Transient plane source (tps) sensors for simultaneous measurements of thermal conductivity and thermal diffusivity of insulators, fluids and conductors

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Abstract. Thermal conductivity and thermal diffusivity are two important physical properties for designing any food engineering processes¹. The knowledge of thermal properties of the elements, compounds and different materials in many industrial applications is a requirement for their final functionality. Transient plane source (tps) sensors are reported² to be useful for the simultaneous measurement of thermal conductivity, thermal diffusivity and volumetric heat capacity of insulators, conductor liquids³ and high-T_C superconductors⁴. The tps-sensor consists of a resistive element in the shape of double spiral made of 10 micrometer thick Ni-foils covered on both sides with 25 micrometer thick Kapton. This sensor acts both as a heat source and a resistance thermometer for recording the time dependent temperature increase. From the knowledge of the temperature co-efficient of the metal spiral, the temperature increase of the sensor can be determined precisely by placing the sensor in between two surfaces of the same material under test. This temperature increase is then related to the thermal conductivity, thermal diffusivity and volumetric heat capacity by simple relations^{2,5}. The tps-sensor has been used to measure thermal conductivities from 0.001 $Wm^{-1}K^{-1}$ to 600 $Wm^{-1}K^{-1}$ and temperature ranges covered from 77K- 1000K. This talk gives the design, advantages and limitations of the tpl-sensor along with its applications to the measurement of thermal properties in a variety of materials.

1. Introduction

Recently a new transient technique has been proposed for thermal transport studies of solid materials' and a general theory of this transient plane source (TPS) technique is outlined below. The general theory is followed by proposed approximations for arrangements, which can be referred to as "hot square" and "hot disk". It is well known that the thermal conductivity as well as the thermal diffusivity of solids varies extensively depending on the structure, density, porosity, electrical conductivity, etc., of different materials. Frequently these properties also exhibit a strong dependence on temperature and pressure.

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The motive behind the development of the TPS technique has been to cover as large ranges of the transport properties as possible and at the same time be able to apply the technique to a large number of different materials. It is also clear that maximum applicability as well as convenience can be achieved by the use of a "resistive element" both as heat source and temperature sensor. A transient plane source element used both as a heat source and a temperature sensor may consist of a pattern of a thin layer of an electrically conducting material, the temperature coefficient (TCR) of which is such that the temperature increase of the element can be precisely deduced from recording of its resistance. In case a metal foil is used as a TPS element it may be supported on both sides by thin insulating layers. Such an arrangement also makes it possible to apply the TPS element for thermal conductance studies of electrically conducting materials. It should also be noted that we will be discussing TPS elements with approximately the same overall length-to-width dimensions. The reason for this particular choice of pattern is the desire to keep the sample dimensions as small as possible.

2. THEORETICAL BACKGROUND

The TPS method considers the three-dimensional heat flow inside the specimen. The specimen is regarded as an infinite medium. The technique uses a "resistive element" (the TPS element) both as a heat source and temperature sensor. The time-dependent temperature increase $\Delta T(t)$ gives rise to a change in the electrical resistance, R(t), of the conducting pattern shown in Fig. 1, and may be expressed as

$$R(t) = R_0 (1 + \alpha \,\overline{\Delta T(t)}) \tag{1}$$

Here R_0 is the resistance of the TPS element before transient recording has been initiated, α is the temperature coefficient of resistivity (TCR), and $\Delta T(t)$ is the mean value of the time-dependent temperature increase of the TPS element.



Figure 1.Principal electrical circuit for the modified bridge arrangement, showing TPS sensor (R_0) with specimen, standard resistance (R_s), potentiometer, dc power supply, DASU, and points of measuring voltages.

For convenience and as an experimental requirement, to obtain the thermal conductivity and thermal diffusivity simultaneously, the mean temperature change of the sensor is defined in terms of the dimensionless variable τ , where

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$$\tau = \left(\frac{t}{\theta}\right)^{\frac{1}{2}} \tag{2}$$

and

$$\theta = \frac{r^2}{a} \tag{3}$$

Where t is the time measured from the start of transient heating, a is the thermal diffusivity of the specimen, θ is the characteristic time, and r is a constant that represents a measure of the overall size of the resistive pattern (=radius of hot disk).

