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Design of a Guarded Hot Plate for Measuring Thin Specimens of Polymer and Composite Materials

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ABSTRACT

The National Physical Laboratory (NPL) has developed a new design of guarded hot plate apparatus specifically for absolute measurements on thin specimens of medium thermal conductivity materials, such as the polymer composites that are becoming more widely used in aerospace and other advanced manufacturing sectors. Although NPL has an existing measurement facility based on a commercially manufactured apparatus conforming to ASTM E1530, this current facility is not based on an absolute measurement technique and is not able to provide the low measurement uncertainty or flexibility that is increasingly being demanded by industrial users of this NPL service.

The target specification for this new NPL guarded hot plate is the measurement of materials with thermal conductivity in the range 0.1–10 $W \cdot m^{-1} \cdot K^{-1}$ using specimens with thickness of 1–20 mm and over the temperature range −100°C to 250°C. This is achieved using a new design of guarded heater plate and a temperature-controlled environmental chamber. This chamber can be evacuated and specific gases can be introduced, enabling measurements on porous materials under a wide range of environments. It can be used in either a single specimen or a double specimen configuration, and with specimen diameters of either 75 mm or 50.8 mm that is used in many older styles of comparative measurement apparatus. During the commissioning of this new measurements facility, it is planned to investigate various approaches for reducing thermal contact resistance between the specimen and plates. This facility will then provide the flexibility for meeting a wider range of requirements from industrial customers.

Keywords: thermal conductivity, guarded hot plate, polymer composite, ASTM E1530.

1. INTRODUCTION

There is a growing demand for accurate thermal conductivity data from medium thermal conductivity materials such as composites, polymers, and ceramics. These data are required for modeling the heat management in advanced engineering applications such as those found in the nuclear, oil and gas, processing, automotive, and aerospace sectors.

For example, continued improvement in lightweight aerospace components requires development of new composite materials with better thermal performance, and the next generation of nuclear power plant will require a range of accurate thermophysical property data. Also, in the United Kingdom, the exploitation of remaining pockets of oil reserves is prohibitively expensive due to the high cost of pipeline, and lower uncertainty in thermal conductivity data for the polymers used as thermal insulation would allow reductions in the weight and diameter of the pipeline.

Although NPL currently has one of the most comprehensive thermal conductivity measurement capability in the world, the majority of industrial users are not able to provide samples of medium thermal conductivity material in sufficiently large dimensions for our current national standard apparatus (Stacey, 2002; Stacey, Salmon, & Simpkin, 2010; Stacey & Williams, 1999). It is therefore necessary to use a commercially built comparative apparatus conforming to ASTM E1530 (ASTM, 2011), which NPL has evaluated to have an estimated measurement uncertainty of ±7.5% ($k = 2$) and is therefore not able to provide the uncertainty of ±3.0% or better that is required by many advanced manufacturing companies.

The new facility developed by NPL and described in this article will make steady-state measurements on medium thermal conductivity materials and with sample dimensions that are practical for advanced industrial composite materials and applications. It should provide expanded scope and a significantly

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reduced measurement uncertainty, as well as the flexibility to make measurements in different gas environments and under compressive loads.

2. APPARATUS DESIGN

2.1 Principle of the guarded hot plate technique

The guarded hot plate is an absolute measurement technique based on steady-state unidirectional heat flow and creates a temperature gradient across a specimen, by sandwiching it between a heater plate and an isothermal cold plate. In the case of the new design of guarded hot plate described in this article either a double or a single specimen configuration can be used. In the double specimen configuration, there is a specimen on either side of the heater plate and then cold plates on either side of the two specimens. In the single specimen configuration, there is a specimen and cold plate on one side of the heater plate and on the other side are insulation, an auxiliary heater plate matched to the temperature of the primary heater plate, another layer of insulation, and another cold plate. This stack of specimens and plates is mounted horizontally, so that heat flows vertically from the heater plate, through the specimen(s) to the cold plate(s).

The temperatures on either side of the specimen are measured, and the heat flux is determined by the amount of electrical power supplied to the heater. The electrical power is maintained at a fixed value to obtain an appropriate steady-state temperature gradient through the specimens. Then knowing the specimen thickness and the area through which the heat passes, the apparent thermal conductivity of the specimen can be calculated.

