniformly absorbed in the small depth g at the front surface $x=0$ of a thermally insulated solid of uniform thickness L cm, the temperature distribution at that instant is given by

$$T(x,0)=Q/DCg \quad \text{for} \quad 0<x<g$$

and

$$T(x,0)=0 \quad \text{for} \quad g<x<L.$$

With this initial condition, Eq. (1) can be written as

$$T(x,t)=\frac{Q}{DCL}\left[1+2\sum_{n=1}^{\infty}\cos\frac{n\pi x}{L}\frac{\sin(n\pi g/L)}{(n\pi g/L)}\times\exp\left(\frac{-n^2\pi^2}{L^2}\alpha t\right)\right], \quad (2)$$

where D is the density in g/cm³ and C is the heat capacity in cal/g°C. In this application only a few terms will be needed, and since g is a very small number for opaque materials, it follows that $\sin n\pi g/L\approx n\pi g/L$. At the rear surface where $x=L$, the temperature history can be expressed by

$$T(L,t)=\frac{Q}{DCL}\left[1+2\sum_{n=1}^{\infty}(-1)^n\exp\left(\frac{-n^2\pi^2}{L^2}\alpha t\right)\right].$$

Two dimensionless parameters, V and ω can be defined

$$V(L,t)=T(L,t)/T_M \quad (4)$$

$$\omega=\pi^2\alpha t/L^2. \quad (5)$$

T_M represents the maximum temperature at the rear surface. The combination of 3, 4, and 5 yields

$$V=1+2\sum_{n=1}^{\infty}(-1)^n\exp(-n^2\omega). \quad (6)$$

Equation (6) is plotted in Fig. 1.

[3] H. S. Carslaw and J. C. Jaeger, *Conduction of Heat in Solids* (Oxford University Press, New York, 1959), 2nd ed., p. 101.

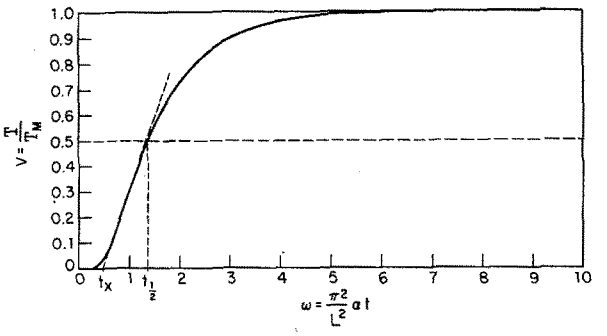

FIG. 1. Dimensionless plot of rear surface temperature history.

Two ways of determining α have been deduced from Eq. (6) and Fig. 1. When V is equal to 0.5, ω is equal to 1.38, and so

$$\alpha=(1.38L^2/\pi^2 t_{\frac{1}{2}}), \quad (7)$$

where $t_{\frac{1}{2}}$ is the time required for the back surface to reach half of the maximum temperature rise.

The time axis intercept of the extrapolated straight line portion of the curve in Fig. 1 is at $\omega=0.48$, which yields another useful relationship,

$$\alpha=(0.48L^2/\pi^2 t_x), \quad (8)$$

where t_x is the time axis intercept of the temperature versus time curve.

It is not necessary to know the amount of energy absorbed in the front surface in order to determine the thermal diffusivity. However, this quantity must be determined if measurements of specific heat or thermal conductivity are required. The product of the density and the heat capacity of the material is given by

$$DC=Q/LT_M, \quad (9)$$

and the thermal conductivity is found from the relationship

$$K=\alpha DC. \quad (10)$$

The foregoing treatment has neglected the variation of thermal diffusivity with temperature. Although the method produces an effective value of diffusivity for the sample, an effective value of the corresponding temperature is yet to be determined. This problem is common to all types of diffusivity measurements and is usually minimized by the fact that the range of temperatures in a single measurement is kept as small as possible. Clearly, the time of transit of the heat pulse will depend upon the range of temperature encountered en route. Without attempting a rigorous analysis, an effective temperature was simply picked as the time average of the mean of the front and back surface temperatures up to the time that the rear surface reaches one-half of its maximum value.