3. EXPERIMENTAL ARRANGEMENT

The bridge circuit used is shown in figure. The electrical circuit consists of an HP 6633B dc power supply, R_s -standard resistance, potentiometer, and digital data acquisition switch unit (DASU) Agilent 34970A with internal DMM and TPS element R_0 . DASU is used to measure quasi simultaneous voltages at the points d and e (U_{Rs}), f and g (U_{R0}), and b and e (U_{Rt}) of the circuit before and after the transient run. Also, the voltage variations ($\Delta U(t)$) (200 measurement points) across the bridge circuit during the transient run are measured by DASU. The dc output power of the power supply unit that is used for balancing the bridge and heating the TPS element is controlled through the computer. For balancing the bridge, a very low current is supplied to the circuit so that it works for balancing but does not actually heat the TPS sensor. The modified bridge circuit has many plus points as compared to the earlier used bridge, like minimal circuit components, reduced time between two consecutive measurements, better accuracy, high speed, better reproducibility and least error in results.

4. RESULTS AND DISCUSSION

With the purpose of demonstrating that the TPS technique using the modified setup could be used down to the temperature of liquid nitrogen, measurements on fused silica, carbon steel, and silver chloride crystals were performed. The main aim of the whole study is to develop a simple system providing higher accuracy that can be used for the measurement of the thermal transport properties of high-T_c superconductors. After verifying the performance of the setup, the superconducting samples with nominal composition $Bi_{1.6}Pb_{0.4}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_Y$ were studied. The dimensions of the four types of specimens and radii of the TPS sensor used are given in Table I.

S. No	Test specimen	Diameter (<u>+</u> 0.05)mm	Length (<u>+</u> 0.05)mm	Sensor radius (<u>+</u> 0.001)mm	Probing depth (<u>+</u> 0.001)mm	$ au_{ m max}$
1	Fused Silica	(a) 35.05	(a) 20.30	(i) 9.734 (sensor 1)	(i) 8.645	(i) 0.50
		(b) 35.00	(b) 20.35	(ii) 3.300 (sensor 2)	(ii) 6.500	(ii) 0.98
2	Carbon Steel (SS215/3)	(a) 38.15	(a) 19.70	9.734	15.260	0.78
		(b) 38.25	(b) 19.50			
3	Silver Chloride	(a) 25.40	(a) 4.30	3.300	3.972	0.60
	crystals	(b) 25.40	(b) 4.25			
4	$\begin{array}{ccc} {\bf 4} & & Bi_{1.6}Pb_{0.4}Sr_{1.6}Ba_{0.4}C\\ & & a_2Cu_3O_y \end{array}$	(a) 28.01	(a) 11.12	3.300	7.473	0.41
		(b) 28.02	(b) 11.31			
	Superconductor					

Table 1. Dimensions of Test Specimens, along with Sensor Radii, Probing Depth, and τ_{max}

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The measurements are carried out as a function of temperature for all four specimens. Due to the availability of data for the thermal conductivities of the studied specimens from other sources (with different techniques), comparisons of thermal conductivities are made.

4.1. Fused Silica

Data for the silica specimens were taken with sensors having radii of 9.734±0.001 mm (sensor 1) and 3.300±0.001 mm (sensor 2). The variation of the thermal conductivity with temperature is shown in Fig. 2 and the results are tabulated in Table II. The results obtained are compared with the recommended experimental values and also with the previously calibrated bridge circuit. The differences do not exceed 1% at any temperature. Previously reported differences were 1% at room temperature and 4% at low temperatures. These results show that the performance of ATPS (Advantageous Transient Plane Source - Specific method used in the present study) has been improved.

The measurement times used were 10 to 20s for sensor 1 and 10s for sensor 2. The currents used for balancing were in the range of 1.5 to 3 mA and for measurements, the current values ranged from 149 to 325 mA. Balancing currents and measurement currents decrease as the temperature is increased as the resistance of the TPS sensor and the connecting leads changes with temperature.



Figure 2. Variation of the thermal conductivity of fused silica samples withtemperature.

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4.2. Carbon Steel

Carbon steel specimens, SS215/3, obtained from Analytical Standards AB Sweden, were analyzed from room temperature down to the boiling point of liquid nitrogen. A plot of the thermal conductivity variation with temperature and composition of the specimen is shown in Fig. 3, and the results are tabulated in Table III. A similar trend has been observed in an earlier study for an almost similar composition. They made measurements from room temperature to 573 K. The values for thermal conductivity are in agreement with this report for the same temperature range. Detailed comparisons are made with the data from an earlier measurement. The thermal conductivity values decrease at low temperatures due to the reduction of the electronic contribution to conduction.

4.3. Silver Chloride Crystal

Silver chloride crystals are a useful material for deep IR applications where sensitivity to humidity is a problem. A major use is in the manufacture of small disposable cell windows for spectroscopy, known as a "minicell". The structure of these crystals is cubic, fcc.