This simple calculation, based on Fourier's law, assumes that all of the power supplied to the heater goes through the specimen to the isothermal cold plate, and that none of the heat is lost to the environment at the edges of the heater plate and/or specimens. The primary means of ensuring that all the heat flows through the specimen, normal to the plane of the plates, is a lateral guard heater that surrounds the main "metering area" heater and is separated from it by a small gap. This lateral guard is controlled to be the same temperature as the metering area by means of an electronic controller responding to the output of a differential thermocouple that crosses back and forth across the guard-center gap. Therefore, as the immediate surroundings of the metering area are at the same temperature as the metering area, there is no net lateral heat flow.

In the single specimen configuration, an auxiliary heater plate is also controlled to be at the same temperature as the metering area by means of an electronic controller and differential thermocouple in order to ensure that there is no net heat flow normal to the plane of the heater plate and in the opposite direction to the specimen.

To further reduce heat transfer at the edges of the specimen, the international standard ISO 8302:1991 (ISO, 1991) recommends that the environment directly around the specimen/plate stack be controlled at the mean measurement temperature. This is particularly important when there is a large difference between the laboratory temperature and the mean specimen temperature.

2.2 Specification

The target specification for this new NPL guarded hot plate is absolute measurements on materials with thermal conductivity in the range 0.1–10 $W{\cdot}m^{-1}{\cdot}K^{-1}$ using specimens with thickness of 1–20 mm and over the temperature range −100°C to 250°C. This is the same range as NPL currently offers to industrial customers using a commercially built comparative apparatus, which conforms to ASTM E1530 (ASTM, 2011) and is calibrated against a number of NPL and European certified reference materials, including Pyrex glass (Williams & Shawyer, 1991) and Pyroceram 9606 (Salmon, Roebben, Lamberty, & Brandt, 2007). However, this existing testing service is no longer considered sufficiently accurate or flexible to meet future requirements.

The only comparable absolute guarded hot plate design previously built by a national measurement institute is at Physikalisch-Technische Bundesanstalt, Germany (Hammerschmidt, 2002; Hemminger & Jugel, 1985), but this system is limited to a minimum specimen thickness of 5 mm, maximum thermal conductivity of 6 $W{\cdot}m^{-1}{\cdot}K^{-1}$ and also in terms of environment and compressive load.

The target measurements uncertainty for the new NPL guarded hot plate is ±3.0% or better, and it should also be able to be evacuated so as to have specific gases introduced, enabling measurements on porous materials under a wide range of environments. It is to be used in either a single specimen or a double specimen configuration, and with either a specimen diameter of 75 mm or the 50.8 mm used in many older styles of comparative measurement apparatus. During the commissioning of this new measurements facility it is planned to investigate various approaches for reducing thermal contact resistance, so the design needs to allow for different approaches to temperature measurement and the use of viscous fluids between the specimen and plates.

2.3 Guarded heater-plates

The new heater plates are 75 mm in diameter, constructed of aluminum and include a design of center-guard gap profile that allows for measurement of thin specimens of material by having a gap with a width of 0.2 mm exposed to the specimen. The internal cross-sectional profile of the gap is diamond shaped, similar to that used in a guarded hot plate designed by NIST for high temperature insulation (Zarr, Flynn, Hettenhouser, Brandenburg, & Healy, 2005), and is 4.9 mm at the widest point inside the plate. This design maximizes the thermal resistance of the gap, while still enabling tight definition of the area through which heat is applied to the central metering section of a thin specimen.

Each heater plate is assembled from four aluminum components, two forming the central metering section and two forming the lateral-guard section. The upper center and upper guard components have a thickness of 2.35 mm, while the lower components have a thickness of 2.65 mm and grooves machined in their inner surfaces for resistance heating wires. The difference in thickness of the upper and lower components is so that the heating wires will be located at the midpoint through the thickness of the heater plate. The two central metering section components are disc shaped and the two lateral-guarded section components are ring shaped. Heater wire is mounted in grooves in the lower components and then the upper components placed on top. These assemblies are fixed together using aluminum interference pins forced into holes aligned through the plates. The central metering section and lateral-guard section are held apart at three points by spacer blocks mounted inside the gap cavity. These spacers have cross-sectional dimensions of 2 mm $\times$ 3 mm and are made of a calcium silicate ceramic (density of 1400 $kg{\cdot}m^{-3}$ and thermal conductivity about 0.49 $W{\cdot}m^{-1}{\cdot}K^{-1}$) in order to maintain the separation between the sections, but with the minimum of cold-bridging across the gap.

