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## Flexible Buffer Materials to Reduce Contact Resistance in Thermal Insulation Measurements

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#### ABSTRACT

Thermal insulation test methods approach their lower limits as thermal resistance falls below 0.1 m<sup>2</sup>·K/W. This is the minimum value specified in ASTM C 518 (ASTM International, 2010b) while ASTM C 177 (ASTM International, 2010a) proposes about 0.06 m<sup>2</sup>·K/W. Nevertheless these are the test methods, along with their ISO equivalents, required by Australasian building codes and directed at many products and materials with thermal resistance on the low side of 0.1 m<sup>2</sup>·K/W. Alternatives, such as ASTM E 1530 (ASTM International, 2011), cover much lower resistances but require carefully prepared small specimens and very-high contact pressures and are therefore largely unsuitable for both technical and compliance reasons. For these low resistances, the insulation test methods face large errors because of interface resistance between specimen and the apparatus hot and cold plates. Staying with C 518, the problem can be avoided by using direct measurement of the test specimen surface temperatures, but this is difficult, has its own accuracy issues, and is often impractical for commercial laboratories. This technique is generally used in conjunction with interface materials such as flexible foam between the specimen and the hot and cold plates, to enhance contact and also provide an access path for temperature sensors. The alternative prospect of using these interface materials to ensure good specimen contact has been studied, in conjunction with a simple two-step thermal resistance determination based on the difference between presence and absence of the test specimen.

This article presents results of a study using this difference approach for the measurement of 12 highly conducting materials, including sheets of aluminum, phenolic, HDPE, MgO, bonded rubber and cork granules, PMMA, and compressed wood fiber. For each material, repeated measurements have been performed with four different interface or "buffer" materials: PVC, silicone, EVA, and nitrile. Silicone sponge provides the most uniform results, consistent with a measurably lower hysteresis. The difference technique yielded a lower indicated thermal resistance than direct measurement by between 0.003 and 0.01 m<sup>2</sup>·K/W, with some variation depending on the specimen surface characteristics and to a lesser extent on the choice of buffer. Larger differences were associated with bowed, uneven or roughly surfaced specimens. The difference-technique results have greater variability, but they may be seen as better estimates of the actual specimen resistance, as contact resistance is much lower for soft-surface interfaces. An interface resistance of up to 0.01 m<sup>2</sup>·K/W is large enough to be of significance in many thermal measurements.

Keywords: thermal resistance, measurement, test methods, contact resistance, interface resistance.

#### 1. INTRODUCTION

A laboratory seeking to have a capability for thermal resistance measurement around and below 0.1 m<sup>2</sup>·K/W, might consider the option of an ASTM E 1530 apparatus. However E 1530 covers the range of 0.001–0.04 m<sup>2</sup>·K/W, leaving a gap up to the low end of C 518 where neither method is optimum. E 1530 has a strong focus on interface resistance. Test specimens, typically 50 mm in diameter, are required to have tightly-controlled flatness ( $\pm$ 0.025 mm). Gimbal joints are required at the load application points to maintain an even contact pressure of at least 70 kPa, and typically over 200 kPa, which is 100 times higher than C 518 contact pressures. A heat-transfer medium is recommended for the contacting faces. Finally, the calculation method is by comparison with a known similar material measured under similar conditions so that extraneous sources of thermal resistance, from the interface as well as internal to the apparatus, are automatically accommodated.

The attention that E 1530 affords to issues of interface resistance serves to highlight the absence of such

considerations in the insulation standards, where the focus is on high-performance materials. However, energy performance regulations require data to be available for all building elements, not just insulations. In Australia, the primary source for standardized tabulations of such data is the AIRAH Handbook (AIRAH, 2013). It lists the thermal resistance of 10 mm gypsum plasterboard, e.g., as 0.059 m<sup>2</sup>·K/W. Values like this are typical of the raw data required for building thermal modeling software. In general, it is also a requirement that such data be obtained by measurements in compliance with the Australasian & New Zealand insulation standard, AS/ NZS4859.1 (Standards Australia, 2006). This standard in turn calls up the ASTM test methods C 518 and C 177 and their ISO equivalents. Australian Building Regulations are also tied directly to AS/NZS4859.1. This provides a disincentive for the use of alternative techniques such as E 1530 or the laser flash standard E 1461 (ASTM International, 2013) which might not be completely excluded but have "last resort" status at best. In any event, there are definite advantages with the "insulation" test methods, which assess a sizeable sample of a "product" rather than a small, carefully prepared test specimen. Real-world products may be composite, textured, layered, profiled, or otherwise complex, may have uneven surfaces, and may lack small-scale uniformity. These are all manageable issues with the insulation test methods, particularly the larger apparatus.

