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Accurate contact resistance characterization for thermal conductivity measurement with the Heat Flow Meter method

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Abstract. A common method to measure the thermal conductivity of low-conductive materials is to impose a known temperature difference across the thickness of the specimen, and to measure the resulting heat flow once steady-state conditions are reached. In particular, international standards, i.e. ASTM C518 and ISO 8301, define the characteristics of the guarded Heat Flow Meter apparatus and its measurement procedure. However, the actual measured quantity is the overall thermal resistance, which is given by the series of the contact resistance between the specimen and the temperature-controlled clamps, and the resistance of the specimen itself. Thus, the contact resistance must be correctly quantified in order to retrieve an accurate measurement. To this end, common practices are either to rely on a database of known contact resistances for material classes, or to use the “double thickness” method, which allows to eliminate the contribution of the contact resistance by carrying out the measurement on two specimens of the same material, but different thickness. While the first method is rather useless for accurate and reliable measurements, especially of unknown or innovative materials, the latter works only if the contact resistances, and therefore the surface finish, are the same for every surface of the specimen set. This paper presents an analysis of the uncertainties associated with the evaluation of the contact resistance carried out on several samples, and proposes a method to reduce such uncertainties, i.e. by inserting elastic thermal pads of known thermal conductivity between the specimen and the instrument. The results of the validation of the method are also shown, with an analysis on the improvement of the measurement accuracy for specimens with high roughness and irregular surfaces, or with conductivities beyond the instrument declared range.

1 Introduction

The advancement of material sciences has led to an increased understanding of structures at micro- and nanoscales which, coupled with the development of a plethora of new production technologies, is giving birth to many innovative materials. Among them, a non-exhaustive list of macro categories includes phase-change materials, metallic foams, polymer composites, high-temperature insulation materials, as well as recycled-content building materials. An adequate characterization of their thermophysical properties, and, among them, thermal conductivity, is needed by engineers to adopt them in new designs, and research should be carried out to establish whether traditional measurement techniques can be applied to new materials.

Focusing on materials with low to medium thermal conductivity, i.e. from $0.1 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ to $10 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$, Yüksel [1] and Hammerschmidt et al. [2] provide good reviews of standard measurement techniques, the former focusing on building materials and the latter on high-temperature insulation



materials, but their observations have general purpose. In particular, in [2] is pointed out that the vast majority of thermal conductivity measurements on solids world-wide are performed in a range from $0.01 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$ to about $7 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$, at temperatures between $10 \text{ }^\circ\text{C}$ and $70 \text{ }^\circ\text{C}$, and therefore instrument manufacturers design their instruments predominantly for this market segment, which is also highly regulated by international standards. In this range, steady-state methods, based on establishing a one-directional, stationary heat flow across the specimen by imposing a controlled temperature difference, can directly provide the thermal conductivity, without the need of knowing other thermophysical properties, with obvious advantages on measurement accuracy. Among the two main techniques, i.e. the Guarded Hot Plate (GHP) and the Heat Flow Meter (HFM), the GHP, described by the standard ASTM E1530 [3], is regarded as the most accurate, while the HFM, described by the standards ISO 8301 [4] and ASTM C518, can still provide a good measurement accuracy, with a relatively quicker test and a simpler, less expensive, apparatus.

The main issue, both for GHP and HFM techniques, is to either lower the contact resistance with respect to the sample resistance in order to make it negligible, or to establish a reliable method to take it into account. To this end, both standards prescribe that the specimen should have smooth and parallel faces, and the GHP test further prescribes that a thin layer of heat transfer compound or a thin layer of elastomeric heat transfer medium should be applied to the sample's faces in contact with the instrument. The HFM standard is less strict regarding sample preparation, although it warns about the limitations on the measurement accuracy due to high contact resistances and specimen non-homogeneity.