The dimensionless parameter $V(L,t)$ at the rear surface was given by Eq. (6). The dimensionless parameter $V(0,t)$ at the front surface obtained in a similar manner

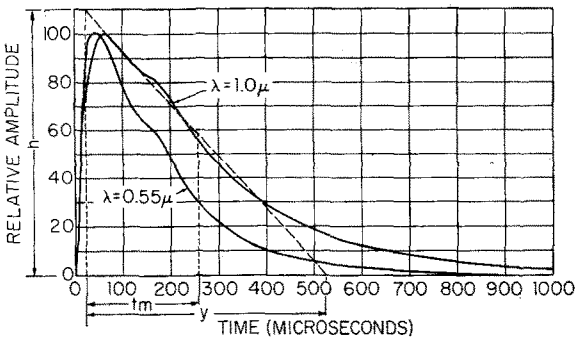

FIG. 2. Irradiance history of the flash lamp at two different wavelengths. Dashed lines represent the approximation used for the front surface temperature rise calculations.

is given by

$$V(0,t)=1+2\sum_{n=1}^{\infty}\exp(-n^2\omega), \tag{11}$$

and the mean value of $V(L,t)$ and $V(0,t)$ is

$$\frac{V(0,t)+V(L,t)}{2}=1+2\sum_{n=1}^{\infty}\exp(-4n^2\omega), \tag{12}$$

and the effective value of V is

$$V_e=1+\frac{2}{\omega_{\frac{1}{2}}}\int_0^{\omega_{\frac{1}{2}}}\sum_{n=1}^{\infty}\exp(-4n^2\omega)d\omega, \tag{13}$$

where $\omega_{\frac{1}{2}}=\pi^2\alpha t/L^2=1.38$,

$$V_e=1+\frac{2}{4(1.38)}\left[\sum_{n=1}^{\infty}\frac{1}{n^2}(1-\exp(-4n^2 1.38))\right]=1.6, \tag{14}$$

and finally, the effective temperature is given by

$$T_e=V_eT_M=1.6T_M. \tag{15}$$

MAXIMUM FRONT SURFACE TEMPERATURE

The front surface temperature may initially rise to a very high value because the energy is delivered in a very short time. It is important to know the upper limit of this temperature in order to avoid operating through temperature regions which may contain phase changes in the material being tested. An estimate of this temperature can be obtained from the following model. Consider the shape of the input power versus time curve to be a negative-going saw tooth given by $h(1-t/y)$ where the constants h and y are obtained by the construction shown in Fig. 2, and t is actually measured from the leading edge of the constructed triangle. Because the back surface temperature does not begin to rise within the duration of the input pulse, the specimen can be treated as a slab of infinite thickness. Carslaw and Jaeger[4] treat the case of an input power function $f(t)$ incident on the face, $x=0$, of a slab of infinite thickness and find that the temperature is given by

$$T(x,t)=\frac{1}{DC(\pi\alpha)^{\frac{1}{2}}}\int_0^t f(t-z)\exp(-x^2/4\alpha z)dz/z^{\frac{1}{2}}, \tag{16}$$

where z is simply a variable of integration, and so at the front surface of the specimen, irradiated by the flash lamp, this temperature is given by

$$T(t)=\frac{h}{DC(\pi\alpha)^{\frac{1}{2}}}\int_0^t\left[1-\frac{t-z}{y}\right]dz/z^{\frac{1}{2}}. \tag{17}$$

At t_m, the time of the maximum front surface temperature, $\partial T/\partial t=0$. Direct differentiation of Eq. (17) yields by Leibnitz' Rule

$$\frac{\partial T}{\partial t}=\frac{h}{DC(\pi\alpha)^{\frac{1}{2}}}\left[\frac{-1}{y}\int_0^{t_m}dz/z^{\frac{1}{2}}+t_m^{-\frac{1}{2}}\right]=0, \tag{18}$$

and so $t_m=y/2$. The maximum temperature T_f reached at the front surface is then given by combining Eqs. (17) and (18).