It is therefore not surprising that the CSIRO thermal laboratory has an ongoing focus on the thermal insulation test methods, ASTM C 518 in particular, and has a particular interest in accurate measurement of low-resistance specimens. This study of the use of flexible buffer materials, and measurement by difference, is indicative of this emphasis and has provided an opportunity for closer scrutiny of a technique we have used for some time.

#### 2. BACKGROUND

#### 2.1 Measurement techniques

From a technical perspective, a C 518 apparatus optimized for operation up to 10 m<sup>2</sup>·K/W might be expected to struggle with measurement more than two orders of magnitude lower. Unfortunately, this is not widely acknowledged. The specifications for commercial apparatus often quote conductivity range rather than resistance and do not generally impose low-resistance limits. Lower thermal resistances are associated with higher heat flows, which would seem to be no harder to measure accurately. This rational of course ignores the issue of interface resistance.

ASTM C 518 does contain a clause requiring rigid or high-conductance specimens to have careful surface preparation. It states that surfaces should be made

flat and parallel to the same degree as the heat flow meter and that plate-mounted temperature sensors "may" be used if thermal resistance is sufficiently high. In fact few test specimens could be supplied, or modified, to achieve this flatness. As for temperature sensors, commercial apparatus generally have only the plate-mounted option. In order to use external sensors, the laboratory must set up a separate measurement system running in parallel with the builtin instrumentation. This introduces data management and calibration issues, especially for thermocouples where a different wire calibration and a different cold junction compensation system would be required. The European standard, EN 12664 (BSI, 2001), has a focus on materials of medium to low thermal resistance and presents considerable detail on techniques for external temperature measurement which are suggested for thermal resistances of up to 0.5 m<sup>2</sup>·K/W in some cases. EN 12664 also emphasizes specimen uniformity, especially flatness.

Despite the difficulties, the use of external temperature sensors is an effective technique to bypass interface resistances. In order to accommodate and protect the sensor wires, sheets of foam or similar material are generally used between either side of the specimen and the test plates. The alternative of machining grooves in the specimen to carry the sense wires may be feasible, but is often impractical, and introduces other errors. In any case, specimens of this type are potentially heavy, friable, abrasive and of uncertain thickness uniformity. Therefore, the foam sheets also protect both the apparatus and the specimen. Corsan and Williams (1980) have studied the potential errors with this technique. More recently, Campbell and Rose (n.d.) describe its use with concrete test specimens in a Netzsch Application Note. This technique has been employed for many years with test materials such as rammed earth and brickwork walling weighing as much as 500 kg (Zsembery, Clarke, & McNeilly, 1996). Our older C 518 rigs use in-house data acquisition, including four precision thermocouples on each side of the test specimen as an integrated softwareselectable option. EN 12664 refers to "contact sheets". The term "interface material" is also used, at the risk of confusion with heat transfer pastes used for semiconductor cooling and similar applications. The term "buffer" sheets, or materials, is used to describe the foam material used in this way. These buffer sheets have significant thermal resistance, and they buffer the specimen both physically and thermally in the test apparatus.

With careful setup and uniform specimens, external thermocouples may be used quite successfully in conjunction with buffer sheets. Plate and specimen surface temperatures are consistent and effectively define the thermal resistance of each buffer as well as the test specimen, in proportion to the temperature differences. Interface resistance appears considerably reduced, as might be expected from contact with a soft buffer material. This consistent behavior suggests that the external thermocouples might actually be dispensed with. The difference between two measurements – buffers in conjunction with test specimen and buffers alone – represents the test specimen resistance, along with some smaller contact resistance terms. These two measurements are more straightforward than using external thermocouples. This option is described in C 518, proposing the use of a "thin sheet of suitable homogeneous material", measured separately.

Brzezinski and Tleoubaev (2002) and Tleoubaev and Brzezinski (2007) have further developed an alternative dual-measurement technique originally suggested by Filla and Slifka (1997). With two test specimens identical except in thickness, a pair of measurements provides sufficient data to factor out the interface resistances, assuming these are constant. Although novel and effective, the technique requires a pair of uniform materials, available in different thickness having identical conductivities.

It is observed that for specimens with significant non-uniformity in thickness, external temperature readings can be quite variable, raising concerns about how representative any chosen sensor locations might be. Corsan and Williams (1980) confirm these large variations in temperature by computation. In comparison to a determination of thermal resistancebased temperature readings at a few chosen locations, a determination based on subtracting the thermal resistance of buffer sheets would seem to have some immunity from local effects, because all components are intrinsically spatially-averaged.

