

## CHARACTERIZATION AND THERMAL PROPERTIES EVALUATION OF THIN FILMS, WAFERS AND SUBSTRATES

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### ABSTRACT

Materials in use or under consideration for many applications in new and emerging technologies are often available only in small quantities and many times in the form of thin films, wafers and sheets. Such size and form limitations present a number of challenges to those wishing to evaluate thermal performance characteristics. This has resulted in a need to develop totally new transient or modify current transient and steady state techniques.

Various new or modified techniques to measure the thermal diffusivity, thermal conductivity, specific heat and thermal expansion over broad temperature ranges will be described. Illustrations of applications to and results on semiconductors, superconductors, diamonds, polymers, composites and layered structures will be given and discussed.

### INTRODUCTION

The second half of the twentieth century has seen the subject of the measurement of thermal properties change from an exercise mostly of academic interest to a complex issue employing the attention of large numbers of workers in all sectors of industry, government and academia. The explosion in materials development for all of the many emerging technologies, and especially electronics, has produced very substantial quantities of new and improved but often expensive materials and composites in

different forms. This has stimulated a corresponding need for evaluation of their thermal properties more rapidly and often under conditions that cannot be satisfied using the appropriate classical methods.

Excluding cost considerations, the overall change may be summarized as being the combined result of five specific but interrelated changes:

- availability of materials only in limited quantities and forms
- applications replacing materials as the major driving force
- small "non-ideal" rather than large "ideal" specimens
- attraction of transient over steady-state measurements
- apparatus automated rather than manually operated

The past ten years can well be summarized as the "thin" decade. Materials development became one that involved sheets, wafers and films provided by various processes. This was stimulated especially by advances in semiconductors, superconductors and directional polymers combined with corresponding applications needs in electronics packaging in general and microelectronics in particular. For thermal conductivity this further reduction in the most critical specimen dimension provided new and greater problems for the experimentalist. Whereas in the previous two decades successful techniques had been developed to measure specimens of up to a few millimeters thick we were now faced with microns and Angstroms.

The present paper discusses several techniques and instruments that we have developed specifically during this time to provide reliable results of thermal conductivity, specific heat and thermal expansion on small and especially thin specimens. This discussion, while providing mostly a number of examples of measurements of thermal diffusivity and derived thermal conductivity for different applications also includes results of measurements of thermal expansion and specific heat.

## SPECIFIC HEAT

For the past two decades or more the predominant method used over a moderate temperature range has been the differential scanning calorimeter. Its attraction is that it uses only a very limited amount of material (milligram quantities) and is based on the principles of differential thermal analysis using a tool available in many laboratories.

Although it is in very wide use there are some drawbacks to its general use particularly for composites and other heterogeneous materials where small quantities of specimen are not representative of the bulk material. Furthermore, the results obtained on different forms (powder, granular, solid, etc) of the same material are often variable and dependent on heating rate and are often not sensitive enough to

quantify second and third order transitions. Other techniques are necessary to supplement DSC for measurements on small specimens.

### A.C. Calorimeter

One method that has been developed, as a very sensitive calorimetric tool and particularly useful for materials in the form of thin films and sheets is ac calorimetry (Kraftmakher, 1962). Figure 1 illustrates schematically the principle on which the ACC calorimeter is based. The front surface of a specimen is irradiated by light from a halogen lamp chopped at frequency,  $f$ , of usually between 0.01 and 30 Hz. By using a specially designed lock-in amplifier the temperature amplitude  $T_{ac}$  is measured with high sensitivity on the rear surface of the specimen.

