Uncertainties Associated with Imperfect Surfaces in Thermal Conductivity Measurements Using Standard Hot-plate Equipment

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Contents

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The United Kingdom Accreditation Service (UKAS) is recognised by the UK Government as the national body responsible for assessing and accrediting the competence of organisations in the fields of calibration, testing, inspection and certification of systems, products and personnel.

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1 Introduction

1.1 The standard ISO/IEC 17025 used for laboratory accreditation by UKAS requires a laboratory to produce for all measurements an estimate of the uncertainty of its measurements using accepted methods of analysis, through the production and application of suitable uncertainty of measurement procedures. This requirement is relevant not only to any thermal conductivity measurements made but also to any in-house calibrations.

1.2 Several guidance publications on the application of these requirements in the case of particular items of equipment and forms of measurement are listed in *UKAS Publications,* M4. UKAS publication LAB 12 gives general guidance on the expression of uncertainty of measurement in testing.

1.3 This publication (LAB 35) provides guidance for laboratories needing to meet these requirements as applied to the measurement of thermal conductivity. By following this guidance laboratories will be able to demonstrate at assessment that they meet these requirements. Alternative methods may be used provided they are shown to give an equivalent outcome.

1.4 The methods permitted by UKAS for measuring the thermal conductivity of insulating solids in the range 0.01 to 2 W/m K are strictly the recommendations embodied in ISO 8302 : 1991. Additional supporting documents based on ISO 8302 and produced by CEN that are also permitted by UKAS are EN 1946-2, EN 12664 and EN 12667 and EN 12939.

1.5 It is of the utmost importance to ensure that the tolerance on flatness of 0.025% of the maximum plate dimensions specified for both the plates and specimens is not only met but, when feasible, improved upon, since defects (cavities, etc) introduced at the interfaces can seriously impair measurement accuracy. This publication outlines the main features of this problem and provides guidance whereby this source of measurement uncertainty may be kept to a minimum.

2 Effects of defects on temperature distribution

2.1 The defects of concern here are shallow interfacial defects of extended area associated with the departure from flatness of the items mentioned above. Such defects, by altering the thermal resistance at the interfaces, disturb the idealized linear heat flow conditions assumed to exist in the apparatus, leading, under certain conditions, to pronounced temperature variations on the specimen surfaces (Fig. App A-2) and a consequent sharp increase in the uncertainty of the measurement.

3 Variables affecting magnitude of temperature perturbations

3.1 It has been shown in NPL Report QU57 (1980) that the magnitude of these temperature variations on the specimen surfaces depends not only on the depth and lateral dimensions of the defect (or defects) involved, but also on variables such as the conductivities and thicknesses of the specimens and the thermal contact sheets employed. Thus it is found that the temperature perturbations increased in magnitude as the following variables increase in value:

(a) the effective *thermal resistance* of the defect;

(b) the *lateral dimensions* of the defect;

(c) the *heat flux* required to produce the appropriate temperature drop through the specimen which, according to ISO 8302, should not be less than 10 K;

(d) the reciprocals of the *conductivity* and of the thickness of the *thermal contact sheets.*

To preserve accuracy, it is necessary to ensure that as many of these variables as possible are kept to a minimum.

3.2 The general precautions that should be taken become apparent on closer examination of the variables.

(a) Since the effective *thermal resistance* of the defect depends on its depth, and the thermal conductivity of the medium in its immediate vicinity, it is evident that efforts should be made to ensure that all surfaces are as nearly plane as is reasonably possible and that air pockets, which are characterised by a high thermal resistance, are excluded from all surfaces.

(b) The *lateral dimensions* of the defect are important because of the transverse thermal link through the specimen itself, which tends to even out temperature variations on planes normal to the direction of heat flow. This effect is most pronounced when the lateral dimensions of the defect are small, but becomes relatively unimportant when the effective diameter or equivalent dimensions of the defect exceeds about 25 mm. It follows that, compared with defects of extended area, scratches and similar line defects have little effect on the temperature distribution on the specimen surfaces, and it is to extended defects that consideration is given in this publication.

(c) The *heat flux* required to produce a 10 K temperature drop through the specimen becomes large when the thermal conductivity of the material under test is relatively high and / or when relatively thin specimens are used. It may be concluded, therefore, that extra care should be taken in respect of the flatness of all surfaces when the material to be tested has a relatively high conductivity (eg in the range 1 to 2 W / m K) and that as a general rule it is advisable to use specimens not thinner than about 50 mm, unless this is unavoidable.