Buffer sheets also offer plate protection with heavy or abrasive specimens, even if interface resistance is not an issue. Polyurethane panels faced with granite chips are a notable example.

#### 2.2 Theoretical considerations

A heat flow meter (or guarded hot plate) apparatus is a means of applying Fourier's heat conduction equation in a constrained way. Ideally there will be uniform (usually rectilinear) geometry, uniform plate temperatures, unidirectional heat flow, and a uniform test specimen. Under these conditions, the temperature difference divided by the heat flow over the metered area is a direct measure of the total thermal resistance ( $R_t$ ) between the points of temperature measurement. Figure 1 shows one of the alternative geometries which uses two heat flow meters, one imbedded in each plate. To illustrate the types of resistance term, a buffer material is shown only on top of the test specimen in Figure 1.



Figure 1. Heat flow meter (HFM) apparatus with specimen and a buffer material present.

For a direct measurement where no buffer material is used, the test specimen is in contact with both plates, and there is a simple series combination of thermal resistances.

The relationship can be expressed as

$$R_{t} = R_{p_{1}} + R_{p_{1}-s} + R_{s} + R_{s-p_{2}} + R_{p_{2}}$$
(1)

where

 $R_{\rm t}$  is the total thermal resistance between the plate temperature sensors;

 $R_{p_{a}}$  is the internal thermal resistance of plate 1;

 $R_{p_{1}-s}$  is the interface thermal resistance between plate 1 and the specimen;

 $R_{\rm s}$  is the specimen thermal resistance;

 $R_{s-p_2}$  is the interface thermal resistance between plate 2 and the specimen;

 $R_{p_{n}}$  is the internal thermal resistance of plate 2;

 $R_{t}$ , as measured, is a good approximation for  $R_{s}$  only if the other four terms in Equation (1) are small. Commercial apparatus relying on embedded plate temperature sensors almost invariably measure  $R_{t}$ . The other four terms do not exist if external temperature sensors are used at the specimen surfaces. However, this is difficult to do without introducing other errors, especially because the temperature difference across the specimen may be relatively small.

Values for  $R_{p_1}$  and  $R_{p_2}$  are not generally published in equipment specifications. There is evidence that they are indeed quite small, especially for modern equipment which generally employs metal plate facings. In any case, they are extremely difficult separate from the interface resistances  $R_{p_1-s}$  and  $R_{s-p_2}$  and for most purposes can be lumped together. These terms are widely ignored and are insignificant in many cases. Where test specimens have surface characteristics similar to calibration specimens, the interface resistance is already factored into the calibration. However, this is really valid only for cases where the test and calibration specimens have similar thermal (and interfacial) properties.

The interface resistances are intended here to include classical "contact resistance" as might be measured between flat mating surfaces of a certain roughness with a certain contact pressure. However, they also encompass gross effects arising with realworld specimens where imperfect flatness leads to voids, airspaces, and generally uneven contact. The literature provides very little guidance as to values for interface resistance that might apply for a typical thermal conductivity measurement. The range may be very broad even though insulation test apparatus use a relatively narrow range of contact pressures, generally ~2 kPa. Tleoubaev and Brzezinski (2007) report a value of 0.003 m<sup>2</sup>·K/W as the total of the interface and internal terms in Equation (1) for highlyflat Pyroceram. Values much higher than this are conceivable. At a 0.3-mm void, the local thermal resistance will be approximately 0.01 m<sup>2</sup>·K/W.

Extending the components of Figure 1 to the case where a lower buffer sheet is also present, the total thermal resistance,  $R_{t_i}$ , is composed of a long chain of series components as follows:

$$R_{t_{i}} = R_{p_{1}} + R_{p_{1}-b_{1}} + R_{b_{1}} + R_{b_{1}-s} + R_{s} + R_{s-b_{2}} + R_{b_{2}} + R_{b_{2}-p_{2}} + R_{p_{2}}$$
(2)

When the buffer sheets are measured alone, the total thermal resistance  $R_{t_a}$  is

$$R_{t_{ii}} = R_{p_1} + R_{p_1 - b_1} + R_{b_1} + R_{b_1 - b_2} + R_{b_2} + R_{b_2 - p_2} + R_{p_2}$$
(3)

The difference is therefore

$$R_{\text{diff}} = R_{t_i} - R_{t_{ii}} = R_{b_1 - s} + R_s + R_{s - b_2} - R_{b_1 - b_2}$$
 (4)

