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Sub-Millisecond Measurements of Thermal Conductivity and Thermal Diffusivity Using Micrometer-Sized Hot Strips

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ABSTRACT

A new measurement technique based on the transient hot strip technique has recently been developed for studying anisotropic thermal transport properties of thin crystalline films. A micrometer-sized hot strip sensor is evaporated on the surface of the crystalline film sample, which has been deposited on a substrate wafer of limited thickness. From a pulsed transient recording, using sub-millisecond square-shaped pulses, a thermal probing depth that is less than the film thickness is assured. In the ongoing work of verifying the technique, we show results from measurements on *z*-cut crystal quartz and fused silica, using thermal probing depths of only 30 μm, which closely conform to bulk values found in the literature.

Keywords: transient Hot Strip (THS) technique, anisotropic, thermal transport properties, crystalline film, thermal probing depths.

1. INTRODUCTION

At the Transient Plane Source or Hot Disc technique encompasses transient heating of Hot Disc sensors (cf. ISO 22007-2) or Hot Strip sensors (Gustafsson, Chohan, Ahmed, & Maqsood, 1984; Gustafsson, Karawacki, & Khan, 1981; Gustavsson, Nagai, & Okutani, 2003). The sensors are made of a thin-metal foil etched in the shape of a double-spiral strip (Hot Disc sensor) or a straight strip (Hot Strip sensor). A wide range of applications have been possible to cover with this technique using a single square-shaped heating pulse and with application to different sample geometries (bulk, slab, stacked films, thin film etc.). Samples incorporating intrinsic anisotropy properties can also be determined, provided the sample is homogeneous. With the Hot Disc sensor, both the thermal conductivity and thermal diffusivity tensors of samples with uniaxial symmetries can be determined from a single-transient recording. However, for Hot Strip sensors, the corresponding tensors for samples also with biaxial symmetries can be determined. This is achieved by repositioning the Hot Strip sensor along two orthogonal directions and performing two separate transient recordings.

These transient methods, contrary to steady-state methods (e.g., Hot Plate method), make it possible to experimentally avoid the net thermal contact resistance between the heating element and the bulk surface of the sample. This facility, together with the high testing sensitivity, allows one to obtain intrinsic bulk thermophysical data, filtering out surface effects (surface roughness, wettability, mounting pressure, sensor coating protection material). This renders a unique capability to utilize the Hot Disc sensor or Hot Strip sensor for production- or assembly-line quality control, an added advantage being that single-sided and nondestructive (stethoscope-type) tests can also be performed. The experimental duration of the square-shaped heating pulse provides a direct link

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between the thermal probing depth and the test time, through a relation described in ISO 22007-2.

In the continued efforts to reduce the thermal probing depth, commercially available equipment has recently been made available for single pulse durations of only 20 milliseconds, which reduce the sample thicknesses with approximately an order of magnitude compared to standard measurements over a few seconds. To further reduce the thermal probing depth, down to the range of a few micrometers, we recently demonstrated miniaturized Hot Strip sensors that are deposited onto the sample and heated by a train of square-shaped pulses well separated by the choice of a duty cycle to 5% (Ma, Gustavsson, Haglund, Gustavsson & Gustafsson, 2014). Since this technique is specially adapted for studying anisotropic thermal transport properties of a <50-μm-thin crystalline film that is deposited on a substrate of limited thickness (e.g., semiconductor wafer materials are typically ~200–500 μm thick), it is referred to as the Slab Pulse Transient Hot Strip (Slab PTHS) technique. This technique can of course be used to study isotropic properties of similar thin amorphous films as well. We here present some new measurement results on *z*-cut crystal quartz and fused silica in the continued work of verifying the technique.

2. SLAB PTHS TECHNIQUE

A series of periodic square-shaped current pulses, with a low duty cycle (F) of 5%, are fed to the strip sensor via a pulse generator in an AC-coupled network as shown in Figure 1. The subsequent increased electrical resistance of the strip ($R(T)$) from Joule heating is monitored by recording the average voltage increase in the strip using a low-pass filter and a nanovoltmeter.