(d) The temperature variations on the specimen surface are accentuated when the *conductivity* and *thickness of the thermal contact sheets* assume their lowest values. Material of moderately high conductivity having the maximum thickness of 3 mm recommended in the standard is therefore to be preferred if all other requirements are satisfied. However, a factor that must take precedence over other considerations is the compressibility of the thermal contact material used, which should be large enough to ensure that all surface imperfections are taken up and air pockets excluded. As a general rule, silicone rubber sheets fall far short of this requirement and foamed silicone rubber sheets of fine cellular construction are to be preferred, despite their relatively low conductivity of about 0.1 W/m K, unless a more suitable, non-porous, material of higher conductivity is available.

3.3 The effects described in paragraph 3.2 are illustrated in Fig App A-1 which was drawn using the expressions derived in the NPL QU57 (1980) Report. In these graphs the normalized change in the temperature difference between the specimen faces $\varepsilon_t = (\theta_n - \theta_f)/\theta_n$, caused by the presence of a single interfacial defect of depth 0.2 mm and effective lateral diameter of 150 mm, is plotted against λ_2 the thermal conductivity of the specimens under test, for different values of d and t, the thickness of the specimens and thermal contact sheets, respectively. Here θ_f is the temperature drop through the specimen on a line directly beneath the centre of the defect, and θ_n the temperature drop in the unaffected regions well clear of the defect, as illustrated in Fig App A-2. The importance of the compressibility of the thermal contact material may be gauged by comparing Figs App A-1 a and b with c and d.

4 Relationship between temperature variations and measurement uncertainty

4.1 The relationship between these temperature variations, and the level of uncertainty they introduce into the thermal conductivity determination, is discussed in the NPL QU57 Report. It is not straightforward, since it varies with the nature and number of defects present (depressions or protuberances on different surfaces), their lateral shape and area, and their location relative to the positions of the thermocouples on the specimen faces.

4.2 For a pair of similar defects situated on either side of the hot plate, the uncertainty in the measurement could range from close to zero to 0.8 ε_t. In general, however, one would expect it to be less than 0.5 ε_t. Thus, with reference to Fig App A-1, assuming that 3 mm foamed silicone rubber thermal contact sheets are being used with 50 mm thick specimens and that the depth of the interfacial cavities (now filled with foamed rubber) are no greater than the 0.025% of the maximum plate dimension tolerance (0.08 mm for a 305 mm plate) on flatness for plates and specimens allowed in ISO 8302, it can be seen that in no case is the uncertainty caused by the defects likely to exceed 1%, which for most practical measurements may be regarded as satisfactory. It is most important to point out, however, that the thermocouple pairs on the opposite faces of each specimen have been assumed to be directly above one another. Should they be staggered, then the above uncertainty would be increased almost three-fold and only for specimens of conductivity lower than about 0.3 W/m K would it be less than 1%. It should also be realised that the temperature variations on the specimen surfaces, regardless of their size, will not be detected if the defect is located symmetrically between, or above, the thermocouples since now all the thermocouples read the same temperature.

5 Recommendations

5.1 Under no circumstances should measurements be carried out using equipment or specimens whose surfaces deviate from flatness by more than the prescribed tolerances of 0.1 mm. This value should be regarded as a minimum requirement, and closer tolerances be aimed for as follows:

(a) about 0.025 mm for the flatness of the hot and cold plate surfaces;

(b) about 0.05 mm for the flatness of the surfaces of specimens prepared from easily machinable, homogeneous materials. However 0.2 mm tolerance is more realistic for materials more difficult to machine accurately such as masonry and similar inhomogeneous materials.

5.2 Preferably, specimens should not be thinner than 50 mm, especially if their conductivity is rather high.

5.3 It is essential that the thermal contact sheets used should have adequate compressibility to take up all deviations from flatness on surfaces, including their own. A foamed silicone rubber sheet of fine cellular structure (density 24 kg/m^3, thickness 3 mm) has been found satisfactory, although ideally a non- porous material of higher conductivity would be preferred.

5.4 Care should be taken to ensure that thermocouples on opposite faces of specimens are placed directly above one another. Further, the thermocouples should be symmetrically distributed on specimen faces to avoid incorrect averaging of the temperature drop across non-parallel specimens.

6 Conclusions

6.1 If the above tolerances and recommended procedures are adopted then, for the reasons outlined in paragraph 4.1, the uncertainty in the thermal conductivity measurement associated with interfacial defects is unlikely to exceed 1%, even in the case of high density masonry specimens. It will be significantly smaller for lightweight and aerated concrete specimens because of their lower conductivities, whilst for other homogeneous, isotropic materials, which can be machined to closer tolerances, it will become negligible.

6.2 These levels may be compared with the acceptable overall uncertainty bands, which are summarised in Appendix A.

Appendix A

Uncertainty levels in thermal conductivity measurements using standard hot-plate equipment

A1 Normally, with most homogeneous materials, the uncertainty of measurement will be expected to be less than ±3% for conductivities less than 0.15 W/m K and less than ±5% for higher conductivities up to 2 W/m K.