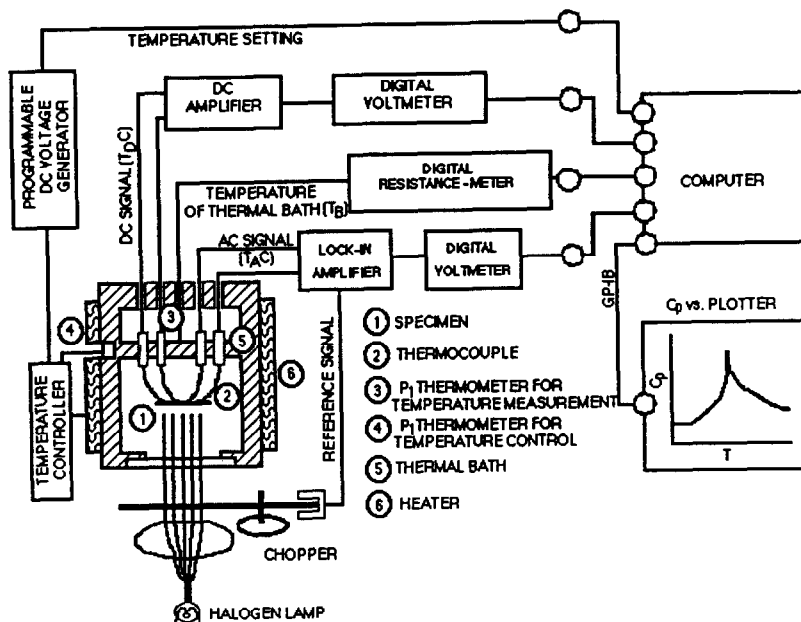


Figure 1. Schematic of the principle of ac calorimetry.

The heat capacity  $C$  is given by:

$$C = \frac{Q}{\omega T_{ac}} \quad [1]$$

Where  $Q$  is the heat flow during one cycle,  $\omega$  the frequency and  $T_{ac}$  is an amplitude of temperature variation.

Absolute values can be obtained if  $T_{ac}$  values are measured for a specimen(s)

of known specific heat, e.g. sapphire, and the slopes of the plots of  $1/T_{ac}$  vs frequency compared. However, the technique is more valuable for determining changes in heat capacity e.g. due to phase transitions, over a very small temperature range. Since the actual rise in temperature during a measurement is only milliKelvins the conditions may be described as stationary or quasi-adiabatic. The sensitivity is some  $10^2$  to  $10^3$  greater than that obtained by DSC measurements.

In this measurement and that of thermal diffusivity, as discussed later, the thickness of the specimen has to be less than the thermal diffusion length which is given by  $(2D/\omega)^{1/2}$ . In the measurement a small specimen of the order of a maximum of 4mm x 4mm x 0.1 to 0.3mm for absolute values and 2mm x 2mm by 0.01mm for relative values is supported inside the furnace by two 25 $\mu$ m thermocouple wires welded together and attached with a silver paste. Helium gas is introduced in the specimen cell to maximize heat transfer between the specimen and surroundings.

This precise high sensitivity apparatus has been used for a variety of applications particularly in studying minute phase transitions (Hatta and Ikushima, 1981). Materials that have been studied include polymers, liquid crystals, dielectric and superconductive ceramics. The latter studies have been made at temperatures below 90K with a modified system using liquid helium. (Kishi and Ishikawa, 1988), (Ishikawa, 1988)

The first of these was particularly useful in that it not only provided values of specific heat for YBCO, type ceramics but more importantly showed that specific heat measurement by this method was an invaluable tool to investigate material quality. Furthermore, it was more sensitive than techniques such as electrical resistivity or ac susceptibility.

Following measurements on over fifty specimens it was found that both the specific heat jump  $\Delta C_p/T_c$  at  $T_c$  but also the shape of the  $C_p$  v  $T$  curve provided important information on the material quality. The authors divided the superconducting specimens into four groups A to D; those of high, fair, deteriorating and poor quality. For group A a clear jump existed with  $\Delta C_p/T_c$  exceeding 40mJ/mol $\cdot$ K<sup>2</sup>; for Group B the value was between 20 and 40 mJ/mol $\cdot$ K<sup>2</sup>. Group C did exhibit superconductivity but the  $C_p$  behavior did not show a clear jump due to effects of moisture or other parameter. Group D did not exhibit any anomalous behavior and were not superconducting.

In the second study carried out on four specimens fabricated from sintered fine 4N powders of Y<sub>2</sub>O<sub>3</sub>, BaCO<sub>3</sub> and CuO four times between 880 and 930°C followed by oxygen annealing below 600°C, the expected sharp single peak transition having a  $\Delta C_p/T_c$  value in excess of 50 mJ/mol $\cdot$ K<sup>2</sup> was found for three specimens. However, for one specimen, distinct double peaks separated by a 3K interval were observed,

indicating the existence of a second superconducting phase akin to the orthorhombic phase in the surface layers. Independent electrical resistivity and magnetic susceptibility measurements confirmed the existence of this second superconducting phase.

The ac calorimeter technique has also been used to measure the heat capacity, particularly the temperature dependence, of very small volumes of fluids in form of a thin film between two plates (Smaarduk and Mochel, 1978) A major disadvantage to this method is that the absolute value is less easy to measure since the heat capacity contribution of the metal plates dominates the total heat capacity since the specimen thickness has to be less than the thermal diffusion length. Furthermore, it is difficult to maintain exactly the same thickness dimensions on changing the test specimen to the reference.

