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Measurement of heat flow through vacuum glazing at elevated temperature

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Abstract—A method is described for measuring the radiative and gas conductance of vacuum glazing while at room or elevated temperatures. Such measurements are used to determine the residual gas pressure, permitting studies to be made of the accelerated degradation of vacuum glazing. The heat flows associated with this method have been analysed using a detailed finite element model and the technique has been calibrated against both high accuracy measurements on a guarded hot plate apparatus, and calculations of the thermal conductance due to radiative heat transfer. The technique is reasonably accurate—total uncertainties are less than about ± 5% and measurements are reproducible to about ± 2%, for temperatures over the range 20–200°C. Copyright © 1996 Elsevier Science Ltd.

1. INTRODUCTION

Vacuum glazing is a transparent thermal insulator with applications in energy efficient windows. It consists of two sheets of glass separated by a narrow evacuated space [1–3]. The principles of operation of vacuum glazing are identical to those of a Dewar flask—the vacuum eliminates conductive and convective heat flow due to gas between the two glass plates, and low emittance coatings on one or both of the inner surfaces of the structure reduce radiative heat transport to a low level. The manufacture [4] of vacuum glazing requires the formation of an hermetic (leak free) seal around the periphery of the two glass sheets, the establishment and maintenance of a high level of vacuum (below ~0.1 Pa) between the sheets, and the incorporation of an array of high strength mechanical support pillars in order to maintain the separation of the glass sheets under the influence of atmospheric pressure.

The useful service life of vacuum glazing depends on, among other things, the stability of the internal vacuum. The vacuum may degrade, either catastrophically due to a crack in the glass, or over an extended period as the result of outgassing. Measurements of the thermal conductance of vacuum glazing over the past few years using a guarded hot plate apparatus [5, 6] have shown that the thermal conductance of all leak-free samples stored at room temperature is extremely stable [1, 7]. However, vacuum degradation can occur when the samples are aged at elevated temperature [1, 8]. Previously, we have aged samples using conventional ovens, where it has been necessary to cool the glazings to room temperature so that they can be measured using the guarded hot plate apparatus. Although these measurements have been useful for identifying the processes which govern the degradation of the vacuum in these aged samples, a quantitative analysis of these degradation processes has not been possible due to the complicated thermal history of the samples.

In an effort to quantify the adsorption, desorption and diffusion processes, we have developed a method (called the transient technique in this paper) for measuring the thermal conductance of vacuum glazing samples while they are maintained at elevated temperatures. In this technique, a rapid step change in temperature is applied to one side of the vacuum glazing and the small resultant variations in temperature on the other side are measured as a function of time. The total thermal conductance of the glazing can be estimated from these measurements and the thermal conductance due to residual gas obtained by subtracting that due to radiative heat flow. The gas pressure can then be estimated—in particular, over the molecular flow regime the thermal conductance due to gas is proportional to gas pressure.

This paper describes the transient technique of measuring thermal conductance as applied to vacuum glazing. We first describe the apparatus and measurement technique. An analysis of the measurements is then given in terms of a simple lumped parameter model and a detailed finite element model. We show how the finite element model is used to calibrate the transient method against estimates made with a simple lumped-parameter model. This calibration is confirmed by measurements with a high accuracy guarded hot plate apparatus. Following a description of the relation between gas pressure and thermal conductance, we illustrate the application of this technique to measurements on vacuum glazing samples, showing how the degradation processes can be studied.

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NOMENCLATURE

c	specific heat of soda-lime glass sheets
C	thermal conductance
E	activation energy
k	Boltzmann's constant
l	thickness of soda-lime glass sheets
M	molar mass of gas
N_A	Avogadro's number
p	gas pressure
T	temperature
$\langle T \rangle$	average temperature
t	time.

Greek symbols

α	accommodation coefficient
Δ	change in variable
γ	ratio of specific heats
ρ	density of soda-lime glass sheets.

Subscripts

i	initial temperature
s	surface
gap	internal gap in glazing
gas	gas in internal gap
ghp	guarded hot plate
max	maximum rate of change of temperature
Tot	total
vis	viscous
0	copper disc after temperature step
1,2	inner surfaces of glass sheets.