A2 For concretes, the uncertainty levels quoted should lie between the following upper and lower limits:

(a) aerated autoclaved concretes having densities up to 900 kg/m^3 and conductivities up to 0.25 W/m K

±3% to ±5%

(b) lightweight aggregate concretes having densities between 900 and 1500 kg/m^3 and conductivities from 0.2 to 0.6 W/m K

±5% to ±7.5%

(c) concretes having densities between 1500 and 1850 kg/m^3

±7.5% to ±15%

(d) dense concretes having densities above 1850 kg/m^3

±10% to ±20%

A3 Laboratories are advised that the overall uncertainty specified for any particular measurement will depend on the nature of the material being tested, for example on its homogeneity, porosity and surface texture. If the uncertainty levels specified above are unlikely to be achieved, for example with abnormally coarse, porous materials, then the customer should be informed and the test certificate endorsed accordingly.

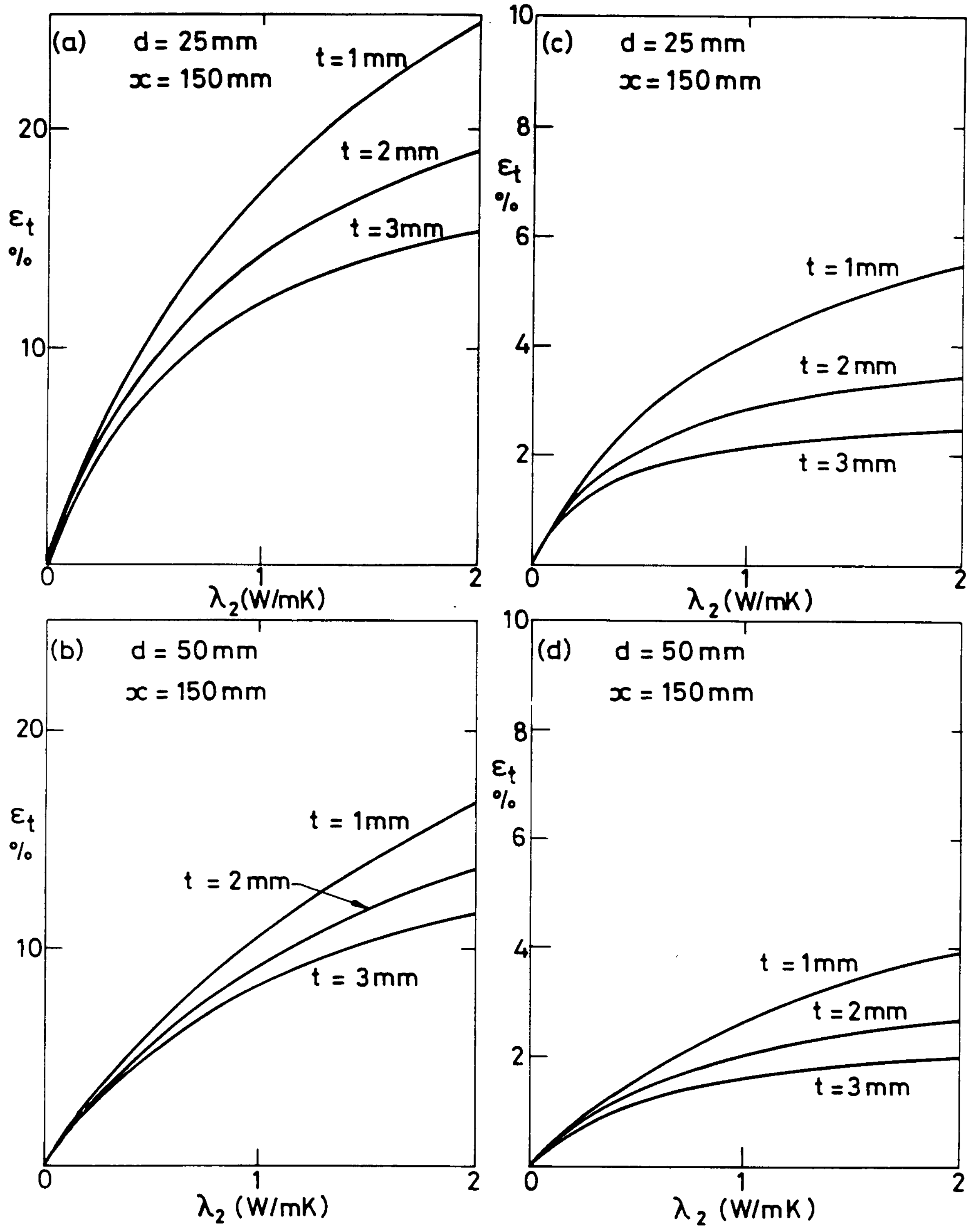

Fig App A–1 **Variation of the parameter ε_t associated with a 150 mm x 0.2 mm deep interfacial cavity with λ_2, the thermal conductivity of the specimens, and d and t, the thickness of the specimens and the thermal contact sheets. The results using an incompressible thermal contact are shown in Figs a and b. The results using foamed silicone rubber sheets are shown in Figs c and d**