An alternative new approach has been to contain the specimen (<1 $\mu$ l volume) in a very fine steel hypodermic tube, 130  $\mu$ m internal diameter and 170  $\mu$ m external, such that the specimen dimensions and conditions do not change during a measurement. (Yao and Hatta, 1988) Values of absolute heat capacity can be obtained by undertaking measurements on the empty cell and the cell filled with the fluid and evaluating the results in terms of heat capacity per unit length of the cell.

#### THERMAL DIFFUSIVITY/THERMAL CONDUCTIVITY

There are two widely used transient techniques for measurements of thermal diffusivity and derived thermal conductivity. In both cases it is possible to measure specific heat directly by modifying the basic technique to include measurements on a specimen(s) of known heat capacity.

##### *Transitory temperature ("Flash") method*

This method was developed originally about 1960 by Parker et al (Parker, Jenkins, Butler and Abbott 1961) for measuring the thermal diffusivity, and specific heat under certain conditions, of small, flat, uniform, homogeneous discs using a flash lamp source of energy. Since that time the basic method, now using a laser energy source, and its many variations and modifications based on a variety of models for different applications has evolved into the one that has been used for the broadest range of materials over a very wide temperature range. One only has to examine the contents of the proceedings of the many conferences and the general literature to realize the full extent of the materials and applications to which it has been addressed and the vast numbers of measurements that have been made with its use. This method has had a significant impact on the measurement of thermal diffusivity of small specimens. With this history, many workers have attempted to apply its use to very thin specimens, currently with limited success for some applications.

The principle and application are well documented and need not be discussed in detail. The front face of a specimen is subjected to a short burst of radiant energy for 1ms or less. The temperature history of the rear surface is measured and the thermal diffusivity,  $D$ , computed from values of the half time (time to reach 50% of maximum) and square of thickness. Other percentage times can be used depending on the analytical model used.

The prime advantages are utilization of small disc shaped specimens of the order of 10mm diameter and 1 to 4 mm thick, depending on the thermal properties of the material, and short measurement times of much less than one second.

While this technique may be considered revolutionary it is by not means a panacea for all materials and applications. There are instances where the method has been misapplied to an inhomogeneous or highly anisotropic material. A more common occurrence has been in the mistaken application of the common relationship relating  $D$  to the thermal conductivity  $\lambda$ , volume specific heat  $C_p$  and density  $\rho$

$$D = \lambda / \rho C_p \quad [2]$$

in order to derive thermal conductivity from results. It must always be remembered that use of the term  $D$  applies only for the case where predominant heat transmission is by the solid conduction mode. It is not truly applicable to inhomogeneous materials, where other heat transmission modes, especially radiation, occur. Where this does occur care has to be taken to coat the surfaces of the transparent or semitransparent specimen with an opaque graphite or other film.

The flash method has been used for a variety of coated materials such as sprayed oxide layers of various thickness of 1mm and less on metals. Thermal conductivity is obtained using an appropriate double or multilayer analysis (Larson and Koyama, 1968). This assumes that there is no interfacial contact resistance between coating and substrate and that the properties of the substrate are known.

We have used this technique successfully for polymer films having thicknesses in excess of 0.1mm. For such materials radiation transfer was minimized by coating the front surfaces, using a carbon spray. It was found necessary to attenuate the energy from the laser source by a factor of 10 or greater to reduce the front face temperature rise to less than 5°C and to ensure that the specimen was not damaged by overheating.

Another application we have examined is one using a modified two dimensional heat flow analysis to measure larger area thinner specimens more specially in a comparative property mode. This technique can have a significant impact on future NDT evaluation of the homogeneity and uniformity for quality control and quality assurance of silicon wafers and thin layered composites used for electronic packaging.

The principle is illustrated in Figure 2. The temperature history at point,  $r$ , off-center can be derived analytically assuming that the specimen is an infinite plate; the thickness is very much less than the distance  $r$ ; and that there are no heat losses. Although the analysis indicates it is possible to derive an absolute value for thermal diffusivity the technique appears to have more potential when used in a comparative mode by using a standard specimen(s) of known properties.

Preliminary measurements have been undertaken on a silicon wafer using a previously measured stainless steel as a reference material. An overall value of thermal diffusivity of  $0.71\text{cm}^2/\text{s}$  was obtained. This is lower than accepted values of over  $0.8\text{cm}^2/\text{s}$  for silicon but is believed to be caused by variations due to imperfections found in the wafers.

#### *ac Calorimeter Method*

This recent modification of the previously discussed ac calorimeter technique was developed specifically for very thin specimens of a variety of materials and including composite layered structures (Hatta, 1985).