For the AgCl crystals, only sensor 2 was suitable. Fig. 4 shows the variation of thermal conductivity with temperature for the silver chloride crystals, and the results are tabulated in Table IV. A very good match is observed for the available recommended data, and also the thermal conductivity values are extended down to liquid nitrogen temperature. These data will be helpful for application to optical windows used at low temperatures. The thermal conductivity values of AgCl are proportional to T^{-1} in the temperature region of the measurement. The detailed calculations will be published elsewhere.

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Table 2. Thermal Conductivities (in $W \cdot m^{-1} \cdot K^{-1}$) of Fused Silica from Other Sources and from Both Sensors Used in the Present Work

S. No.	$T(\mathbf{K})$	(i) <i>^a</i>	(ii) ^{<i>b</i>}	Sensor 1	Sensor 2
1	50	0.34±0.03	_	_	_
2	60	0.41 ± 0.03	_	_	_
3	70	0.48 ± 0.03	_	_	_
4	80	0.55 ± 0.03	_	_	_
5	82	_	0.595 ± 0.020	0.5542 ± 0.0007	_
6	88	_	_	0.6041 ± 0.0008	0.6061 ± 0.0008
7	89	_	0.625 ± 0.021	_	_
8	90	0.62 ± 0.04	_	_	_
9	97	_	_	0.6744 ± 0.0009	_
10	100	0.69 ± 0.03	_	_	0.6840 ± 0.0009
11	103	_	0.696 ± 0.023	_	_
12	106	_	_	0.7048 ± 0.0010	_
13	111	_	0.768 ± 0.024	_	_
14	121	_	0.821 ± 0.024	_	_
15	125	0.83 ± 0.03	_	_	0.8287 ± 0.0011
16	131	_	_	0.8501 ± 0.0011	_
17	144	_	0.933 ± 0.027	_	0.9306 ± 0.0013
18	150	0.95 ± 0.03	_	_	_
19	157	_	_	0.9782 ± 0.0013	_
20	160	_	0.995 ± 0.028	_	_
21	172	_	1.010 ± 0.028	_	1.0471 ± 0.0014
22	175	1.05 ± 0.04	_	_	_
23	177	_	1.026 ± 0.025	1.0465 ± 0.0014	_
24	188	_	_	1.0899 ± 0.0015	1.1077 ± 0.0015
25	200	1.14 ± 0.04	_	_	_
26	204	_	_	_	1.1186 ± 0.0015
27	217	_	_	1.1791 ± 0.0016	_
28	225	_	1.155 ± 0.023	_	_
29	244	_	_	1.2517 ± 0.0017	_
30	250	1.28 ± 0.04	_	_	_
31	263	_	_	1.3144 ± 0.0018	_
32	266	_	_	_	1.3127 ± 0.0018
33	273	1.33 ± 0.04	_	_	1.3391 ± 0.0018
34	281	_	1.342 ± 0.026	_	_
35	296	_	_	_	1.3873 ± 0.0019
36	300	1.38 ± 0.04	_	_	_
37	301	_	_	1.3940 ± 0.0019	_
38	303	_	1.386 ± 0.027	_	_
39	350	1.45 ± 0.04	_	_	_
40	400	1.51 ± 0.04	_	-	_

^{*a*} Touloukian and Powell, 1970 [7] ^{*b*} Calibrated bridge by Maqsood et al., 1996 [6] IOP Conf. Series: Materials Science and Engineering **51** (2013) 012006 doi:10.1088/1757-899X/51/1/012006



Figure 3. Variation of the thermal conductivity with temperature for the stainless steel No. 215/3 0.9% carbon

S. No.	<i>T</i> (K)	TPS [6]	This work
1	81	_	23.595 ± 0.033
2	83	23.62 ± 0.81	24.120 ± 0.034
3	93	24.81 ± 0.74	_
4	99	_	25.553 ± 0.036
5	109	28.08 ± 0.78	_
6	126	_	31.248 ± 0.044
7	134	32.55 ± 0.80	_
8	149	_	33.973 ± 0.047
9	174	34.45 ± 0.81	_
10	191	_	35.292 ± 0.049
11	210	35.88 ± 0.72	_
12	217	_	36.327 ± 0.051
13	242	36.70 ± 0.73	_
14	254	_	37.068 ± 0.052
15	266	37.79 ± 0.75	_
16	290	37.91 ± 0.75	_
17	298	_	38.495 ± 0.054

Table 3. Thermal Conductivity (in $W \cdot m^{-1} \cdot K^{-1}$) of Stainless Steel from Circuit Used Previously and the Present Work

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Figure 4. Variation of the thermal conductivity with temperature for AgCl crystals. Solid line is $\lambda = cT^{-1}$ fit, where c is a constant.