2. APPARATUS AND MEASUREMENT TECHNIQUE

2.1. *Vacuum glazing*

The vacuum glazings constructed at the University of Sydney [1–3] consist of two sheets of glass, typically 4 mm thick, hermetically sealed around the perimeter with a low melting point 'solder glass'. The sheet glass used is a soda-lime composition (approximately 73%SiO_2, 17% Na_2O, 5% CaO, 4% MgO) and is either uncoated, or has a transparent low emittance coating (Pilkington K-glass) [9] on the internal surface in the glazing. The K-glass has a pyrolitically deposited doped tin oxide coating applied to one surface. The two sheets of glass are separated by an array of pillars, which are either made of ceramic material or a high strength metal alloy and are approximately 0.2 mm in diameter and $\sim$0.15 mm high. The pillars are spaced by about 25 mm on a square grid. These dimensions permit several design criteria to be met involving trade-offs between stresses induced in the glass sheets and the thermal conductance of the pillar array [10]. This design process is described in more detail elsewhere [1, 7, 11].

2.2. *Measurement of thermal conductance*

The measurement of local thermal conductance in vacuum glazing presents considerable challenges. In particular, the highly insulating evacuated space is located between two relatively highly conducting glass plates. Moreover, for small area measurements, the absolute amount of heat which flows is quite small. Great care is therefore necessary to avoid effects due to parasitic heat flows, either laterally along the glass plates, or through the wires which make electrical connection to the instrumentation in contact with the sample. There are also significant difficulties in separating the heat flows due to the three different contributing factors: radiation, gas conduction and thermal conduction through the pillars (additional heat can also flow through the bonded edge seal in a practical application of vacuum glazing, but this need not concern us here). Effects due to the pillars can be effectively eliminated by locating them sufficiently far from the measurement point [6]. In all of the work discussed here, the samples are constructed with a small area, approximately 50 mm across, which contains no pillars and is far from the edge seal. The separation of contributions due to gas and radiation requires a detailed knowledge of the infrared optical properties of the internal surfaces, and very careful attention to the way in which the radiative heat exchange between the surfaces is calculated [12].

The thermal conductance of samples of vacuum glazing has been measured using two techniques, one providing high resolution and accuracy at room temperature and the other (the subject of this paper) permitting somewhat less accurate measurements to be made while the glazing is at elevated temperatures. The small area guarded hot plate apparatus used for high resolution measurements was designed and constructed specifically for measuring the thermal conductance of vacuum glazing and has been described in detail elsewhere [6]. In this work, the guarded hot plate was used primarily to provide reference values for a calibration of the transient technique, as described in Section 3.3.

In the second (transient) technique, the thermal conductance was measured by applying a rapid step change of temperature on the exterior of one side of the glazing ('hot' side) and measuring the rate of temperature rise on the other ('cold') side of the glazing. The rate of change of the temperature on the cold side of the glazing can be directly related to the thermal conductance of the glazing, as shown in Section 3. A schematic diagram of the apparatus, including the oven to be described below, is shown in Fig. 1.