Several studies on best practices to reduce the effect of the contact resistance on conductivity measurements have been carried out, either considering sample preparation or measurement analysis. Tleoubaev and Brzezinski [5] describe the "Two Thickness Method", which consists in measuring two samples of the same material and different thickness and combining the results to exclude the contact resistance from the calculation of the thermal conductivity. Stacey et al. [6] analyze the effect of different interface materials, e.g. enhanced greases and foils, on measurement repeatability at various temperatures, while Clarke et al. in their papers ([7], [8], [9]) focus on the use of buffer materials for measuring building materials with low surface homogeneity.

In this framework, the goals of this work are to verify the uncertainty value associated with thermal conductivity measurements with the HFM technique and to illustrate the issues associated with the contact resistance for different materials, i.e. PMMA, enhanced rubber and AISI 308 steel, which represent, respectively, a reference material, a material with non-homogeneous surface finish, and a high-conductivity material for the method under investigation, with the final aim to gain knowledge and understanding of the HFM method for measuring more complex materials. Moreover, an analysis has been carried out to verify if the use of silicone thermal pads as contact enhancers can improve the measurement quality.

2 Materials and methods

2.1 HFM method

The HFM method is based on imposing a controlled temperature difference ΔT between the two surfaces which clamp the specimen. The resulting heat flux q is measured when stationary conditions are reached. Under the hypothesis of 1-D heat transfer, the total thermal resistance R_{tot} can be retrieved from equation 1:

$$R_{tot} = \frac{\Delta T}{q} \quad (1)$$

where R_{tot} is the sum of the thermal resistance of the specimen, given by the ratio of its thickness s and its thermal conductivity λ , and two extra resistances R_{c1} , R_{c2} , as in equation 2,

$$R_{tot} = R_{c1} + R_s + R_{c2} = R_{c1} + \frac{s}{\lambda} + R_{c2} = R_{c,tot} + \frac{s}{\lambda} \quad (2)$$

The terms R_{c1} and R_{c2} depend on many factors, and include not only the contact resistance between adjoined surfaces, which may depend on surface roughness, planarity, rigidity, but also all thermal resistances between the temperature sensors and the samples' surfaces, e.g. contact agents and thermal pads with their respective contact resistances. For smooth specimens with parallel surfaces, low thermal conductivity and high thickness, these terms can be neglected without affecting the measurement outcome. However, if such conditions are not verified, two methods can be employed to measure λ without the need of directly evaluating $R_{c,tot}$.

The *double thickness* method is based on using two samples with the same thermal conductivity and surface finish, hence under the assumption of having the same contact resistances, but different thicknesses, i.e. s_1 and s_2 :

$$R_{tot,1} = R_{c1} + R_{s,1} + R_{c2} = R_{c1} + \frac{s_1}{\lambda} + R_{c2} \quad (3a)$$

$$R_{tot,2} = R_{c1} + R_{s,2} + R_{c2} = R_{c1} + \frac{s_2}{\lambda} + R_{c2} \quad (3b)$$

Subtracting equations 3a and 3b, it is obtained

$$R_{tot,1} - R_{tot,2} = \frac{s_1 - s_2}{\lambda} \quad (4)$$

Hence,

$$\lambda = \frac{s_1 - s_2}{R_{tot,1} - R_{tot,2}} \quad (5)$$

Moreover, considering R_{tot} as a function of thickness s , equation 5 shows that λ is the reciprocal of the slope of the straight line which passes through the points $(s_1, R_{tot,1})$ and $(s_2, R_{tot,2})$. If multiple samples of various thickness were available, i.e. multiple points in the s - R_{tot} plane for the same material were known, the concept can be extended by calculating λ as the reciprocal of the slope of the linear square approximant $R_{tot}(s) = \alpha \cdot s + \beta$, as shown in equation 6. This can be called the *multiple thickness* method.

$$\frac{dR_{tot}}{ds} = \alpha = \frac{1}{\lambda} \quad (6)$$

Either way, it is then possible to calculate a-posteriori the total contact resistance $R_{c,tot}$ with equation 2, which also implies that $R_{c,tot}$ is the y-intercept β of the curve $R_{tot}(s)$, when multiple thicknesses are analyzed.