$$T_f=\frac{h}{DC(\pi\alpha)^{\frac{1}{2}}}\int_0^{y/2}\left[\frac{1}{2}+\frac{z}{y}\right]dz/z^{\frac{1}{2}}=\frac{4hy}{3DC(2\pi\alpha y)^{\frac{1}{2}}}. \tag{19}$$

If all of the energy of the pulse was represented by the area of the constructed triangle, Q would be equal to $\frac{1}{2}hy$. Since this is not the case, a correction factor β is introduced which is equal to the ratio of the area of the total pulse to that of the triangle.

$$Q=\tfrac{1}{2}\beta hy. \tag{20}$$

Taking Eq. (20) into account, Eq. (19) may be rewritten

$$T_f=\frac{8Q}{3\beta DC(2\pi\alpha y)^{\frac{1}{2}}}, \tag{21}$$

or since $Q=DCLT_M$,

$$T_f=\frac{8}{3\beta(2\pi y)^{\frac{1}{2}}}LT_M/\alpha^{\frac{1}{2}}. \tag{22}$$

The parameters β and y are characteristic of the flash lamp, and for the lamp used in this work they were estimated from the irradiance versus time curve at 1 μ to be 1.3 and 5×10^{-4}, respectively. A closer approximation would be obtained if the total radiant power curve could have been used instead of the power in a single wavelength band. The maximum temperature rise in any of the samples tested in this report can be estimated from Table I and Eq. (23).

$$T_f=38LT_M/\alpha^{\frac{1}{2}}, \tag{23}$$

where L is in centimeters, α is in centimeters squared

[4] H. S. Carslaw and J. C. Jaeger, *Conduction of Heat in Solids* (Oxford University Press, New York, 1959), 2nd ed., p. 76.

TABLE I. Thermal diffusivity.

Material	Alloy	Sample size cm	L Thickness cm	Thermal diffusivity[a] $cm^2\ sec^{-1}$ at 22°C	Thermal diffusivity[b] $cm^2\ sec^{-1}$ at 22°C	Thermal diffusivity[a] $cm^2\ sec^{-1}$ at 135°C	α Other sources $cm^2\ sec^{-1}$ at 20°C	T_M Final sample temperature rise °C
Aluminum	...	1.9 cm square	0.352	0.94	0.86	0.89	0.86[c]	2.4
Copper	OFHC	1.9 cm square	0.312	1.15	1.07	1.04	1.14[c]	1.8
Iron	Armco	1.9 cm square	0.100	0.19	0.18	0.15	0.17[c]	4.3
Nickel	...	1.26 cm round	0.100	0.16	0.15	0.14	0.16[c]	3.9
Silver	...	1.9 cm square	0.322	1.61	1.42	...	1.70[c]	2.5
Tin	...	1.9 cm square	0.306	0.39	0.35	0.33	0.38[c]	3.7
Zinc	...	1.9×1.16 cm rectangle	0.282	0.40	0.45	0.37	0.41[c]	2.7
Magnesium	HK31	1.9 cm square	0.352	0.54	0.48	0.57	0.56[d]	2.8
Steel	1020	1.9 cm square	0.100	0.14	0.13	0.13	0.15[d]	4.5
Steel	4340	1.9 cm square	0.107	0.096	0.10	0.090	0.091[d]	4.1
Titanium	6AL-4V	1.9 cm square	0.100	0.019	0.027	0.02	0.019[d]	6.0

[a] Obtained from Eq. (7).
[b] Obtained from Eq. (8).
[c] Taken from reference 5.
[d] Interpolated or extrapolated from reference 1.

per second, and T_M is the maximum back surface temperature.