 $R_{\rm diff}$  has only three interface terms, all involving a soft-material interface.  $R_{\rm b_1-b_2}$  is subtractive although expected to be the smallest term, as it is for a soft-soft interface. The key question is therefore whether  $R_{\rm diff}$  (by calculation) is a better approximation for the specimen thermal resistance,  $R_{\rm s}$  than  $R_{\rm t}$  (by direct measurement). This study compares measurements of  $R_{\rm t}$  and  $R_{\rm diff}$ , under the assumption that both will lead to an overestimate of  $R_{\rm s}$  because some interface terms are present in both cases. However the softmaterial interface terms should be quite small and  $R_{\rm diff}$  should be much closer to  $R_{\rm s}$ .

A buffer material must have carefully considered stiffness and resiliency. It needs to be soft enough to afford low interface resistance and to accommodate test specimens with uneven surfaces. This requires

some compression at the high points, where the local contact pressure will be significantly higher, in proportion to the spatial extent of these areas. This, however, also means that the material will be sufficiently soft for there to be some residual compression even when contact is uniform, because as a first approximation, deformation will be proportional to pressure (in accordance with Hooke's law). For difference measurements with uniform specimens, the same thickness reduction occurs for the specimenpresent and the specimen-absent measurements, and therefore any compression cancels out. Compression will however be spatially irregular when any sample non-uniformity exists, making this cancellation somewhat inaccurate. Further considerations are the consistency (repeatability) of apparatus-loading pressure and the potential for hysteresis and creep in the buffer material.

A greater variance has been observed when buffer materials are used, beyond what would be expected because of the uncertainty implications of subtracting two numbers. The experimental program incorporated repeat measurements to study the extent of this variance. The sources of uncertainty appear to be complex and are addressed empirically at this stage.

#### 3. SELECTION OF TEST SPECIMENS

Table 1 summarizes the 12 specimens chosen for study. All were 600 mm<sup>2</sup>. Specimens 1 and 2 were aluminum sheet, which is so conductive that thermal measurement is overwhelmed by interface resistance. Specimen 1 was flat while specimen 2 had a bow of ~3 mm in one plane. The instrument plates flattened the bow out almost completely, but it was of interest to see what differences remained between the two sheets. The resistance of the HDPE and phenolic paper specimens was two orders of magnitude higher than the aluminum although still so low that interface effects predominate. Both had smooth flat faces, offering good surface contact at least. All other specimens were resistive enough for C 518 measurement to be conceivable. Specimen 5 was composed of fused rubber and cork granules, predominantly rubber. Although uniform and flexible, it was quite rough on both sides. Specimen 6, the MgO board, was smooth on one side, rough on the other. It also had a 1-mm bow at the midpoint which was largely eliminated by plate pressure. Specimens 7 and 8 were PMMA (acrylic) specimens from different sources with a slight thickness difference. Specimen 9 was a commercial flexible PVC flooring material. It was used as a pair of 1.5 mm sheets back to back with the softer base surfaces outermost and the decorative upper surfaces in contact. It was a composite material with four or more layers, one with glass-fiber reinforcing, and had

| Table | 1. | The  | 12 | test | specimens  |
|-------|----|------|----|------|------------|
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| Specimen number | Description                                  | Thickness (mm) | Density (kg/m³) | Thermal resistance (m <sup>2,</sup> K/W) | Generic thermal conductivity (W/m·K) |
|-----------------|--|----------------|-----------------|--|--------------------------------------|
| 1               | Flat aluminum sheet                          | 2.5            | 2,720           | 0.00001                                  | 220                                  |
| 2               | Bowed aluminum sheet                         | 3.0            | 2,680           | 0.00001                                  | 220                                  |
| 3               | HDPE clear sheet                             | 1.5            | 960             | 0.003                                    | 0.50                                 |
| 4               | Phenolic paper board                         | 1.6            | 1,430           | 0.006                                    | 0.27                                 |
| 5               | Granulated rubber & cork<br>underlay         | 3.2            | 650             | 0.028                                    | -                                    |
| 6               | MgO board                                    | 15.9           | 1,440           | 0.028                                    | -                                    |
| 7               | PMMA (Acrylic) A                             | 5.8            | 1,190           | 0.031                                    | 0.19                                 |
| 8               | PMMA (Acrylic) B                             | 6.1            | 1,130           | 0.032                                    | 0.19                                 |
| 9               | Flexible PVC flooring<br>(pair of sheets)    | 3.0            | 760             | 0.033                                    | -                                    |
| 10              | "Masonite" hardwood                          | 5.4            | 950             | 0.038                                    | 0.14                                 |
| 11              | Corrugated polypropylene<br>("fluteboard") A | 3.3            | 170             | 0.062                                    | -                                    |
| 12              | Corrugated polypropylene<br>("fluteboard") B | 5.0            | 180             | 0.081                                    | -                                    |

Note: Thermal resistance is derived from generic thermal conductivity where this is known, otherwise from direct measurement.

limited compressibility associated only with the bottom foam layer.