2.2 Experimental apparatus and procedures

The instrument used in the experimental campaign is the FOX 50 by TA Instruments, shown in figure 1. Its declared thermal resistance measurement range is $0.003 \text{ m}^2 \cdot \text{K} / \text{W}$ to $0.05 \text{ m}^2 \cdot \text{K} / \text{W}$. The temperature range of the plates is $-10 \text{ }^\circ\text{C}$ to $110 \text{ }^\circ\text{C}$. It can accommodate cylindrical samples up to 62 mm-diameter, and, while cylindrical shapes are highly recommended, the measurement can still be effective if the specimen fully covers the central $25 \text{ mm} \times 25 \text{ mm}$ heat flux transducer and lateral heat dispersions can be considered low enough to establish a 1-D temperature gradient. The maximum specimen thickness is 25 mm. The apparatus can also be operated in vacuum conditions.

The operator sets the average temperature T_{ave} of the plates, their temperature difference ΔT and the direction of the heat flow, i.e. upwards or downwards. The instrument clamps the sample, while

measuring its thickness, and provides the heat-flow-meter readings when stationary conditions are reached, i.e. the difference between the upper and lower sensor is below a threshold value.

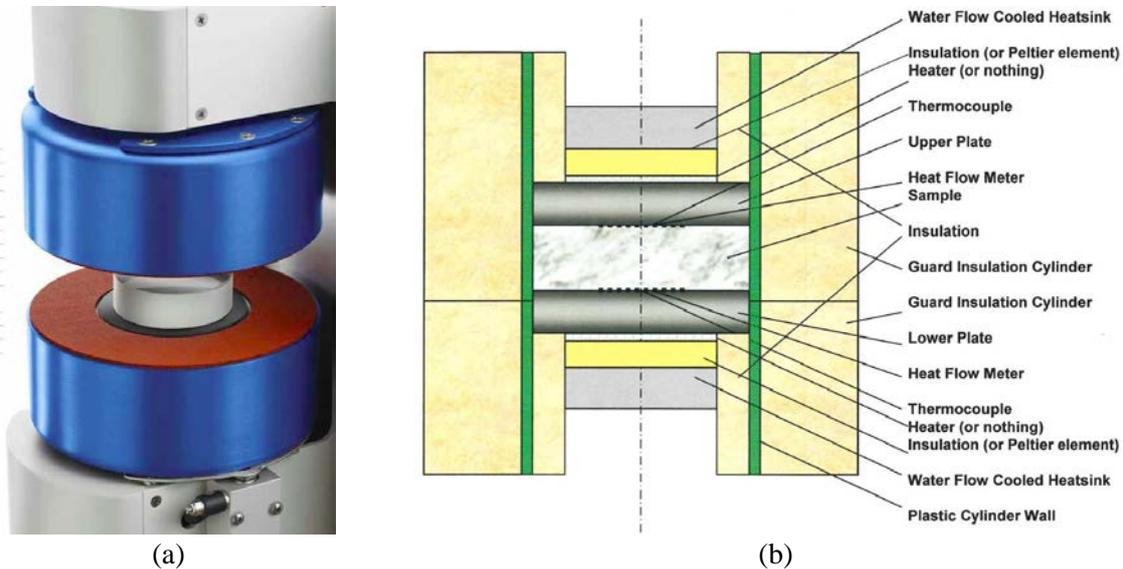


Figure 1. FOX 50 HFM, photo and schematic, source FOX 50 manual

The reported test campaign has been carried out at a mean temperature $T_{mean} = 25\text{ }^{\circ}\text{C}$ with a temperature differential between the hot and cold plates $\Delta T = 10\text{ }^{\circ}\text{C}$, with downward-pointing heat flux, so the upper and lower setpoint temperatures were $T_{up} = 30\text{ }^{\circ}\text{C}$ and $T_{down} = 20\text{ }^{\circ}\text{C}$, respectively. Tests with $\Delta T = 5\text{ }^{\circ}\text{C}$ have also been carried out, but this variation does not influence the results for the presented samples, so their results are omitted for brevity.