EXPERIMENTAL PROCEDURE

The success of the method depends upon adequately meeting the boundary conditions of the theory. The front surface of the sample must be uniformly irradiated in a time short compared to the rise time of the back surface temperature, the thermocouple must measure the actual back surface temperature, and all losses must be as small as possible. Furthermore, the signal must be large enough to be well above the noise level present in the recording system, and the bandwidth of the amplifier and recorder must be wide enough to pass the signal without distortion.

The dimensions and shapes of the samples used are listed in Table I, along with the data obtained. The flash tube used was a commercially available unit (GE FT524) consisting of a four-turn quartz spiral with a Pyrex envelope, dissipating 400 joules of energy in each flash. The relative irradiance versus time curve for this lamp is shown in Fig. 2. The front surfaces of the samples were blackened with camphor black to increase the amount of energy absorbed, to ensure that all parts of the sample had equal absorption, and that the absorptivities of all specimens were identical. These samples were mounted in a ceramic holder approximately 1 cm from the envelope of the flash lamp with the plane of the front surface parallel to the axis of the quartz spiral and with 0.12-mm diam Chromel-Alumel thermocouple wires pressed against the back surface. The wires were separated by 1 to 2 mm where they contacted the surface so the electrical circuit was through the sample under test. This ensured that the temperature recorded was actually that at the back surface and not at some other wire junction. The output voltage from the thermocouple was presented on an oscilloscope and photographed with a Polaroid land camera.

The maximum signal voltages were from 40 to 400 μ, indicating a back surface temperature rise of 1° to 10°C, and were too low to record directly on the oscilloscope, which had a maximum gain of 1 mv/cm. A differential transistor preamplifier was therefore designed and built. This preamplifier had a gain of 36 and was very quiet, stable, and linear under the conditions of operation. The two transistors were mounted in a block of brass to ensure that they would both be at the same temperature, but no other attempt was made to stabilize the gain against temperature changes, as the unit was to be used only in the laboratory. The gain was measured so that the sample temperature rise could be determined, but it does not enter into any of the other calculations. The sample holders must be opaque to prevent any irradiation of the back surface of the sample, of low thermal conductivity to reduce edge losses, physically strong, and capable of standing the high irradiant thermal flux. The holder used was machined from lava and baked at 1100°C to give a hard ceramic material of considerable strength. The holder would accomodate samples 0.7 to 1.9 cm round or square and up to 0.5 cm thick, holding them in place with small spring wire retainers which obscured very little of the front surface. The thermocouple wires were clamped in a small pin vise and connected to a firmly mounted plug to simplify the connection to the external circuit. This plug formed the cold junction. The thermocouple support and the ceramic piece could be readily moved relative to each other, which considerably facilitated sample changing. The entire assembly of lamp and sample holder was mounted with conventional laboratory clamps on an inexpensive optical bench, which was used primarily because it provided a firm and solid support. The system is shown in a pictorial schematic in Fig. 3.

The 135°C measurements were obtained by heating

the sample holder and sample with an infrared lamp. The output of the thermocouple was read on a millivolt meter during the heating process, which took 20–30 min to reach equilibrium. The cold junction and the flash lamp were air-cooled during this time.

The high voltage trigger pulse and the large current pulse which accompany the discharge of a flash lamp caused some electrical interference with the signal and required careful shielding of the wires and of the lamp, except for a small window through which the sample was exposed. It was not possible to completely eliminate the transient, but it was reduced to a point where it did not interfere with the amplifiers and to where it actually provided a convenient time zero point from which to measure. Because it proved to be convenient to have a length of baseline to assist in lining up the reading instrument, a time delay generator was designed and built which fired the lamp from 0.3 to 4700 msec after the oscilloscope had been triggered.

The minimum thickness of the sample is determined by the requirement that the flash duration must be short compared to the time the temperature begins to rise at the back surface. A sample that is too thin results in the recording of a low value of diffusivity. On the other hand, if the sample is too thick, the sensitivity is reduced and the time for losses to occur is increased. Using the flash lamp described previously, a satisfactory thickness is about 1 mm for samples of diffusivity less than 0.2 cm^2/sec and about 3 mm for samples of higher diffusivity.