Specimen 10 was a typical board of Masonite material with one very smooth and one rough-textured surface. Specimens 11 and 12 were examples of corrugated polypropylene "twin-wall" sheet. Specimen 11 was a thinner, light-duty material, as used in signage, with thinner walls and closer flutes. Both were sealed at the ends to prevent air movement through the flutes.

#### 4. EVALUATION OF BUFFER MATERIALS

Details of the buffers are given in Table 2. All were evaluated as pairs with one on either side of the test specimen. The PVC flooring material used as a buffer was identical to that used as a test specimen, with a back-to-back pair giving a total thickness of 3 mm. The silicone sponge was a grade described as "mediumsoft" and "low compression set". The EVA and nitrile foams were both of much lower density but product specifications were not available.

During the measurement program it became apparent that the buffers differed not only in terms of consistency in results but also in terms of the specimen thermal resistances they suggested, presumably as a result of differing contact resistance. Differences in compressibility and resilience of the foams were thought to be relevant and worthy of investigation. ASTM D 1056 (ASTM International, 2007) provides guidance for making such assessments on rubber foams.

Compressibility is measured as the force required for 25% compression and "compression set" is evaluated

as the rate of recovery after 22 hours of compression. A test protocol was devised to adapt the intention of these tests to a deflection and time frame that is more appropriate for buffer materials. A 50 mm steel disk resting on a sample of buffer material was loaded progressively up to about 3 kPa, followed by a 60-minute hold and progressive unloading to form a set of hysteresis results. Results are shown in Figure 2. The lower curves show the progressive initial loading and deflection, expressed as a percentage of thickness. The upper curves show the deflection as the load was progressively removed 60 minutes later. There are large differences in hysteresis, the implications of which are considered later.

#### 5. THERMAL RESISTANCE MEASUREMENT RESULTS

Each specimen was measured three times directly (with no buffer material present) and three times with a pair of each of the buffers. Each pair of buffers was also measured three times by itself. A 6K temperature difference was used for all direct measurements to reduce the very high heat flows. Measurements involving the PVC buffers were performed at 10K, the silicone sponge at 14K, and the EVA and nitrile buffers at 20K temperature difference.

A buffered result is obtained by subtracting a buffers measurement from a specimen-plus-buffers measurement, as described in Equation (4). Because three values were obtained for each of these measurements, the subtraction can be performed in nine different ways to obtain nine thermal resistance

| Buffer number | Description                            | Thickness (mm) | Density (kg/m³) | Thermal<br>resistance (m <sup>2.</sup> K/W) | Thermal conductivity<br>(W/m·K) |
|---------------|--|----------------|-----------------|---|---------------------------------|
| 1             | Flexible PVC flooring (Pair of sheets) | 3.0            | 760             | 0.035                                       | 0.087                           |
| 2             | Silicone sponge                        | 6.5            | 440             | 0.081                                       | 0.080                           |
| 3             | EVA foam                               | 4.2            | 31              | 0.12  | 0.035                           |
| 4             | Nitrile foam                           | 7.3            | 71              | 0.21  | 0.035                           |

Table 2. The four buffer materials.

Note: Properties apply for a single buffer (on either side of test specimen).



# Compression Removed Compression Applied

Figure 2. Compression performance of the four buffer materials. Lower curve shows deflection when compression applied, upper curve when load removed after 60 minutes.

values. It might be statistically more rigorous to perform eighteen individual measurements for the construction of nine difference pairs but the difference measurements as calculated do represent a complete set of possible outcomes from the measurements performed.

Results are presented in Figure 3, showing nine calculated results for each buffer material. The first sketch shows thermal resistance results for the two aluminum sheets. The directly-measured

value was consistently just above 0.008 m<sup>2</sup>·K/W for both, unaffected by the flatness difference between them. It is apparent that this resistance is almost entirely composed of interface components, as the sheets themselves account for only 0.00001 m<sup>2</sup>·K/W, effectively zero on the scale used. The scale is fine enough however to reveal considerable disparity in results for difference measurement with buffers. It also shows a correlation between the consistency of results

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with any particular buffer and the degree of hysteresis in the material as indicated in Figure 2. Specifically, the silicone provides the least variability, followed closely by the EVA. The PVC is significantly worse and the nitrile is worse still. This trend is apparent throughout. For the aluminum sheets, measurements with the silicone buffers at least provide some consistency and suggest a thermal resistance of around 0.003 m<sup>2</sup>·K/W, less than half the value by direct measurement and therefore much closer to the correct value. Also evident in the figure is another recurrent characteristic of the nitrile buffers – a lower indicated thermal resistance.