This thinner and simpler design is achieved by utilizing a “hard” anodized coating on the four aluminum component plates. This clear hard anodizing is to a depth of 0.05 mm and is achieved using sulfuric acid according to standard BS 5599 (BS, 1993). Testing at NPL has shown that this type of anodizing maintains its integrity and provides sufficient electrical insulation over the required temperature range.

The use of this hard anodized coating enables an uninsulated resistance heater wire to be mounted in the heater plate, rather than using a mineral-insulated cable heater. This has the advantage of being able to deliver a much higher density of heat flux, and also the lower minimum bend radius enables a much more tightly packed and well-defined pattern to be used for mounting the heater. The pattern used can be seen in Figure 1 and thermal modeling carried out during the design phase predicted this pattern and a 2.35 mm thickness of aluminum should provide reasonable temperature uniformity (0.05 K) at the surface of the heater plate.

The resistance heating wire used in the central metering section and lateral-guard section of the plates is 80% nickel and 20% chromium and has a diameter of 0.25 mm. The nominal resistance of the wire at 20°C is 21.7 $\Omega{\cdot}m^{-1}$, and it has a nominal temperature coefficient of 0.00011 $\Omega{\cdot}\Omega^{-1}{\cdot}K^{-1}$. The advantage of using heating wire instead of cable heaters is a higher density of heat flow. However, a practical disadvantage is that the current carrying wires need to be attached to the heater wire at the point where it leaves the plate, which creates a mechanically weak point and means the plates have to be handled with great care. There is also insufficient room to attach additional noncurrent carrying wire to measure the potential drop across the heating wire within the central metering area at the point where it leaves the metering area (as would be done with a larger heater-plate). This means that the leads used for measuring the potential drop also need to be attached where the heating wire leaves the edge of the plate. This arrangement requires a small correction to be made to the measured potential drop based on knowledge of the resistance of the heater wire and the length of the heater wire passing through the guard section. Another potential issue

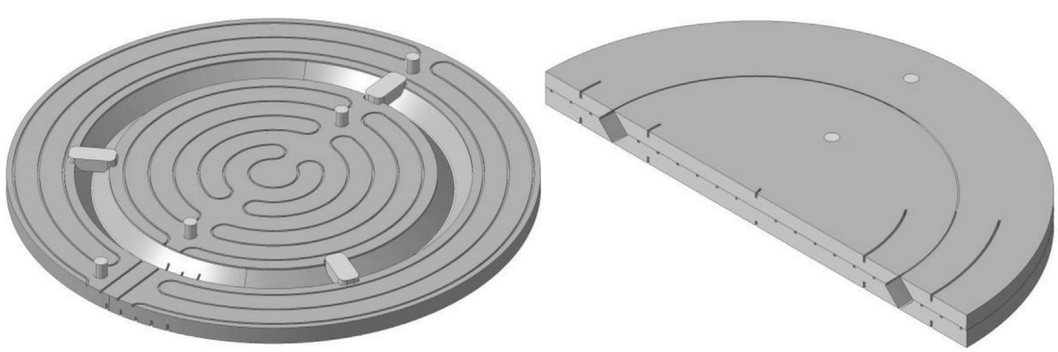

Figure 1. Horizontal and vertical slices through guarded heater-plate design.

when the heater plate is used in a double-specimen configuration is that heat from the wires entering the plate through the sides of the grooves will need to pass through two additional layers of coating to travel upward compared with downward. This may cause a small discrepancy in temperature differences across the two specimens that would need to be considered in the measurement uncertainty.

The temperature of the heater plate surface is measured using E-type mineral insulated thermocouples with an external sheath diameter of 0.25 mm and sensitivity of about 60 μV·K^{-1} at 20°C. The initial design shown in Figure 2 included one thermocouple on each side of the heater-plate and a differential thermocouple (bare wire of 0.075 mm diameter and 10 junctions on each side) mounted in groves in the anodized surface and used for controlling the lateral-guard section heater. However, in practice this design proved too difficult to assemble due to the fragility of the differential thermocouple chain during welding and installation. Using a larger diameter wire for the differential thermocouple on this size of plate would have increased the amount of cold-bridging across the center-guard gap and affected temperature uniformity.