2.3 Samples

Three materials have been tested in this experimental campaign, as they are representative of different measurement issues, and for each material a set of samples of several thickness has been tested. PMMA samples with diameter $D = 60\text{ mm}$ and $D = 50\text{ mm}$ are used to test the instrument's accuracy with a well characterized reference material and to verify whether a diameter variation can affect the measurement outcome. Enhanced rubber samples are representative of samples with an uneven surface finish. Finally, AISI 308 is a high-conductive material with respect to the instrument measurement capabilities, so the contact resistance term is predominant with respect to the material's resistance. Therefore, only an effective strategy to take it into account can lead to a meaningful measurement.

Table 1. Sample list and size

PMMA, $D = 60\text{ mm}$		PMMA, $D = 50\text{ mm}$		Rubber, $D = 60\text{ mm}$		AISI 308, $D = 60\text{ mm}$	
label	s / mm	label	s / mm	label	s / mm	label	s / mm
P60A	2.59	P50A	2.46	RA	1.96	SA	15.01
P60B	2.79	P50B	2.69	RB	3.35	SB	20.12
P60C	4.27	P50C	4.24	RC	4.55	SC	25.10
P60D	4.78	P50D	4.83	RD	5.44		
P60E	5.23	P50E	5.16				
P60F	9.86	P50F	9.75				



Figure 2. PMMA samples

2.4 Silicone thermal interface pad

Each sample set has been measured in two conditions, i.e. first without contact enhancers and after with a silicon thermal adhesive pad applied to both contact surfaces. The pad is 1 mm-thick, with a declared thermal conductivity of $2.0 \text{ W}\cdot(\text{m}\cdot\text{K})^{-1}$, and is cut to shape and applied to each face of the specimen with care to avoid any air pocket.

2.5 Uncertainty analysis

The analysis presented in this paper is aimed at evaluating the accuracy of thermal conductivity measurements for different samples, and, while many factors may influence it, the Type A uncertainties associated with the instrument's sensors provide the lowest possible uncertainty, applicable for samples with no contact resistance, i.e. $R_c = 0$, and for ideal 1D behaviour. In these cases, equations 1 and 2 directly provide the thermal conductivity from the measurements of temperature difference ΔT , heat flux q and thickness s . Being σ the standard deviation of a set of measurements, $\sigma_{\Delta T}/\Delta T$ and σ_q/q are the relative uncertainties with 95 % confidence interval declared by the manufacturer for temperature difference and heat flux measurements, respectively, and σ_s is the uncertainty of the thickness measurement, shown in equation 7. The uncertainty propagation leads to equation 8 for the relative uncertainty of the total resistance R_{tot} .

$$\sigma_s = \pm 0.025 \text{ mm} \quad (7)$$

$$\frac{\sigma_{R_{tot}}}{R_{tot}} = \left(\left(\frac{\sigma_{\Delta T}}{\Delta T} \right)^2 + \left(\frac{\sigma_q}{q} \right)^2 \right)^{1/2} = ((0.2 \%)^2 + (0.5 \%)^2)^{1/2} = 0.54 \% \quad (8)$$

Combining the two accordingly to equation 2, the relative uncertainty for the thermal conductivity is, for a 2 mm-thick sample is

$$\frac{\sigma_\lambda}{\lambda} = \left(\left(\frac{\sigma_s}{s} \right)^2 + \left(\frac{\sigma_{R_{tot}}}{R_{tot}} \right)^2 \right)^{1/2} = ((1.25 \%)^2 + (0.5 \%)^2)^{1/2} = 1.36 \% \quad (9)$$

The same uncertainty is 3.5 % according to the manufacturer, as it takes into account also the uncertainties associated with the calibration process.

It is possible to estimate the uncertainties associated with the *multiple thickness* method, i.e. by analysing the results of multiple measurements of samples with different thickness with a linear regression on the s - R_{tot} plane.