Figure 3. Thermal resistance of specimens 1–12 by direct measurement and with four buffer materials.

Although the range for nitrile is very large, i.e. from 0.004  $m^2$ ·K/W to the impossible negative value of -0.004, the mean is reasonably close to the correct (near-zero) value. Nitrile has the highest compressibility of all the buffer materials but only by a small margin. It appears likely that the very "accommodating" nature of the foam, manifest as its high hysteresis, may be accompanied by the potential for very-low contact resistance.

Initial results for EVA foam with specimens 1 and 2 were not consistent with the other 10. Because these were the only low-emittance specimens, radiation transparency was suspected. Consistency for the EVA returned, as in Figure 3, after the aluminum was sprayed flat black.

Although specimens 3 and 4 had considerably higher thermal resistances than specimens 1 and 2, they were clearly still too low for C 518 measurement, even via difference measurement with buffer materials. The expected values were 0.003 m<sup>2</sup>·K/W for the HDPE and 0.006 m<sup>2</sup>·K/W for the phenolic paper. Measured values were either too high or too great in variance, as the figure shows. As with the aluminum sheets, results for these materials using the silicone buffers were at least reasonably consistent and much closer to the correct value.

The other eight specimens had a thermal resistance of  $\geq$ 0.03 m<sup>2</sup> K/W, high enough for measurement by C 518 to be considered. The plots of Figure 3 display results in order of increasing thermal resistance and show a trend toward increasing consistency as interface resistance become a smaller proportion of the total. There are some important similarities, and differences, through the series. Accepting the ever-present high variance for all measurements involving the nitrile foam, guite consistent results are evident for the two samples of acrylic, including the relative performance of the four buffers. Results for the two fluted plastics are similarly consistent, although the relativities seem to be slightly different. In this case there appears to be a slightly higher thermal resistance with the silicone buffers. The results are consistent enough to suggest that the interface resistances depend to some extent on interaction between the surface characteristics of both the hard and the soft materials in contact.

Specimens 5 and 6 provide further indication of the variability in interface resistance. For these materials, the gap between direct and difference measurement is particularly high. In the case of the underlay material, both surfaces of the bonded rubber and cork granules were quite coarse. Presumably, there was a particularly high interface resistance when this surface was in contact with the apparatus plates. In contact with the buffer materials able to mold to this roughness, it is understandable that the interface resistance was reduced by a greater amount than with flat-surfaced

materials. Additional support to this notion is provided by the results for the nitrile foam buffers. With these, the suggested thermal resistance was particularly low relative to the other buffers. This would be quite consistent with the notion of a highly compliant nitrile surface adapting to a coarse specimen. Results in the case of the MgO board are perhaps more dramatic but also less clear. It was not possible to measure the extent to which the bow in the sample was flattened out within the apparatus, where the loading pressure would have adopted a complex profile across the specimen related to compressibility of the buffer material. Whether by flattening of the specimen or compression of the buffer, results suggest that there may have been some residual airspace (or perhaps very low contact pressure) with three of the buffers, which was not present with the more-compliant nitrile. This is suggested by the dramatically lower thermal resistance obtained with the nitrile buffers for this particular specimen. For these two samples, measurement by difference using the three more-consistent buffers has produced a thermal resistance that is ~0.01 m<sup>2</sup>·K/W lower, a reduction of the order of 30% for these two materials. Beyond this, results for nitrile buffers suggest that even these thermal resistance values are an overestimate. Unfortunately, the ubiquitous variability of the nitrile measurements precludes any real confidence in a quantifying the effect. Accurate thermal conductivity data is not available for either material to provide corroboration.