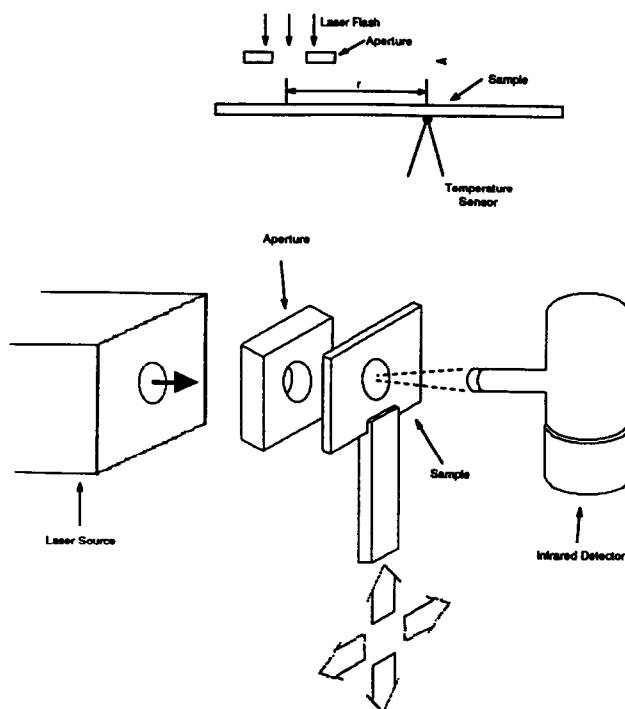


Figure 2. Thermal Diffusivity Measurement on an Infinite Plate: (a) Principle; (b) Schematic of apparatus.

The basic technique and apparatus is somewhat similar to that for the ac calorimeter. This principle is illustrated schematically in Figure 3. The optimum specimen is a 10mm x 4mm by less than 0.33mm thickness rectangle. Instead of illuminating the complete surface of the specimen, only part of it is subjected to the periodic thermal energy from a light source such as a halogen light or laser. The remainder is masked from the chopped light irradiation. The ac temperature at a position of the specimen under the mask is measured using a very fine thermocouple (25  $\mu\text{m}$  wires) attached by means of a silver paste to and supporting the specimen.

The thermal diffusivity,  $D$ , in the direction parallel to the surface of the specimen is obtained by measuring the ac temperature  $T$  as a function of distance  $x$  between the position of the sensor and the edge of the mask. In the instrument, the mask is moved end respect to a fixed specimen.

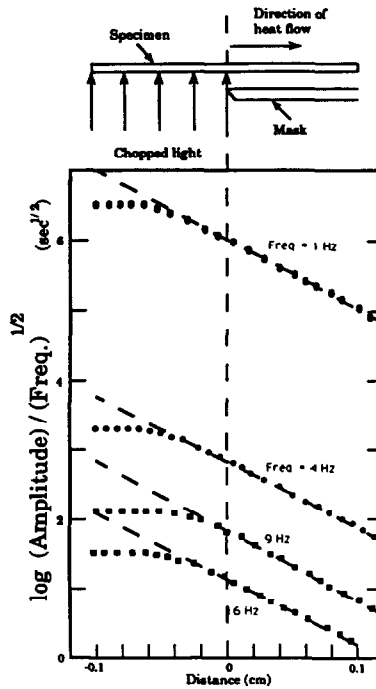


Figure 3. Cross-section view of modified ac calorimeter technique and relation of output to distance for a specimen measured at different frequencies.

The relation  $|T|$  vs  $x$  is given by:

$$|T| = \frac{Q \exp(-kx)}{2\omega c l}$$

[3]



where  $Q$  is the amplitude of the applied energy flux for unit area,  $\omega$  is the angular frequency of the periodic heating,  $c$  the heat capacity of the specimen,  $l$  the specimen thickness and  $k$  is the thermal decay constant  $(\omega/2D)^{1/2}$  or  $(\pi f/D)^{1/2}$  where  $f$  is  $(\omega/2\pi)$  the ac frequency.  $D$  can thus be obtained from the decay constant.

More recent work indicates that  $D$  for the direction perpendicular to the surface can also be determined but with lower accuracy than for the parallel direction (Axumi, Takahashi, Ichikawa, Motonari and Yano, 1987). Measurements can also be made on single wires and fibers (Hatta, Yao, Kato and Maesono 1990).

Frequently,  $k$  is called the thermal wave number and  $1/k$  the thermal diffusion length. In order to satisfy the one dimensional system condition the relationship  $kl < 0.1$  must be satisfied.