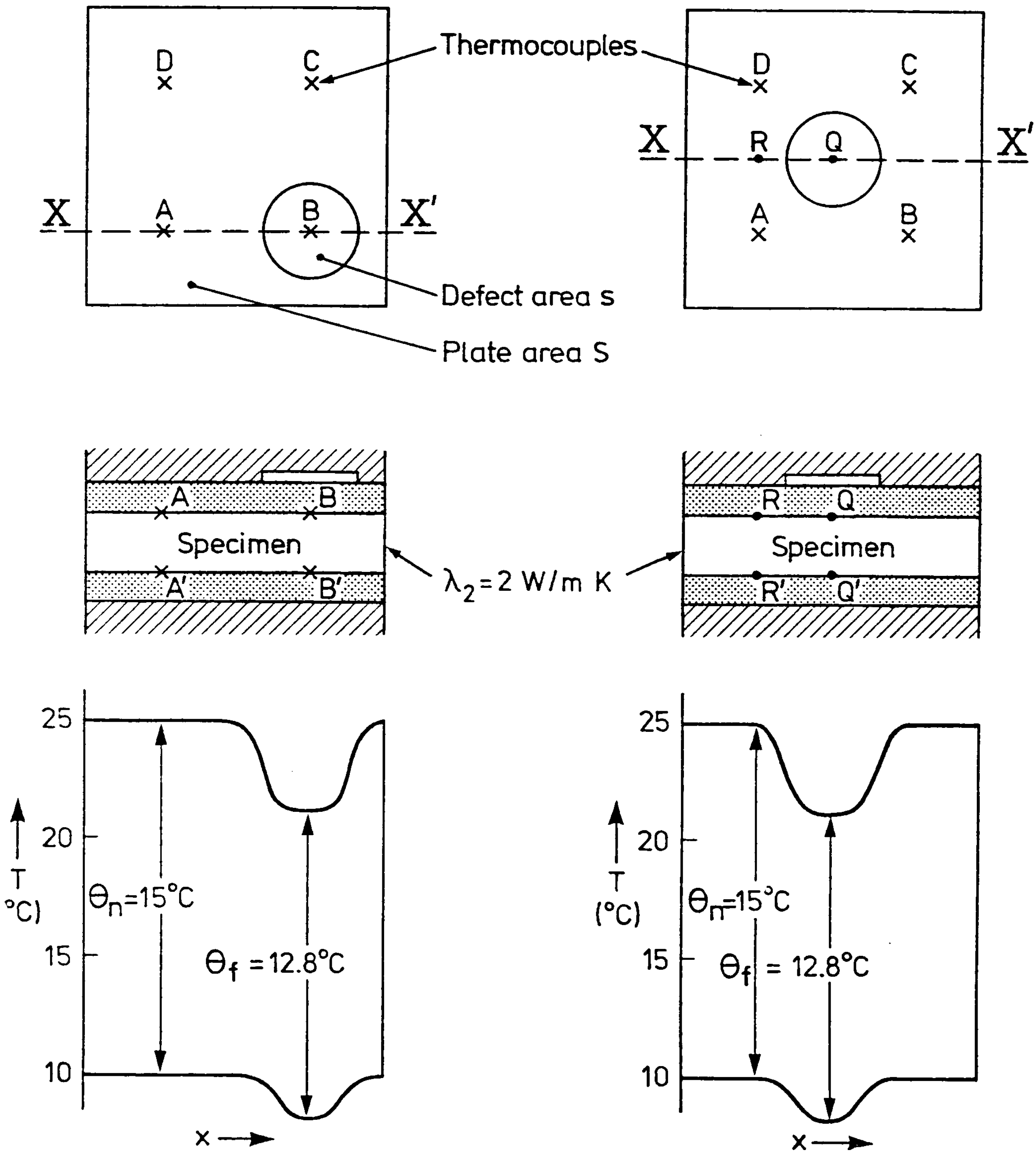

Fig App A–2 **Temperature variations on the specimen surfaces caused by a 100 mm dia x 0.18 mm deep air-filled cavity in the hot-plate surface. The diagrams illustrate how the temperature actually recorded depends on the location of the defect relative to the thermocouples**