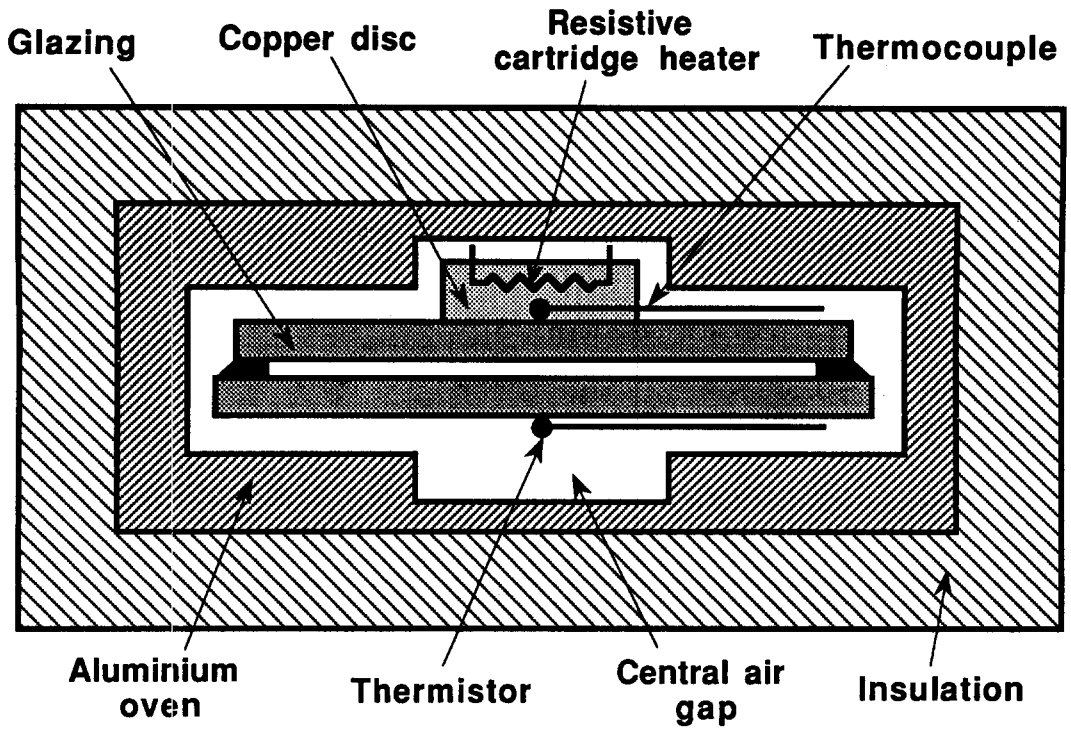

Fig. 1. Schematic diagram of the apparatus used to measure, *in situ*, the thermal conductance of vacuum glazing degraded by baking.

The temperature step on the hot side is produced by applying power to a resistive (cartridge) heater embedded in a 40 mm diameter copper disc. This copper disc is maintained in good thermal contact with the glass by using a heat transfer paste and its temperature is measured with a thermocouple. As show in Fig. 2, the temperature of the copper disk is increased typically by about 20°C within 75 s by applying constant power. The power is then controlled for the remainder of the measurement to keep the temperature of the copper disc roughly constant. The diameter of the copper disc is large enough so that the heat flow through the glazing near the centre of the disc is close to plane parallel, but not so large that heat flow through the pillar array contributes significantly to the measurement. Additionally, the diameter of the disc and the temperature step are small enough that neither the pristine nor the degraded state of vacuum within the glazing is perturbed significantly. In this paper, all data presented are of samples made with glass of nominal thickness of 4 mm.

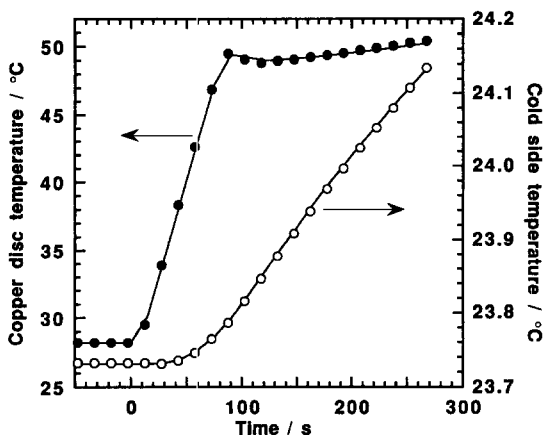

Fig. 2. Example of temperature measurements made in the transient apparatus (data points), with a comparison to modelling results (lines). The temperature of the copper disc (full circles) and that of the cold side (open circles) are shown. The solid lines show the calculated temperatures using the finite element model, where the thermal conductance of the gap is 2.25 W m^{-2} K^{-1}. The heating transient commences at zero time.

The temperature on the cold side of the glazing is measured with an accurately calibrated small (~1.5 mm diameter) semiconductor thermistor, which has a nominal room temperature resistance of either 47 kΩ or 470 kΩ. To reduce the number of significant figures that need to be recorded during a measurement procedure, the thermistor is incorporated in one arm of a Wheatstone bridge. Measurements of the bridge output are converted to resistance and then temperature using the calibration data of the thermistor. Care must be taken to avoid self-heating effects of the thermistor, particularly at high temperatures.

Each glazing is mounted in an oven (see Fig. 1) which is designed to ensure that the lateral temperature non-uniformity across the plates is less than a few K and that the temperature of the plates during a measurement is extremely stable. Central air gaps of 10 mm thickness and 60 mm diameter provide a

reasonably large thermal impedance between the oven and the measurement instrumentation on both sides of the sample.