Some examples of maximum thickness at 1Hz for materials commonly used as thin films range from 0.02 mm for polymers to 0.6mm for copper and 2mm for diamond. Thus, it is seen that measurements are possible a most kinds of materials in the form of thin films and for a broad range of thermal diffusivity values. However, for low thermal diffusivity materials, such as polymers, having thicknesses below  $20\mu$ , measurements should be carried out in vacuum to reduce the conduction effect of the air layer in immediate contact with the specimen surface. This can be significant and if measurements are carried out in air the results are best analyzed in terms of a composite layer consisting of the specimen plus an air layer having a thickness proportional to the air diffusion length ( $l_a^{-1}$ ).

Table 1 contains results of test on polypropylene, glass and stainless steel films measured in vacuum and in air. The table also contains values calculated from the measurements in air assuming the specimen is a composite including the thickness of an air layer of  $0.1 l_a^{-1}$ .

TABLE 1

Results for materials tested in air and vacuum.

Specimen	Thickness ( $\mu\text{m}$ )	D, cm/s		
		Vacuum	Air	Derived
Polypropylene	17	0.00274	0.0048	0.027
Glass	55	0.00536	0.006	0.0053
Stainless Steel	55	-	0.0348	0.0345

The thermocouple itself has to be smaller than  $1/k$ , and has to be attached to the specimen in order to provide a measurable signal. It has been found that the

method of attachment does not affect the accuracy of the measurement. The specially developed lock in amplifier developed for measuring accurately the signals obtained at the low frequencies used (0.01 to 30 Hz) detects only the fundamental sine-wave component and is not affected by the periodic deformation. For thin specimens of layered composites such as a film on a substrate, providing

$$k_i l_i < 1 \quad [4]$$

where  $k_i$  is  $k$  of the  $i^{\text{th}}$  layer and  $l_i$  is the thickness of the  $i^{\text{th}}$  layer measurement of overall values of  $D$  is given by

$$D = \frac{\sum c_i D_i l_i}{\sum c_i l_i} \quad [5]$$

Practically, for a thin film having a thermal conductivity  $\lambda_i$ , thermal diffusivity  $D_1$ , volume specific heat  $c_1$  and thickness  $l_1$  deposited on a substrate of corresponding properties  $\lambda_2, D_2, c_2$  and  $l_2$  the thermal diffusivity in the direction parallel to the surface is given by

$$D_{12} = \frac{D_1 c_1 l_1 + D_2 c_2 l_2}{c_1 l_1 + c_2 l_2} \quad [6]$$

Providing  $l_1 \ll l_2$  then equation (7) becomes:

$$D_{12} = \frac{\lambda_{12} + \lambda_2 l_2}{c_{12} l_{12}} \quad [7]$$

By measuring the properties of the composite and combining them with separately measured or known properties of the substrate material it is possible to obtain the thermal conductivity of the film. Based on current experience using a halogen lamp as the energy source we have found that reliable measurements are possible providing

$$\lambda_1 l_1 \geq \lambda_2 l_2$$

Furthermore, measurements are still possible but with decreasing precision as  $\lambda_1 l_1$  approaches  $0.1 \lambda_2 l_2$ . However, for  $\lambda_1 l_1 < 0.1 \lambda_2 l_2$  the properties of the substrate dominate and measurement are not possible. Currently work underway using a laser as the energy source to see if the above limit can be reduced to below 0.1.

Some examples of the use of this technique for thin specimens are as follows:

## Metals and Ceramics

Table 2 contains results of measurements made on a selection of different materials. The table also includes results obtained using the laser flash method on 1mm thick specimens, together with recommended values from the Thermophysical Properties of Matter Volumes, published by Plenum Press.

Good agreement between the two methods can be seen and the values obtained are within the limit of those given in the basic reference source. Differences can be due to a wide variety of governing parameters such as type, purity, density, structure, etc.

## Semiconductor Superlattice Films for Devices

Superlattice films are being used for many electronic and super-optoelectronic devices to replace conventional multi-element compound semiconductors. With increasing outputs of these devices, a knowledge of their thermal property behavior is required in order to clarify and understand differences between the superlattice and bulk material.

TABLE 2

Thermal Diffusivity at 293K for Several Materials

Material	Thickness $\mu\text{m}$	D, $\text{cm}^2/\text{s}$ (at frequency Hz)					Flash Method	Reference
		1	4	9	12	16		
Ni	200			0-195 $\pm 0.006$	0-194 $\pm 0.006$	0-194 $\pm 0.006$	0.2	0.2-0.25
Si	300	0.83 $\pm 0.02$						0.82-0.95
Stainless Steel	50		0.033 $\pm 0.001$	0.033 $\pm 0.001$		0.033 $\pm 0.001$	0.033	0.032-0.037
$\text{Al}_2\text{O}_3$	100			0.075 $\pm 0.003$		0.073 $\pm 0.003$	0.078	0.068-0.090

One example that has been studied by this technique is the AlAs/GaAs superlattice film used for the clad layer of the surface oscillation laser diode and the GaAs laser (Yao, 1987).