Specimens 9 and 10 provide further insight into the way surface characteristics are likely to affect the interface resistance. In the case of specimen 9, the PVC flooring, direct and difference measurement produced the closest agreement of any of the specimens, with the difference measurement producing only slightlylower thermal resistance values for all buffers. This is consistent with the fact that, of the 12 specimens, only the PVC flooring material had soft surfaces. There is likely to be a lower contact resistance in all measurements cases, whether the contact is with the hard apparatus plates for direct measurement or with other soft buffer materials for difference measurement. One might expect the soft-soft interface between PVC flooring and a buffer to produce a slightly-lower contact resistance, which is what the data suggests. However, it must be remembered that precise information on the scale of these effects is not apparent because direct measurement also includes unknown terms for apparatus internal resistance on both plates. Results for specimen 10 are for a material with a smooth and a rough surface. The relative performance of the buffers follows a similar pattern to the other materials. The reduction in thermal resistance achieved by difference measurement is consistent in which it is roughly intermediate between the rough-surface and smoothsurface values observed for the other materials.

#### 6. DISCUSSION

The difference-measurement technique has shown that it is not without difficulties. Buffer materials must have qualities that sit between excessively soft, leading to significant and variable compression and excessively hard, in which case they may not offer useful reduction in interface resistance. The choice of thickness is similarly balanced between too much thermal resistance and too little thickness in which to accommodate the hard and possibly uneven specimen surface. Only four materials have been tried; there are many other possibilities. Measurements with nitrile material have suggested that softer and more compliant materials might afford very-low interface resistance but the high variability would need to be overcome. Composite materials (of which PVC flooring is an example) might provide better overall performance than simple compositions. Foams loaded with high-conductivity fillers may perform better.

For the technique to provide reproducible results, the apparatus plates must provide reproducible pressure on specimens. This also applies for direct measurements but is even more important with the difference technique because of the need to subtract the results from two measurements which must be made under conditions as close to identical as possible.

No attempt was made to allow the buffer materials to rest in an unloaded state between measurements. The hysteresis issue only became apparent after the test program had well progressed. Undoubtedly some buffers would have been reused before they had fully recovered and would have recompressed further than previously, with thermal resistance commensurately lower. Table 3 suggests that this effect would have been most significant for the nitrile where high thickness, low conductivity, and large hysteresis all combine unfavorably.

**Table 3.** Change in thermal resistance because of change inthickness equivalent to measured hysteresis at 2 kPa loading foreach pair of buffer materials.

| Buffer material                              | Thermal<br>resistance<br>(m²·K/W) | Change in<br>thickness<br>(%) | Change in<br>thermal<br>resistance<br>(m²·K/W) |
|--|-----------------------------------|-------------------------------|--|
| Flexible PVC<br>flooring<br>(Pair of Sheets) | 0.070                             | 1.27                          | 0.0009   |
| Silicone sponge                              | 0.162                             | 0.37                          | 0.0006   |
| EVA foam                                     | 0.24                              | 0.49                          | 0.0012   |
| Nitrile foam                                 | 0.42                              | 1.46                          | 0.0061   |

A recent round robin of 27 laboratories (APLAC, 2010) considered samples of 25 mm glass fiber and 15 mm solid acrylic, the latter having a nominal

thermal resistance of 0.09 m<sup>2</sup>·K/W, lower than the official ASTM C 518 minimum. The laboratories reported uncertainties of up to 2.9% for the glass fiber and 4% for the acrylic. For the glass fiber, at 20°C mean, most laboratories achieved results within their stated uncertainty limits. However, for the acrylic, the range of results was 94% of the median and the interguartile range was 11.8%. Clearly, accurate measurement of the acrylic was more difficult than presumed, with the values from a few laboratories being distant outliers. It is notable that the generic thermal conductivity of acrylic is usually quoted at around 0.18–0.19 W/m·K but the median value for the round robin was 0.16 W/m·K. The difference might in part be explained by interface resistance. For the two 6-mm acrylic specimens of this study, difference measurement using the silicone buffers suggested a mean thermal conductivity of ~0.17 W/m·K while for direct measurement the suggested value was closer to 0.15 W/m·K.

### 7. CONCLUSIONS

The thermal insulation test methods, developed with higher-performing materials in mind, are guite compromised at the low end of their measurement range by the presence of interface resistance. Alternative methods such as ASTM E 1530, appropriate for small uniform samples of muchhigher conductivity, are not suitable for many building and industrial products. The use of conforming buffer materials at the interface between sample and apparatus plates provides a means of reducing interface resistance and extending the lower range of measurement. Although it introduces the requirement for measurement by difference, subtracting the thermal resistance of the buffer materials measured separately, the technique can clearly lead to improved measurement of specimens that have low to verylow thermal resistance. The method is also much simpler for most laboratories than the most-common alternative, which is to use external thermocouples requiring additional instrumentation.