IOP Conf. Series: Materials Science and Engineering **51** (2013) 012006 doi:10.1088/1757-899X/51/1/012006 Table 4.Thermal Conductivity (in $W \cdot m^{-1} \cdot K^{-1}$) of Silver Chloride Crystals from Other Sources and the Present Work

S. No.	$T(\pm 1 \text{ K})$	Other sources [7]	This work
1	84	_	3.3612 ± 0.0047
2	107	_	3.0305 ± 0.0042
3	142	_	2.1661 ± 0.0030
4	153	_	2.0121 ± 0.0028
5	186	_	1.5514 ± 0.0022
6	196	_	1.4823 ± 0.0021
7	221	1.30	_
8	225	1.26	_
9	227	_	1.2511 ± 0.0018
10	228	_	1.2560 ± 0.0018
11	242	_	1.1842 ± 0.0017
12	261	_	1.1643 ± 0.0016
13	269	1.19	1.1612 ± 0.0017
14	278	1.15 ^{<i>a</i>}	_
15	291	_	1.0305 ± 0.0014
16	325	1.09	_

^a Crystran, United Kingdom.

4.4. Superconducting Samples

A Ba-doped, Bi-based, high-T_c superconductor was prepared by a solid state reaction method. The nominal composition used was Bi_{1.6}Pb_{0.4}Sr_{1.6}Ba_{0.4}Ca₂Cu₃O_y. Large-sized samples (diameter 28 mm and length ~11 mm) are investigated for thermal transport properties. The size of the samples used for the thermal conductivity measurements was indeed the largest reported for this kind of measurements. Our thermal conductivity results are plotted in Fig. 5 and are tabulated in Table V. The sample shows the same general trend and at T > T_c the thermal conductivity is slightly decreasing, but at T <T_c, it is enhanced.

Because the compound is metallic above T_c , heat can be conducted by both phonons (λ_{ph}) and electrons (λ_e). Thus, the largest contribution to the total thermal conductivity (λ) above T_c is due to lattice conduction. The sharp up-turn of the measured thermal conductivity and the further increase for $T < T_c$ has three different hypotheses: (i) only the phonons are contributing to this increase (phonon approach); (ii) only the electron contribution is responsible (electron approach); and (iii) both phonon and electron contributions are causing the increase (electron+ phonon approach).

It should be noted that the uncertainties in thermal conductivity values of our data are within 1 %. Comparing the results between different laboratories, one notes that the thermal conductivity depends on a particular sample preparation process, while the temperature dependence of the conductivity is similar for all the samples⁶⁻⁸. So the order of magnitude of the thermal conductivity is comparable to the results obtained by different authors⁹, measured by steady-state methods. Further detail of this material has been discussed by Rehman and Maqsood¹⁰.



Figure 5. Variation of the thermal conductivity with temperature for the superconducting samples

S. No.	$T(\pm 1 \text{ K})$	Thermal conductivity
1	91	1.3791 ± 0.0013
2	93	1.3582 ± 0.0013
3	95	1.2263 ± 0.0017
4	100	0.9883 ± 0.0014
5	107	0.9802 ± 0.0014
6	112	0.9879 ± 0.0014
7	116	0.9917 ± 0.0014
8	119	0.9821 ± 0.0013
9	128	0.9602 ± 0.0013
10	136	0.9638 ± 0.0013
11	145	0.9808 ± 0.0014
12	148	0.9753 ± 0.0014
13	165	1.0032 ± 0.0014
14	188	1.0051 ± 0.0014
15	203	1.0463 ± 0.0015
16	230	1.0752 ± 0.0015
17	251	1.0871 ± 0.0015
18	266	1.0820 ± 0.0015
20	276	1.0811 ± 0.0015

Table V. Thermal Conductivity (in W·m-1 ·k	K-1) of the Superconducting Samples
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5. SUMMARY AND CONCLUSIONS

An improved bridge for the transient plane source has been validated with measurements on fused silica, carbon steel, silver chloride and superconducting specimens. At any temperature, the deviation in the evaluated results of the thermal conductivity is less than 2% when compared with recommended values. With the present arrangement, the bridge circuit components are reduced, the temperature drift of the specimens is very well compensated, and the time of relaxation (time between two reliable readings) is minimized.

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