The second design of heater-plate shown in Figure 3 has the traditional style of guarded hot plate differential thermocouple replaced by four additional mineral insulated thermocouples (external sheath diameter of 0.25 mm) on each side of the heater plate. These additional thermocouples, four in the central metering section and four in the lateral guard section, are connected as a differential within the isothermal box. This arrangement is not as sensitive as the original design, but has been calculated to still be more than sufficient for measurements on the medium thermal conductivity materials primarily targeted. However, it will lead to high uncertainty if the use were extended to low density thermal insulation.

The new apparatus design includes the use of two identical guarded heater plates. One is used as the primary heater plate, which is in contact with the specimen. The other heater plate is used as an additional auxiliary guard in the single specimen configuration in order to prevent heat flow from the back of the primary heater plate.

The features of this new design will contribute to providing a well-defined heat flux from a heater plate having a narrower exposed edge and lower thermal mass than previous designs. When combined with the actively controlled cold plates, environmental chamber, and an optional heated edge guarded, this should provide a reduced uncertainty in the rate of heat flux density passing through the specimen and contribute to reduced overall measurement uncertainty in the thermal conductivity measurements.

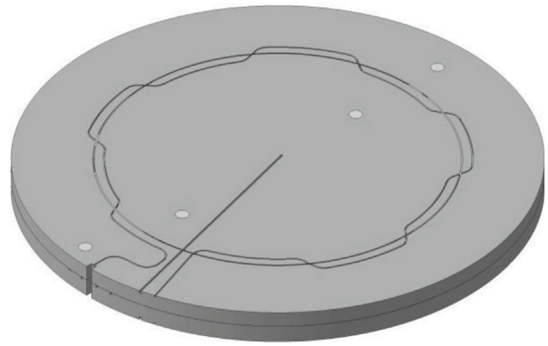

Figure 2. Initial design of heater plate.

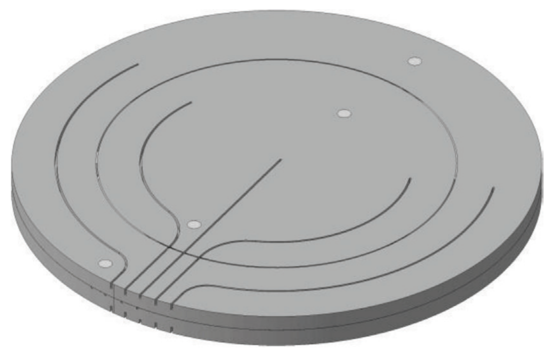

Figure 3. Second design of heater plate.

2.4 Specimen temperature measurement

Accurate thermal conductivity measurement on thin specimens of medium conductivity material involves several difficult technical challenges, including precise measurement of specimen surface temperature in regions of high heat flux. Although the guarded heater-plate and cold-plates have thermocouples in their surfaces, there will be significant contact resistance between these thermocouples and the surfaces of the specimen. Therefore, the intended primary method for measuring the temperature of the specimen surfaces will be to use bare-wire butt-welded thermocouples with a diameter of 0.075 mm, which will be mounted in a 0.1 mm diameter grove machined all the way across the both flat surfaces of the specimen. These butt-welded thermocouples have no visible feature where the junction is formed (having the appearance of a continuous wire) and will be positioned across the specimen with their junction in the center.

The issue of thermal contact resistance still remains significant, especially when measuring materials with a high coefficient of thermal expansion, as differential expansion will cause these specimens to bow. Therefore, the most important phase of commissioning this new facility will be investigating the use of various types of thermal contact media in order to ensure even heat flow from the heater plate to the specimen. This may need to include the use of a thin film between the contact medium and the heater