The utility of the technique hinges on the fact that the interface terms are lower when there is at least one soft material at each interface. However, it is not without difficulties. Variance is higher, and the derived result is still an overestimate because some interfaceresistance terms remain. Softer and more compliant buffer materials result in greater variance, so that the choice of buffer material is a compromise and may depend on the specimen, especially if it has spatial irregularities and non-uniformities.

Among the four buffer materials studied, a silicone sponge produced the most consistent results, combining relatively high thermal conductivity and low hysteresis. Consistent performance as a thermal buffer was clearly associated with low hysteresis and the nitrile, with the highest hysteresis, produced unacceptable results. However, high hysteresis is also associated with compliant surfaces and did appear to give nitrile the lowest interface resistance. These conflicting attributes might be resolved with alternative materials, perhaps composites, which offer the best features of both. In any case, the effects of hysteresis might be reduced by pre-conditioning buffer materials under zero-load for an extended period before use.

Using the technique, measured thermal resistance is typically lower by an amount between 0.003 and 0.01 m<sup>2</sup>·K/W. Interface resistance components of this size are large enough to be of consequence in many thermal measurements.

#### REFERENCES

- AIRAH. (2013). AIRAH Technical Handbook, Australian Institute of Refrigeration, Air-Conditioning and Heating Inc. Melbourne, VIC: Author.
- APLAC. (2010). APLAC TO53. Final Report, Thermal Insulation Proficiency Testing Program.
  Abbotsford, BC: Asia Pacific Laboratory Accreditation Cooperation.
- ASTM International. (2010a). ASTM C177-10. Test method for steady-state heat flux measurements and thermal transmission properties by means of the guarded-hot-plate apparatus. Annual Book of ASTM Standards (Vol. 04.06). West Conshohocken, PA: Author.
- ASTM International. (2010b). ASTM C518-10. Test method for steady-state thermal transmission properties by means of the heat flow meter apparatus. Annual Book of ASTM Standards (Vol. 04.06). West Conshohocken, PA: Author.
- ASTM International. (2007). ASTM D1056-07. Specification for flexible cellular materials – sponge or expanded rubber. 2007. Annual Book of ASTM Standards, (Vol. 08.01), West Conshohocken, PA: Author.
- ASTM International. (2011). ASTM E1530-11. Test method for evaluating the resistance to thermal transmission of materials by the guarded heat flow meter technique. Annual Book of ASTM Standards, (Vol. 14.02). West Conshohocken, PA: Author.

- ASTM International. (2013). ASTM E1461-13. Test method for thermal diffusivity by the flash method. Annual Book of ASTM Standards (Vol. 14.02), West Conshohocken, PA: Author.
- Brzezinski, A., & Tleoubaev, A. (2002). Effects of interface resistance on measurements of thermal conductivity of composites and polymers. *Proceedings of the 30th Annual Conference on Thermal Analysis and Applications* (pp. 512–517). Bowling Green, KY: NATAS.
- BSI. (2001). BS EN 12664:2001. Thermal performance of building materials and products. Determination of thermal resistance by means of guarded hot plate and heat flow meter methods. Dry and moist products of medium and low thermal resistance. London, UK: Author.
- Campbell, R., & Rose, M. (n.d.). *Thermal Conductivity Measurements of Concrete Using the Heat Flow Meter (HFM) and Guarded Hot Plate (GHP) Methods*. NETZSCH Instruments North America. Retrieved from http://www.netzschthermal-analysis.com
- Corsan, J. M., & Williams, I. (1980). *Errors* associated with imperfect surfaces in standard hot-plate thermal conductivity measurements. Teddington, UK: National Physical Laboratory.
- Filla, B. J., & Slifka, A. J. (1997). Thermal conductivity measurements of pyroceram 9606 using a high-temperature guarded-hot-plate. *Thermal Conductivity 24 / Thermal Expansion 12, Proceedings of the 24th International Thermal Conductivity Conference and 12th International Thermal Expansion Symposium* (pp. 85–96). Lancaster PA: Technomic Publishing Company.
- Standards Australia. (2006). AS/NZS 4859.1 (2006) Materials for the thermal insulation of buildings – General criteria and technical provisions. Sydney, NSW: Author.
- Tleoubaev, A., & Brzezinski, A. (2007). Errors of the Heat Flow Meter Method Caused by Thermal Contact Resistance. *Thermal Conductivity 29 / Thermal Expansion 17, Proceedings of the 29th International Thermal Conductivity Conference and 17th International Thermal Expansion Symposium*. Lancaster, PA: DEStech Publications.
- Zsembery, S., Clarke, R. E., & McNeilly, T. (1996). Thermal transmission properties of Australian clay bricks. *Masonry International*, 10(1), 30–34.