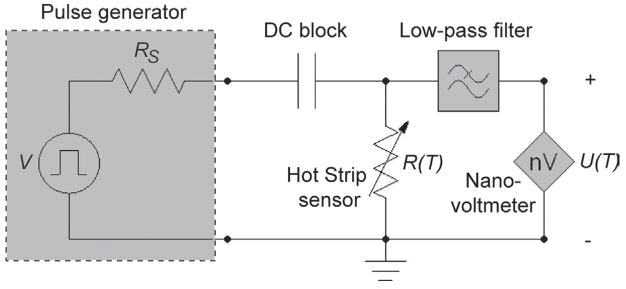

Figure 1. Electrical circuit for the Slab PTHS technique, where the DC block ensures an AC coupled network, and the low-pass filter facilitates the recording of the time-average voltage increase in the Hot Strip sensor by the nanovoltmeter.

The average temperature increase in the strip (ΔT_{MV}) is proportional to the measured resistance increase, and this can also be expressed analytically as a function of pulse duration (FP, where P is the pulse period), c.f. (Ma, Gustavsson, Haglund, Gustavsson, & Gustafsson, 2014):

$$\Delta T_{MV}(\mathrm{FP}) = \frac{P_0}{2\sqrt{\pi}\cdot h\cdot\sqrt{\lambda_1\cdot\lambda_2}}\cdot\frac{1}{\mathrm{FP}} \cdot\int_0^{\mathrm{FP}} \mathrm{d}t\cdot[f(\tau)-F^2\cdot\beta], \tag{1}$$

where the dimensionless time function $f(\tau)$ is given by

$$f(\tau) = \int_0^{\tau}\mathrm{d}x\cdot\left(\mathrm{erf}\left\{\frac{1}{x}\right\}-\frac{x}{\sqrt{\pi}}\left[1-\exp\left\{-\frac{1}{x^2}\right\}\right]\right),$$
$$\tau = \frac{\sqrt{tn\cdot\kappa_2}}{d} \tag{2}$$

Parameter P_0 is the Joule heating power in the strip, and is related to the pulse voltage (V) via

$$P_0 = \frac{V^2\cdot R}{(R+R_s)}, \tag{3}$$

and λ_1 is the thermal conductivity perpendicular to the plane of which the strip is placed on, and λ_2 and κ_2 is the thermal conductivity and thermal diffusivity, respectively, parallel to the plane of which the strip is placed on, but perpendicular to the orientation of the strip. Parameter h is the strip half length, and d is the strip half width. The constant β accounts for a constant "background" temperature difference that is built up over the sample layer and substrate, and thus depends on the thickness and thermal properties of both the sample layer and the substrate (Ma et al., 2014). Using the definitions,

$$T_+ = \frac{P_0}{2\sqrt{\pi}\cdot h\cdot\sqrt{\lambda_1\cdot\lambda_2}}$$

and

$$H(\tau) = \frac{2}{\tau^2}\cdot\int_0^{\tau}\mathrm{d}x\cdot x\cdot f(x),$$

Equation (1) can be written as

$$\Delta T_{MV}(\mathrm{FP}) = T_+\cdot H(\tau)-\beta\cdot T_+\cdot F^2 \tag{4}$$

Thus, a plot of measured ΔT_{MV} versus calculated $H(\tau)$ for different pulse durations (FP) results in a straight line with slope T_+. Since the thermal diffusivity κ_2 is not known, an iteration process is needed to find the τ-value, and thereby κ_2-value, that gives the best straight line fit, see Figure 2. From the slope of

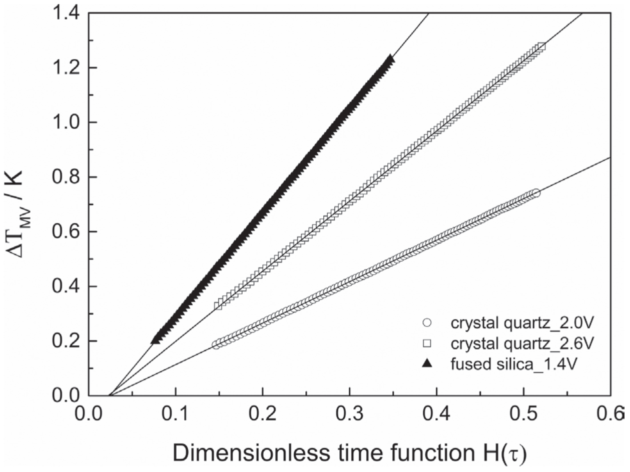

Figure 2. Examples of best straight line fit of calculated dimensionless time function $H(\tau)$ to measured average temperature increase ΔT_{MV} in a 32-μm-wide Hot Strip sensor, using pulse durations between 3 and 100 μs. The data are collected from measurements on a crystal quartz sample with set pulse generator voltage ($V/2$) of 2.0 V, 2.6 V, and fused silica with 1.4 V.