Given the uncertainties on the single measurement, the uncertainties of the slope m and y-intercept q of the regression line, σ_α and σ_β respectively, are calculated using the procedure proposed by Krystek and Anton [11], which provide such values from the uncertainties on the coordinates of the points. While

the latter directly gives the uncertainty of the contact resistance, as $\sigma_\beta = \sigma_{R_{tot}}$, the one for the thermal conductivity is derived from equation 10. These values are reported in the results tables.

$$\sigma_\lambda = \left(\left(\frac{d}{d\alpha} \frac{1}{\alpha} \sigma_\alpha \right)^2 \right)^{1/2} = \pm \alpha^2 \sigma_\alpha \quad (10)$$

3 Results

3.1 PMMA

A first set of trials has been carried out to test the validity of the method, measuring PMMA samples with known thermal conductivity at 25 °C, i.e. 0.1899 W·(m·K)⁻¹ [10]. The sample sets with diameter $D = 60$ mm and $D = 50$ mm have both been tested for comparison. No contact agents have been used for this set of trials. The results are shown in figure 3, while numerical results are listed in table 2.

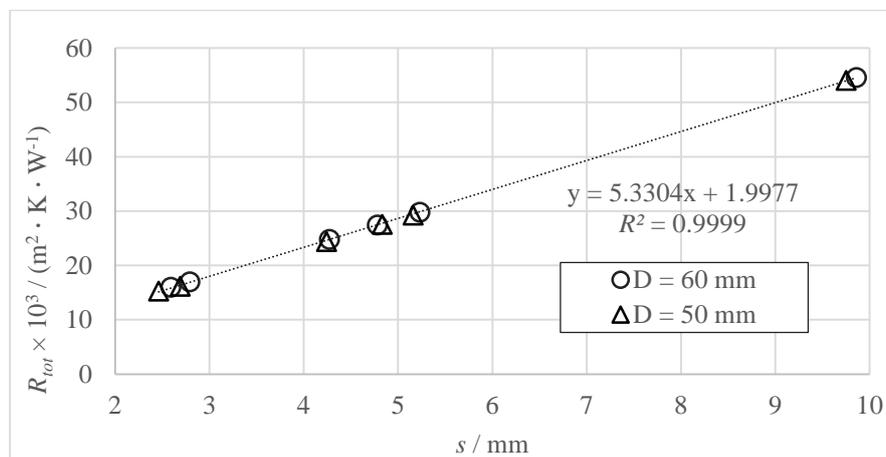


Figure 3. PMMA results, $D = 60$ mm vs $D = 50$ mm, regression line calculated from both datasets combined

Table 2. PMMA results, $D = 60$ mm and $D = 50$ mm

Series	$\lambda / (\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1})$	σ_λ / λ	$R_{c,tot} \times 10^3 / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$\sigma(R_{c,tot}) / R_{c,tot}$	R^2
$D = 60$ mm	0.188	0.9 %	2.14	9.3 %	1.0000
$D = 50$ mm	0.187	1.4 %	2.28	12.4 %	0.9999
Both series	0.188	0.8 %	2.17	7.4 %	0.9999
$D = 60$ mm	0.190*		2.30		-

*Two thickness method

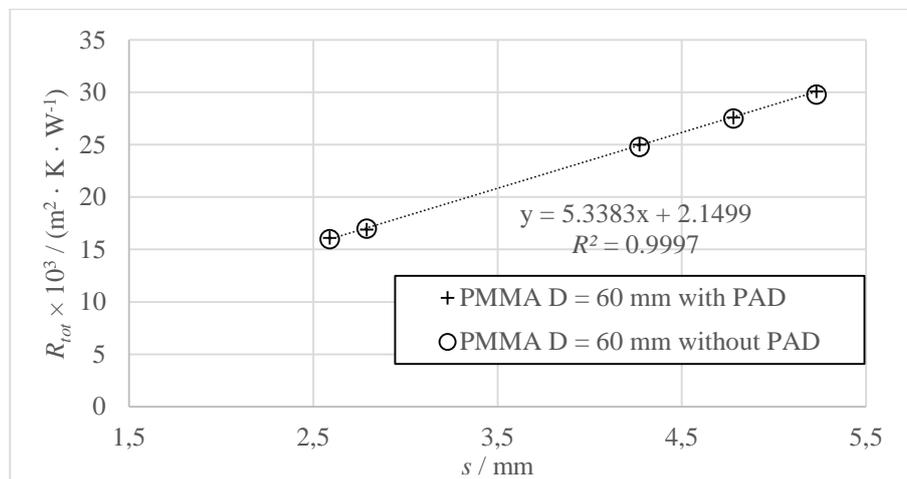
In particular, table 2 lists the thermal conductivity results, obtained from the linear regression of all the measurements of the sets with $D = 60$ mm and $D = 50$ mm separately, and a comparison with the average values obtained from the application of the two-thickness method to each possible combination of the six specimens with $D = 60$ mm. Regarding the conductivity, the maximum error with respect to the reference is 1.5 %. However, results are less uniform on the total contact resistance $R_{c,tot}$, among data sets, even though the measurements show a very high linearity, and this result is reflected in the higher relative uncertainty for the total contact resistance shown in table 2.