To facilitate the reading of the Polaroid land prints, the device shown in Fig. 4 was constructed of $\frac{1}{16}$-in. Plexiglas. The vertical arms pivot about the points labeled A, maintaining the horizontal arms parallel to and equidistant from the engraved center line. In practice, the device is laid on top of the print, the lower horizontal arm is lined up on the base line, the upper arm at the top of the curve, and the fiducial on the zero point of the curve. The half-time is read from the center line in units of distance and can be converted to time by a simple multiplication involving the sweep speed of the oscilloscope. In Fig. 4 a temperature time curve is superimposed on the device to illustrate its use. The vertical scales are to assist in aligning the print accurately with the baseline.

Heat capacity measurements were made by comparing the temperature rise in the sample with that in a silver sample of similar shape and size. The values of heat capacity for silver and of the density for all the samples of the elements were taken from reference 5.

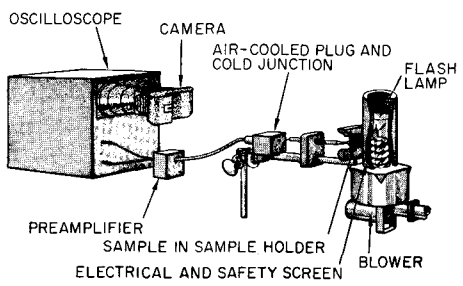

FIG. 3. Pictorial schematic of test setup.

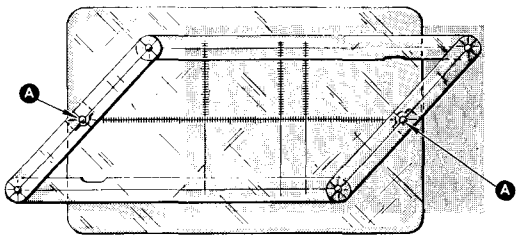

FIG. 4. Reading device.

The shape of the temperature versus time curve, if compared with Fig. 1, will reflect any appreciable deviations from the ideal boundary conditions of the experiment. If the final temperature is approached very slowly after an initially fast rise, or if the curve actually has a peak before seeking a lower temperature plateau, there was a nonuniform distribution of irradiance on the front surface. The latter effect will appear if the supporting structure masks an appreciable portion of the surface. The distortion in the curve is due to two dimensional heat flow which is slow because of the greater distances, and usually has a small effect on the early part of the curve.

A detailed analysis of the effect of cooling losses and the circumstances for which they can be neglected has not been accomplished in this report, but will be part of the basis for further work in connection with extending the flash technique to higher temperatures. The appearance of a flat region at the top of all of the data curves was an indication that cooling losses were not important in this case. Corrective measures to be taken if cooling is excessive would be to use thinner samples or to perform the experiment in a vacuum.

ERRORS AND ACCURACY

The largest errors in the system are probably the nonlinearity and distortion in the oscilloscope-camera recording system, and nonuniformity in the distribution of the radiant energy on the sample surface. The system layout was chosen to minimize the latter effect and careful calibration was used to minimize the former. The final precision of measurement was within $\pm 5\%$ and is about as well as can be done with the recording system that was used. (Tektronix 531 oscilloscope, Fairchild oscilloscope camera with a Polaroid land back.) The accuracy of the system is more difficult to estimate. Tables I and II and Fig. 5 compare the data obtained with some previously published.[3,5] The dotted lines in Fig. 5 represent a difference in the two figures of $\pm 10\%$, and it can be seen that the agreement is within

[5] *Smithsonian Physical Tables*, compiled by W. E. Forsythe (The Smithsonian Institution, Washington, D. C., 1954), ninth revised ed.

TABLE II. Thermal conductivity and heat capacity (same samples as used for Table I).