these straight lines (T_+) the square root product of the thermal conductivities $\sqrt{\lambda_1 \cdot \lambda_2}$ is also obtained. Assuming that the pulses are ideally square shaped, a thermal probing depth (Δ_p) into the sample can be defined as (Ma et al., 2014)

$$\Delta_p = 2 \cdot \sqrt{\kappa \cdot t_{\max}} \qquad (5)$$

where $t_{\max}$ is the longest pulse duration used in the pulsed transient recording, i.e. $t_{\max} = FP_{\max}$. It should also be noted that highest sensitivity – in the dual determination of both the thermal conductivity and the thermal diffusivity from one pulsed transient recording – is achieved when the width of the strip is matched to the thermal probing depth, and where the pulse voltage is adjusted to yield an average temperature increase in the strip ΔT_{MV} ~ 1 K at the longest pulse duration of the recording.

3. MEASUREMENT RESULTS ON CRYSTAL QUARTZ AND FUSED SILICA

We have previously reported measurement results on a 2-inch (50.8 mm in diameter) single crystal quartz wafer from MTI Corporation (500-μm-thick) with a *y*-cut surface orientation, using the Slab PTHS technique (Ma et al., 2014). Two 32-μm-wide Hot Strip sensors were deposited along two orthogonal crystalline (*x* and *z*) directions, and from individual pulse transient recordings the anisotropic thermal transport properties of the uniaxial crystal quartz could be determined, using a thermal probing depth of only ~30 μm. Along the perpendicular (parallel) direction to the z-, i.e., optic axis, a thermal conductivity of 6.48 (11.44) W/mK, and a thermal diffusivity of 3.62 (6.52) mm^2/s, were obtained – which values are in agreement with corresponding literature values for bulk crystal quartz (Clauser & Huenges, 1995).

Now, we present measurement results on a similar 2-inch single crystal quartz wafer also from MTI Corporation (500-μm-thick) but with a *z*-cut surface orientation. Since it is *z*-cut we cannot orient the Hot Strip sensor along the *z*-axis, wherefore the thermal diffusivity perpendicular to *z*-direction ($\kappa_\perp$) and the square root product of the thermal conductivity perpendicular and parallel to *z*-direction ($\sqrt{\lambda_\perp \cdot \lambda_\parallel}$) can only be retrieved from the pulsed transient recordings. Table 1 summarizes results from recordings on four individual 32-μm-wide Hot Strip sensors at three different pulse voltages. Two strips were oriented along the *x*-direction, and two strips were oriented along the *y*-direction. Assuming a commonly found literature value on volumetric heat capacity for crystal quartz of 1.89 MJ/m^{3}K (Manufacturer Data Sheet of Crystal Quartz, 2015), one can extract a thermal conductivity perpendicular (parallel) to the *z*-axis of 6.54 (11.76) W/mK, and a thermal diffusivity 3.46 (6.22) mm^2/s, with a standard deviation of less than 2%. The results are consistent with our previously reported data on y-cut crystal quartz discussed above, and they also imply that the *z*-cut wafer does not display any structural defects within the investigated probing depth of ~30 μm, since thermal transport properties are sensitive to even slight deviations from an ideal crystalline structure. To demonstrate the technique on a sample having an order of magnitude lower thermal conductivity than for crystal quartz, we also present results on a 2-inch (500-μm-thick) fused silica wafer (also from MTI Corporation). Since fused silica has an amorphous (noncrystalline) structure, the thermal transport properties are isotropic wherefore the orientation of the strip is in this case not important. Table 2 lists results from recordings on a 32-μm-wide Hot Strip sensor at five different pulse voltages. For a thermal probing depth of ~30 μm, a thermal conductivity of 1.32 W/mK, a thermal diffusivity of 0.90 mm^2/s, and a volumetric heat capacity of 1.47 MJ/m^{3}K, with a standard deviation of less than 3%, is obtained and aligns with tabulated values on bulk fused silica, see e.g., Manufacturer Data Sheet of Fused Silica, (2015). As can be observed in Tables 1 and 2, no deterministic variation of the obtained thermal transport properties was found when varying the pulse voltage, which supports the analytical model behind the technique.