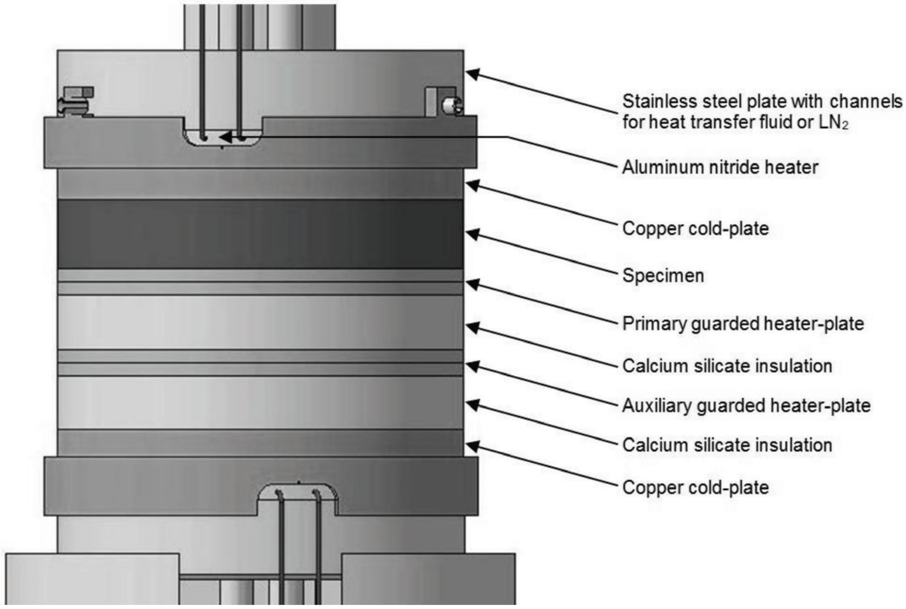

Figure 4. Stack of plates, insulation and specimen in single-specimen configuration.

plate to prevent any fluid based contact media from entering the center-guard gap.

2.5 Cold plates

The isothermal cold plates are included in Figure 4 and consist of three separate components. Heat transfer fluid (specifically formulated silicone oil: −40°C to 250°C) from a heat/chiller recirculating unit or liquid nitrogen is passed through channels in stainless steel plates, which provides the first order of temperature control. Adjacent to these plates are disc-shaped electrical heaters manufactured from aluminum nitride and which provide a finer level of temperature control. The third components are 10 mm thick copper plates, which are in contact with the specimen. The copper provides a high thermal mass for increased stability and a uniform surface temperature. These plates have a mineral insulated E-type thermocouple (0.25 mm diameter) mounted in each flat surface, one adjacent to the heater and one adjacent to the specimen. They are easily removable from the stainless steel plate, so as to allow for handling situations where a specimen becomes unintentionally adhered to the cold plate.

2.6 Environmental chamber

The stack of plates and specimen(s) is mounted within an environmental chamber (Figure 5) in which air or another gas is maintained at the same temperature as the mean specimen temperature. This is similar to a previous NPL guarded hot plate (Stacey, 2002; Stacey & Williams, 1999) and is done in order to minimize the heat loss or gain from the edge of the stack and down the heater and thermocouple leads. However in this guarded hot plate, the environmental chamber consists of a double-walled vacuum chamber in which liquid nitrogen or silicone oil from the heater/chiller recirculating unit can pass between the walls of the chamber and thereby maintain a specified temperature. The heat transfer fluid enters the chamber walls at the top and then flows down a spiral path created by fin welded between the inner and outer walls.

The chamber can be evacuated and a specified gas backfilled or bled through during a measurement. If required, measurements can also be carried out under low pressure or vacuum conditions, but this introduces additional measurement issues due to the significantly increased thermal contact resistance between the specimen and plates.

In addition to the heat transfer fluid in the walls of the chamber, there is also a cable heater mounted near the top of the inside of the chamber, and it is used to give some additional control over the gas temperature inside the chamber. The cable heater is not mounted directly to the inside of the chamber wall, but is fixed to three thin vertical rods mounted at about 10 mm from the wall, so that it heats the air and not the chamber. This type of cable heater is normally designed to be mounted in contact with a high conductivity material, but as it is rated at 700 K above the maximum target temperature (300°C) then this is considered a reasonable application. The position of this cable heater inside the top of the chamber will help create temperature stratification in the gas and thereby reduce the overall convection. Thermocouples suspended in the gas will be used to control the heater so that the gas at the same height as the specimen will be close to the mean specimen temperature.