Material	Alloy	D Density g cm^{-3} 20°C	C Heat capacity cal g^{-1} °K^{-1} 22°C	K Thermal conductivity cal cm^{-1} sec^{-1} °K^{-1} 22°C	C Other sources cal g^{-1} °K^{-1}	K Other sources cal cm^{-1} sec^{-1} °K^{-1}
Aluminum	...	2.70[a]	0.21	0.53	0.23[a]	0.50[a]
Copper	OFHC	8.96[a]	0.097	1.0	0.092[a]	0.92[a]
Iron	Armco	7.87[a]	0.11	0.16	0.11[a]	0.17[a]
Nickel	...	8.90[a]	0.12	0.16	0.11[a]	0.14[a]
Silver	...	10.49[a]	0.056[a]	0.95	0.056[a]	1.00[a]
Tin	...	7.30[a]	0.057	0.16	0.054[a]	0.15[a]
Zinc	...	7.14[a]	0.088	0.25	0.090[a]	0.27[a]
Magnesium	HK31		0.428[b]	0.23	...	...
Steel	1020		0.824[b]	0.12	...	...
Steel	4340		0.833[b]	0.08	...	...
Titanium	6AL-4V		0.614[b]	0.012	...	...

[a] Taken from reference 5.
[b] Density not available. Product of D and C calculated and listed.

these limits in practically all cases. Differences of this magnitude are found in data published by other investigators[3,5,6] and may represent differences in sample purity as well as methods since the parameters reported here are likewise affected by the physical treatment of the metals as well as by the composition.

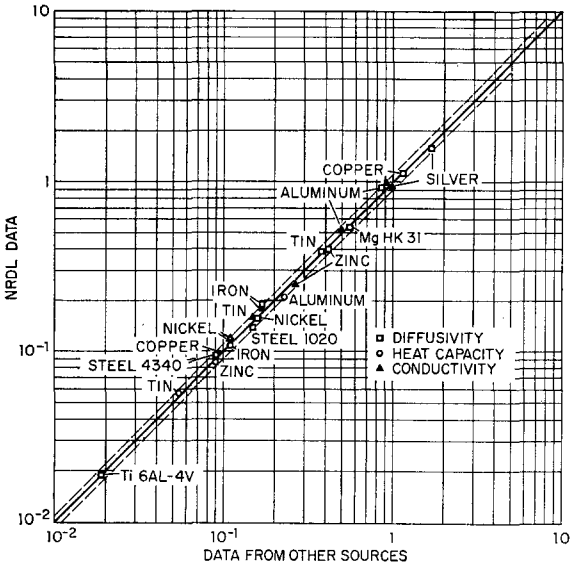

FIG. 5. Graphic comparison of NRDL data and data from other sources at ambient temperatures. The broken lines represent a ±10% variation from the mean.

[6] *ASME; Metal Properties*, edited by S. L. Hoyt (McGraw-Hill Book Company, Inc., New York, 1954).

The values of diffusivity determined by Eq. (7) are considerably less precise than those determined by Eq. (8). This method requires the finding, by eye, of the straight portion of a curve and the extrapolation of this line back to the baseline. This is a subjective method which is rather difficult and one in which a small error in the slope determination results in a relatively large error in the value of the diffusivity. Its advantage is that it is independent of the final height of the curve and does not require that the surface distribution of energy be as uniform as does the half-time method.

CONCLUSIONS

The results shown in Tables I and II indicate the very close agreement between the values measured here and those already published. It is a new system and the results presented are only a preliminary test of the basic method in the ambient and slightly above ambient temperature range and for materials for which values of the thermal properties can be found in the literature.

There are several advantages associated with this system. (1) A minimum of specialized equipment is required. (2) Data reduction is relatively easy. (3) The size of the specimen can be quite small. (4) The system can be used at high or low temperatures by preheating or cooling the specimen. (5) The amount of energy added to the sample to make a measurement is quite low. This should be an attractive feature for low-temperature measurements. (6) The three thermal properties, diffusivity, thermal conductivity, and heat capacity, can be deduced for the same sample with the same equipment.