Table 1. Measured thermal conductivity and thermal diffusivity in direction perpendicular to optic-axis of z-cut single crystal quartz wafer, using the Slab PTHS technique with a 32-μm-wide Hot Strip sensor and a ~30 μm thermal probing depth.

Strip #	Temp. coeff. of resistivity (K^{-1})	Set pulse voltage (V/2) (V)	Output power (P_0) (mW)	ΔT (K)	$\sqrt{\lambda}$ (Wm^{-1} K^{-1})	$\lambda_\perp$(mm^{2}s^{-1})
x-oriented						
1	0.00326	2	75.5	0.732	8.783	3.400
		2.3	99.8	0.974	8.831	3.426
		2.6	127.6	1.278	8.739	3.311
2	0.00330	2	75.5	0.739	8.827	3.458
		2.3	99.9	0.983	8.783	3.421
		2.6	127.7	1.261	8.881	3.526
y-oriented						
3	0.00324	2	75.6	0.741	8.684	3.501
		2.3	100.0	0.989	8.788	3.522
		2.6	127.8	1.268	8.780	3.533
4	0.00326	2	76.1	0.753	8.779	3.479
		2.3	100.6	1.002	8.743	3.444
		2.6	128.6	1.287	8.786	3.485
			Mean value		**8.784**	**3.459**
			Std dev %		**0.57%**	**1.85%**

Table 2. Measured thermal conductivity and thermal diffusivity of fused silica wafer, using the Slab PTHS technique with a 32-μm-wide Hot Strip sensor and a ~30 μm thermal probing depth.

Strip #	Temp. coeff. of resistivity (K^{-1})	Set pulse voltage (V/2) (V)	Output power (P_0) (mW)	ΔT (K)	(Wm^{-1}K^{-1})	λ(mm^{2}s^{-1})	ρCp (MJm^{-3}K^{-1})
1	0.00267	0.8	12.7	0.469	1.329	0.913	1.455
		1.0	19.9	0.737	1.347	0.940	1.433
		1.2	28.6	1.063	1.323	0.905	1.462
		1.4	39.0	1.445	1.307	0.878	1.490
		1.6	50.9	1.883	1.309	0.877	1.493
			Mean value		**1.323**	**0.903**	**1.467**
			Std dev %		**1.22%**	**2.94%**	**1.72%**

REFERENCES

Clauser, C. & Huenges, E. (1995). Thermal conductivity of rocks and minerals. In T. J. Ahrens (Ed.), *Rock physics & phase relations: a handbook of physical constants*. Washington, DC: American Geophysical Union. doi: 10.1029/RF003p0105

Gustafsson, S. E., Chohan, M. A., Ahmed, K., & Maqsood, A. (1984). Thermal properties of thin insulating layers using pulse transient hot strip measurements. *Journal of Applied Physics, 55*(9), 3348.

Gustafsson, S. E., Karawacki, E., & Khan, M. N. (1981). Determination of the thermal-conductivity tensor and the heat capacity of insulating solids with the transient hot-strip method. *Journal of Applied Physics, 52*(4), 2596.

Gustavsson, M., Nagai, H., & Okutani, T. (2003). Thermal effusivity measurements of insulating liquids using microsized hot strip probes. *Review of Scientific Instruments, 74*, 4542.

Ma, Y., Gustavsson, J. S., Haglund, Å, Gustavsson, M., & Gustafsson, S. E. (2014). Pulse transient hot strip technique adapted for slab sample geometry to study anisotropic thermal transport properties of μm-thin crystalline films. *Review of Scientific Instruments, 85*, 044903.

Manufacturer Data Sheet of Crystal Quartz. (2015). Retrieved from: http://www.mtixtl.com/sio2singlecrystalquartz.aspx

Manufacturer Data Sheet of Fused Silica. (2015). Retrieved from: http://www.mtixtl.com/fusedsilicasubstratessio2.aspx