The superlattice film used for the measurement consisted of stacked superlattices built on a substrate (subsequently removed). The thickness of each layer was kept within the range of 50 to 500 Å and the final stack had a total thickness of 10 μm. Results for thermal diffusivity vs thickness of measured layers are shown in Figure 4, together with value for GaAs and the Al<sub>0.5</sub>Ga<sub>0.5</sub>As. The values for the superlattice are lower than the average value of the individual alloys and the approach that the alloy. The values decrease as the thickness of the superlattice layer is reduced and the period of the two lattices becomes shorter.

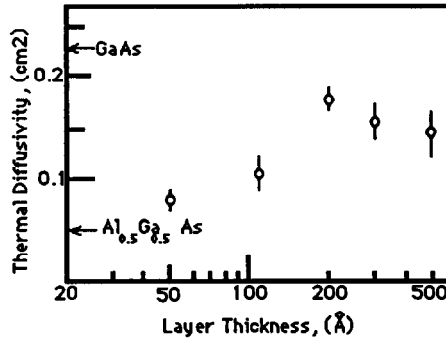


Figure 4. Variation of Diffusivity of AlAs/GaAs Superlattices with Layer Thickness.

### *Diamond Films*

Diamond films are being developed in increasing quantities by various processes (plasma CVD, sputtering etc.) for a variety of applications. Diamond has very important attributes since it is both a very good electrical insulator while having extremely high thermal conductivity (some 2 to 4 times that of copper at 293K).

One investigation on diamond films by this method (Funamoto, Nishikawa, Moriya and Sangboni 1986) studied the effects of methane concentration on the properties of the diamond film. In this 10mm by 4mm silicon wafers  $95 \pm 10\mu\text{m}$  thick were coated with plasma CVD diamond films from 5 to 30 μm thick. The results on a small 100μm long section of specimen indicated that the methane concentration had a significant effect on the crystallinity of the diamond. The crystalline structure becomes closer to that of the diamond (very high thermal diffusivity) as the concentration is decreased. Values for derived thermal conductivity of approximately 900W/mK were obtained for the specimen produced with the lowest concentration.

It should also be pointed out that thermal diffusivity and thermal conductivity are very sensitive to structure differences. Thus, the evaluation technique is not only useful for evaluating the effects of processing but also can be used for studying the existence of local variations of film quality over larger areas of a specimen.

### Optomagnetic Recording Film

Optomagnetic discs with shorter access times are currently being developed to replace both floppy and hard discs. Since the recording medium is irradiated with a laser beam of more than several MHz detailed and sophisticated thermal analysis has to be undertaken to design the recording films. Since the thermophysical properties of some materials in the form of thin films may differ from those of the bulk material (Tye, 1990) it is necessary to measure properties of the films themselves.

The method has been used (Yoneyama, Tanaka, Satoh, Takasuka and Yorozu, 1989) to measure the thermal properties of three types of films shown in Figure (5). The results were then used in a simulation analysis to determine the thermal response when irradiated with two laser pulses (3.7 MHz, pulse width 60ns, speed 5.65 m/s). It was found that structure B provided a lower central temperature for the irradiated area plus a sharper pattern than either structure A or C. This is due to the aluminum coating acting as a heat sink whereas for the other two both the upper and lower layers are low thermal conductivity. This gives rise to higher temperatures and thermal interference with the previous pulse causing a broadening pattern over a larger area.

The above are only a few illustrations of the flexibility and use of this method for a variety of needs. Other work in which we are currently involved includes measurements on ceramic coatings, on structural and electronic components, studies of changes in properties of ion-implanted materials to improve surface characteristics and of amorphous and other materials due to heat treatment.

A. structure	b. structure	c. structure
	Al 400A	
Ta <sub>2</sub> O <sub>5</sub> 800A	TA <sub>2</sub> O <sub>5</sub> 800A	AlN 800A
TbFeCo 800A	TbFeCo 800A	TbFeCo 800A
Ta <sub>2</sub> O <sub>5</sub> 800A	Ta <sub>2</sub> 5 800A	AlN 1200A
substrate (PC)	substrate (PC)	substrate (PC)

Materials	Density (g/cm <sup>3</sup> )	Specific Heat (J/g K)	Thermal Conductivity (W/m K)
TbFeCo	7.8	0.45	40.0
Ta <sub>2</sub> O <sub>5</sub>	8.8	0.40	12.9
AlN	3.8	0.40	24.9
Al	2.7	0.50	200.0
PC	1.3	0.21	0.75

Figure 5. Structure of photomagnetic recording film, and individual property values of components.