The results also allow to analyze the relative weight of the contact resistance $R_{c,tot}$ on the total resistance R_{tot} , which gives a glimpse on the error which is committed when this term is not taken into account, as shown in table 3.

Table 3. Relative weights of the total contact resistance $R_{c,tot}$ on the total measured thermal resistance R_{tot}

Sample	s / mm	$R_{tot} / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$R_{c,tot} = 1.86 \times 10^{-3} \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$	$R_{c,tot} = 2.39 \times 10^{-3} \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$
			$R_{c,tot} / R_{tot}$	$R_{c,tot} / R_{tot}$
P60A	2.59	16.0	11.6%	14.9 %
P60B	2.79	17.0	10.9%	14.1 %
P60C	4.27	24.8	7.5%	9.6 %
P60D	4.78	27.5	6.8%	8.7 %
P60E	5.23	29.8	6.2%	8.0 %
P60F	9.86	54.6	3.4%	4.4 %

The same trials have been carried out for the set of specimens with $D = 60$ mm, with the silicone thermal pad applied to both surfaces of the specimen. Specimen F is excluded from the analysis, since its measured resistance is above the declared range of the instrument. The aim of the trial is to find a reproducible value of $R_{c,tot}$ which can then be used for samples when only one thickness is available.

**Figure 4.** PMMA results, regression line from measurements with pad**Table 4.** PMMA results, without and with pad

Series	$\lambda / (\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1})$	σ_λ / λ	$R_{c,tot} \times 10^3 / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$\sigma(R_{c,tot}) / R_{c,tot}$	R^2
$D = 60$ mm	0.188	0.9 %	2.14	9.3 %	1.0000
$D = 60$ mm PAD	0.187	1.5 %	2.15	12.7 %	0.9997
$D = 60$ mm	0.190*		2.39		-

*imposed reference value

The results, shown in figure 4, highlight that the thermal pad does not change the measurement outcome, as the variation of total resistance for each trial is below the measurement uncertainty. The total contact resistance $R_{c,tot}$ is estimated in two ways, i.e. from the y-intercept of the fitting curve, and by imposing a thermal conductivity equal to $0.190 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ and retrieving $R_{c,tot}$ from equation 2. As it can be seen in table 4, results are $2.15 \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$ and $2.39 \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$ respectively.

3.2 Rubber

The rubber sample set has been measured with and without silicon pad as contact enhancer, and results are illustrated in figure 5 and table 5. Both sets show good linearity, although the use of the pad increases the R^2 value from 0.9879 to 0.9999. The uncertainties seem to increase with the inclusion of the thermal

pad, but those values are dependent only on the measurement uncertainties and on the sample thickness distribution, as it will be further discussed later. A last confrontation is made by imposing the thermal resistance obtained from the PMMA reference case.

Table 6 show the relative weight of contact resistance with respect to the total resistance, which is significant, especially for the low thickness sample.