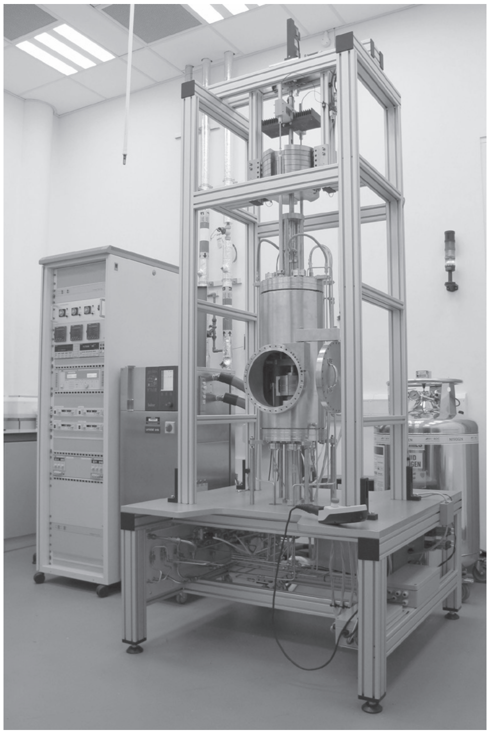

Figure 5. New environmental chamber with external insulation panels removed.

When the target mean specimen temperature is above the 250°C that can be reached by the chamber walls, then an additional heated edge guard can be mounted around the specimen stack. This edge guard is made from a nickel cylinder with a cable heater wound around its outer surface. It is mounted via high density calcium silicate brackets from two vertical columns either side of the plate/specimen stack.

During operation, the whole chamber is surrounded by insulation panels, which help reduce the energy used to maintain the chamber temperature and also act a safety barrier to prevent laboratory staff coming in contact with the chamber.

2.7 Compressive loading system

A compressive load is applied to the specimen during the thermal conductivity measurement in order to ensure good thermal contact between the specimen and plates. A value of 150 kPa has been selected as the nominal compressive load, as this is the same as in the comparative apparatus that NPL currently uses in accordance to ASTM E1530 (ASTM, 2011). The load is provided by a set of weights above the chamber that are mounted on a vertical rod connected to the upper cold plate. The upper cold plate and weights are raised and lowered using a motorized drive system, which is automatically decoupled when the upper cold plate comes to rest on the specimen stack.

A system of balanced bellows is used to allow movement of the vertical rod and weights as they are raised and lowered inside the chamber. In this system, the movement occurs at the midpoint between two stainless steel bellows, which means changes in pressure do not act to extend or compress the bellows and the volume inside the chamber remains constant. The system also means that when one bellows is compressing the other is extending, which means the forces to some extent offset each other and effects on the compressive load applied to the specimen are minimized.

2.8 Specimen thickness measurements

The thickness of the specimen when mounted between the plates is measured by a pair of linear voltage displacement transducers that are mounted on opposite sides (to give an average for the center) of the rod that comes from the top of the upper cold plate and leaves the chamber through the bellows system. The displacement transducers are mounted above the weights and just below where the motorized system decouples when the upper cold plate rests on the stack. This position keeps the transducer sufficiently far away from the sources of heat that may affect their calibration. They are monitored via a high-accuracy calibrated voltmeter that is read by the apparatus control software.

The whole thickness measurement system needs to be calibrated by placing gauge blocks with known thickness and thermal expansion in place of the specimen. The transducer's output are recorded with the chamber at different temperatures covering the range –100°C to 250°C and then another thickness of gauge block is mounted between the plates and the temperature cycle repeated.

3. VALIDATION AND FUTURE DEVELOPMENTS

A series of rigorous performance checks is essential to understanding its limitations and the factors that can influence the resulting measurement uncertainty of a new measurement facility. The approach that will be used in validating this new facility will be along similar lines to that given in clause 2.4 of international standard ISO 8302. This will be followed by a series of validation measurements on an existing NPL reference material of polymethylmethacrylate and European certified reference materials of Pyrex glass (Williams & Shawyer, 1991) and Pyroceram 9606 (Salmon et al., 2007).

There are currently only a few manufacturers in the world that produce commercial instruments for steady-state thermal conductivity measurements on these medium thermal conductivity materials, and these instruments use comparative techniques that require calibration against reference materials. Following validation, the new NPL facility will be used to characterize a wider range of reference materials, including a reference for the thermal resistance of a layered composite.

The frame surrounding the chamber has been designed to operate safely with a compressive load of up to 9 MPa applied to the specimen and a motorized system has been designed to achieve this load. This has not currently been implemented, but may be added at a future date in order to further investigate measurement of thermal conductivity and thermal contact resistance under a wide range of compressive loads.

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