## THERMAL EXPANSION

Accurate measurements of the expansivity of specimens of limited length can be considered analogous to those on materials of very low coefficient of thermal expansion. The change in the measured dimension is very small and thus the technique used has to be one where such small changes can be measured accurately. Push rod dilatometry and thermomechanical analysis techniques are unsuitable for such measurements since the most accurate detection of change in thickness is at best the order of  $\pm 1 \mu\text{m}$ . For small specimens and low expansion materials a detection precision of better than  $0.1 \mu\text{m}$  is necessary.

Optical interferometry offers one of the best means to measure length changes to such higher orders of accuracy. In the past single beam interferometry has been used (Drottning, 1981). However, recently we have developed a dual beam automated system based on a modified Michelson interferometer which now provides twice the sensitivity (Takahashi, Azumi, Amemiya, Kato and Sekino, 1987). In addition, due to the design, the specimens require less critical attention in fabrication or machining.

The method employing a highly stabilized He-Ne gas laser and optical system is illustrated schematically in Figure 6. The test specimen is sandwiched between two mirrors which are maintained parallel to each other by a specially designed parallel springs movement mechanism with zero friction. The mirrors are made of fused silica with the surfaces partially coated with gold. The linearly polarized laser beam is split into two by the beam splitter, these go through the quarter waveplate, are reflected at the inner and outer mirrors and return so creating interference fringes as the specimen changes in length. As the specimen is heated and cooled, the fringe pattern is expanded by the negative cylindrical lens and falls on the linear image sensor which detects movement of the multippeak pattern.

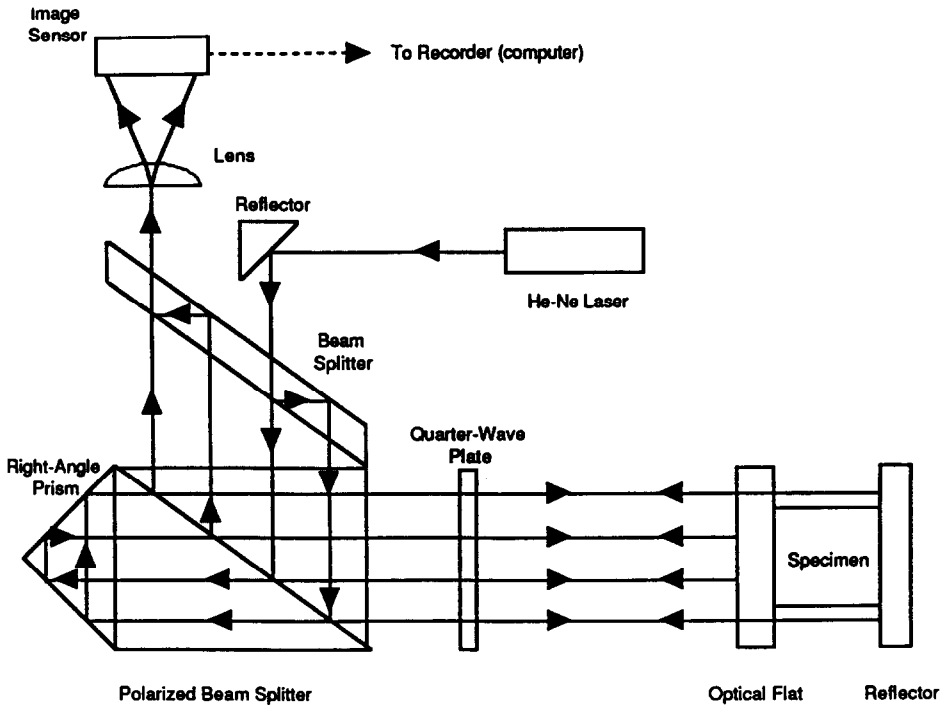


Figure 6. Optical Arrangement of the Dual Beam Interferometer for Linear Expansion Measurement.