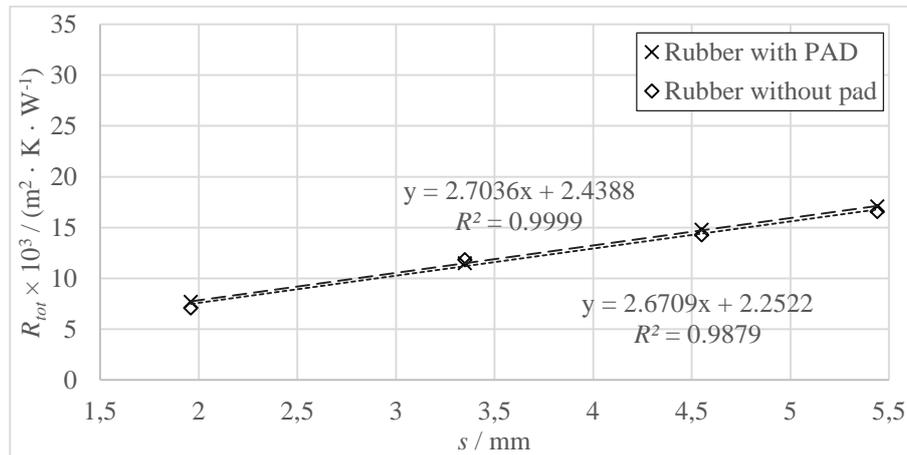


Figure 5. Rubber results, regression line with pad (above) and without pad (below)

Table 5. Rubber results

Series	$\lambda / (\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1})$	σ_λ / λ	$R_{c,tot} \times 10^3 / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$\sigma(R_{c,tot}) / R_{c,tot}$	R^2
No PAD	0.374	1.3 %	2.25	5.8 %	0.9879
With PAD	0.370	1.4 %	2.43	6.9 %	0.9999
With PAD	0.368		2.39*		-

* imposed value from PMMA calibration

Table 6. Relative weights of the total contact resistance $R_{c,tot}$ on the total measured thermal resistance R_{tot}

Sample	s / mm	$R_{tot} / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$R_{c,tot} / R_{tot}$
RA	2,59	16,0	31,0 %
RB	2,79	17,0	21,8 %
RC	4,27	24,8	16,2 %
RD	4,78	27,5	14,0 %

3.3 AISI 308

AISI 308 steel thermal conductivity reference value is $15.2 \text{ W} \cdot (\text{m} \cdot \text{K})^{-1}$ at $20 \text{ }^\circ\text{C}$, so the specimens have to be sufficiently thick in order to fall in the thermal resistance measurement range. The results without application of the thermal pad show poor linearity, with $R^2 = 0.6666$, which leads to a poor estimation of the thermal conductivity, while the adoption of the thermal raises the linearity up to $R^2 = 0.9776$. The linearity is directly associated with the measurement error with respect to the reference value, which is 54.5 % without pad and 5.5 % with pad. The thermal conductivity estimation by using the contact value obtained from the PMMA calibration leads to an error equal to 25.7 %.

Moreover, while only one of this measurement sets produces a reasonable result, it should be pointed out that the uncertainty associated with the measurements, even those with low linearity, does not provide an indication about the absolute error.

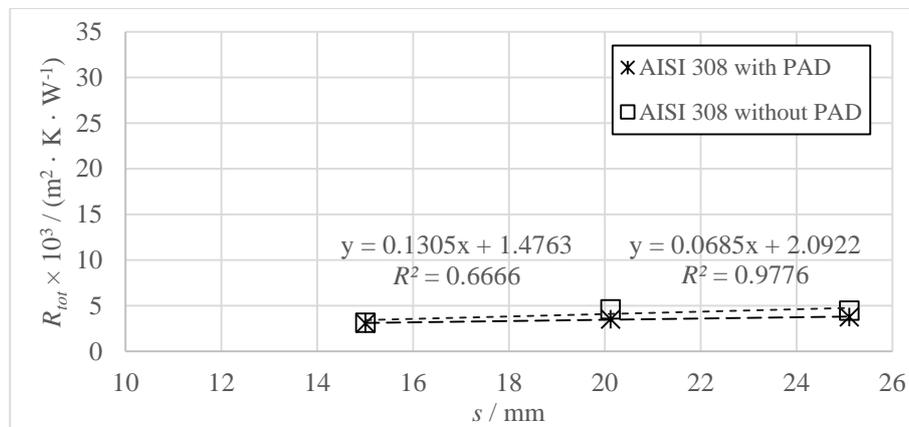


Figure 6. AISI 308 results, regression line without pad (left) and with pad (right)

Table 7. AISI 308 results

Series	$\lambda / (\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1})$	σ_λ / λ	$R_{c,tot} \times 10^3 / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$\sigma(R_{c,tot}) / R_{c,tot}$	R^2
No PAD	7.66	2.3 %	1.48	5.5 %	0.6666
With PAD	14.60	4.2 %	2.09	2.8 %	0.9776
With PAD	19.10		2.39*		-

* imposed value from PMMA calibration

Table 8. Relative weights of the total contact resistance $R_{c,tot}$ on the total measured thermal resistance R_{tot}

Sample	s / mm	$R_{tot} / (\text{m}^2 \cdot \text{K} \cdot \text{W}^{-1})$	$R_{c,tot} = 2,09 \times 10^{-3} \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$	$R_{c,tot} = 2,39 \times 10^{-3} \text{ m}^2 \cdot \text{K} \cdot \text{W}^{-1}$
			$R_{c,tot} / R_{tot}$	$R_{c,tot} / R_{tot}$
SA	15.0	3.09	67.7 %	77.4 %
SB	20.1	3.53	59.2 %	67.8 %
SC	25.1	3.78	55.3 %	63.3 %

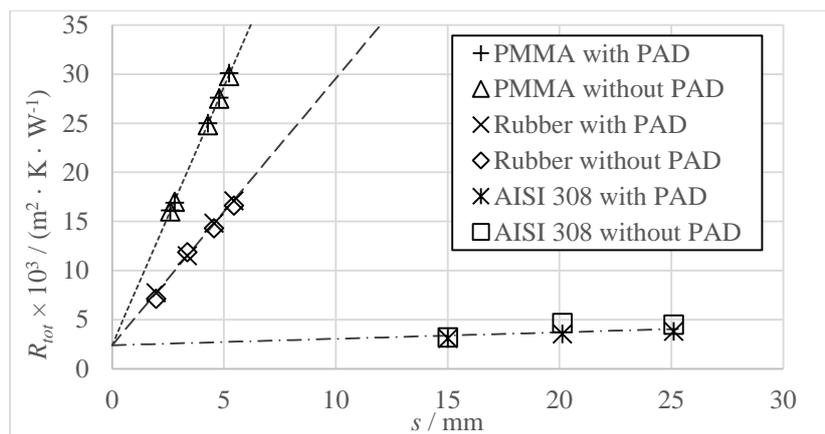


Figure 7. All measured sets, trend lines for reference conductivity values (PMMA and AISI 308) and average measured conductivity for the rubber set

4 Conclusions

This study shows the results of thermal conductivity measurements with the HFM technique of three sets of samples of different materials with various thicknesses, with and without the use of a 1 mm silicone thermal pad applied to the upper and lower surface of the sample as buffer, to analyze if

advanced sample preparation techniques can be substituted with faster methods without sacrificing the measurement accuracy. While the pad leads to minor differences in the measurement of the PMMA samples, it increases the linearity of rubber with irregular surface finish and AISI 308 steel samples. In particular, the thermal conductivity of AISI 308 can only be estimated with the use of the pads, with an obtained error equal to 5.5 % with respect to the reference value. Moreover, the measurement uncertainty, although quantifiable both for the instrument and the regression procedure, cannot describe alone the uncertainty of the measurement, while the linearity of a multiple thickness test, which implies a consistent value of thermal contact resistance between samples, seem to be a more meaningful descriptor. Finally, these considerations are applicable only when samples of multiple thicknesses are available. If only one sample is available, a contact resistance value must be imposed, but the results show a larger uncertainty in its evaluation and, while the thermal pad reduces the difference across samples with different finishes, the application of a general thermal resistance term in measurements which are composed mainly by this term can lead to significant errors.

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