The microcomputer analyzes this every 0.5 seconds to obtain the position of the peaks  $A_0, A_1, A_2, \dots$  and calculates the absolute positive or negative length change  $l$  (nm) using

$$l \text{ (nm)} = \frac{A_1 - A_0}{4} \cdot \frac{632.8 \text{ nm}}{1} \quad [7]$$

with the wavelength of the HeNe laser as the reference for calibration. Dimensional changes can be resolved with an absolute accuracy of  $0.02 \mu\text{m}$  ( $200\text{\AA}$ ) which is equivalent  $1/32$  of the wavelength of the gas laser.

For any measurement of thermal expansion the specimen should have as large a length dimension as practically possible for the particular measurement device. The present system is designed for specimens up to 15mm long and 6mm diameter having rounded ends as opposed to carefully machined parallel flat faces. However, it can be used for thin sheets, certainly for those less than 1mm in thickness. The

specimens are sandwiched between two glass distance pieces of known thermal expansion flat on the inner faces and rounded on the opposite ends.

The high performance of this system has been demonstrated by measurements on reference materials and more recently on two low thermal expansion glasses (Zerodur and ULE) over the approximate range 120 to 420K (Imai, Ikaji, Kishii, Sagara, Aikawa and Kato 1990). In this latter case an intercomparison of three interferometer techniques was organized by the New Glass Forum in Japan in order to ensure that measured values on low expansion functional materials used by the electronics and space industries were comparable to the same order of precision. In this study, the reproducibility of measurement of the linear coefficient by this technique was  $\pm 3$  and  $\pm 3.8 \times 10^{-8}/K$  for the two glasses. The overall uncertainty from the study was  $\pm 4 \times 10^{-8}/K$ .

It can be used for thin materials, as shown in Figure 7. A beryllium sheet approximately 0.3mm thick was heated to over 570K and cooled. A shrinkage at approximately 550K can be seen. This is due to the precipitation of  $\beta$  phase in the supersaturated  $\alpha$  phase to form a stable mixed  $\alpha+\beta$  phase causing a permanent change in thickness on cooling.

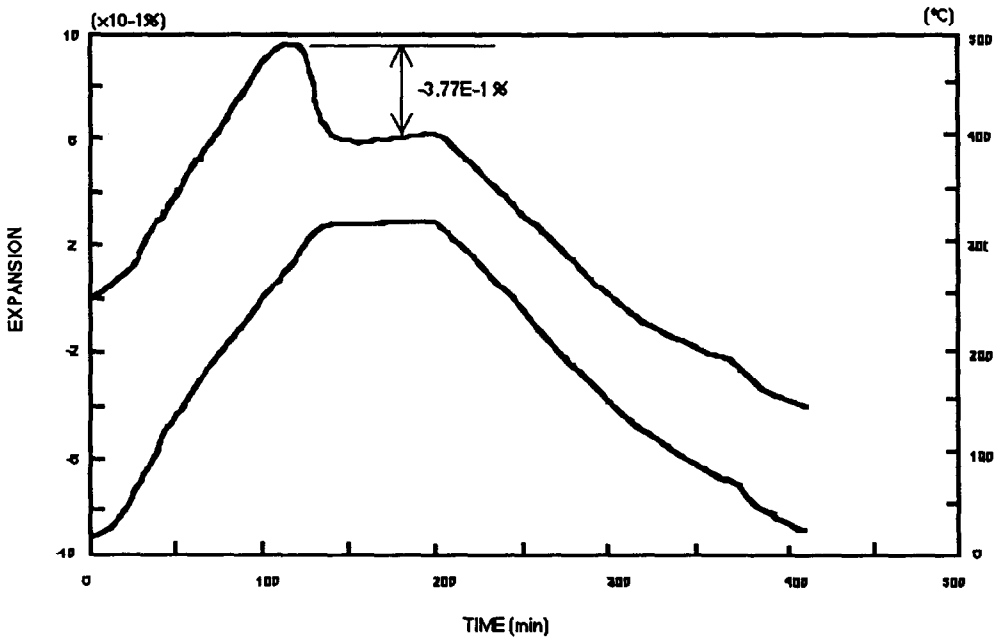


Figure 7. Thermal Expansion of a 0.295 mm Thick sheet of a Beryllium Copper Alloy.



This accurate measuring technique thus provides materials developers and users especially those associated with the electronics industry with three valuable attributes.

- precise measurements of very low and negative expansion coefficients
- quality control and quality assurance of materials where specific matching" or reproducibility to within certain limits of coefficient is required
- a technique for measurement on "thin" specimens

## SUMMARY

A description of the principles and uses of several new or improved measurement systems to evaluate necessary thermal properties of materials available in limited quantities has been given. These provide the materials developers and users, particularly those involved with applications of superconductors and semiconductors and to electronics packaging, with the necessary tools to improve performance and quality of devices and systems.

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