3. ANALYSIS

3.1. *Lumped parameter model*

Useful physical insights into the transient method can be obtained by considering an idealised case where the temperature step (T_0-T_i) occurs instantaneously, and both glass sheets are assumed to be at uniform temperature. In this 'lumped parameter' model, the rate of temperature increase of the cold side is largest immediately after the temperature step, and is given by:

$$dT/dt|_{max} = C_{gap}(T_0 - T_1)/\rho lc. \quad (1)$$

In this expression, C_{gap} is the thermal conductance of the vacuum gap, l is the thickness of the glass plate on the cold side, and ρ and c are respectively the density and specific heat capacity of the glass.

Equation (1) can be used to obtain an approximate estimate of the thermal conductance of the sample C_{gap} since all other quantities in it are either known, or measured in the transient technique. In particular, the measured value of C_{gap} does not depend on the heat losses from the sample to the surroundings, since the measurement is made when these losses are negligible.

3.2. *Finite element model*

In practice, the distributed thermal impedance of each glass sheet, and the finite time required to apply the temperature step, significantly affect the time dependence of the measured temperature on the cold side of the sample. In order to incorporate these factors correctly, and thus to determine C_{gap} accurately, a finite element model was used. This model was developed using a commercial program STRAND6.1 [13].

The finite element model simulates the heat transfer processes between all relevant system components including: the copper heater block, the cartridge heater (including heat transfer within the heater), the thermocouple which senses the temperature of the copper block, the two glass sheets of the glazing and both surfaces of the aluminium oven. It was found to be unnecessary to include the thermistor on the cold side of the sample. To simplify the calculations and decrease the time of the simulations, a cylindrically symmetric geometry is assumed with the axis perpendicular to the plane of the glazing and running through the centre of the circular copper block. It is therefore necessary to model the cylindrical cartridge heater and the thermocouple wire as discs on the axis of the copper block with volumes and thermal masses equivalent to those in the experiment. This simplification of the geometry does not significantly affect the accuracy of the calculations because copper is so highly conducting relative to other components of the system.

Time-dependent boundary conditions are used to simulate the power input to the cartridge heater. The surfaces of the oven adjacent to the sample are assumed to remain at constant temperature.

Figure 2 compares the results from a simulation with experimental measurements for a particular glazing. The value of C_{gap} used in the finite element model is obtained from measurements on the guarded hot plate apparatus. It is clear that the time delays and changes in temperature associated with the thermal mass and thermal resistance of the various elements are modelled accurately. This statement is true regardless of the thermal conductance of the glazing as shown by similar comparisons for samples with conductance up to more than 30 W m^{-2} K^{-1}.

It is also useful to examine the temperature uniformity on the external surface of the cold glass sheet. Figure 3(a)–(b) shows the profiles of both the temperature and the rate of change of temperature from the central axis to a radial position of 20 mm. The uniformity of these profiles shows that it is not critical where the thermistor is placed. Most importantly, at the time when the maximum rate of change of temperature is recorded (about 120 s after applying power to the cartridge heater) the rate of change of temperature of the cold side is uniform to about 2% over an area with a radius of about 5 mm.

3.3. *Calibration*

Any direct estimate of the thermal conductance from experimental measurements of the temperature of the cold glass surface (using equation (1), for example, or with a more complex curve fitting procedure) depends on the physical and thermal properties of the materials involved, particularly the glass sheets. A critical parameter for this calculation is the specific heat of the glass, which varies significantly over the range of temperatures of interest. We were unable to obtain sufficiently accurate values of this quantity, either from the literature [14–17], or with our own experimental measurements. The temperature dependence of thermal conductivity of glass is also important, although to a lesser extent. We therefore chose to calibrate the transient technique by making measurements on reference samples of known thermal conductance. Using this approach, it is possible to achieve quite high accuracy in the estimates of thermal conductance, since the conductances of the reference samples are directly related to the optical properties of glass in the infrared; such parameters have been measured experimentally using spectroscopic methods and are known quite accurately [12, 18].

The values of thermal conductance of the reference samples for the calibration were obtained from both experimental measurements and theoretical calculations. The experimental values were obtained using the small area guarded hot plate apparatus [6], opera-

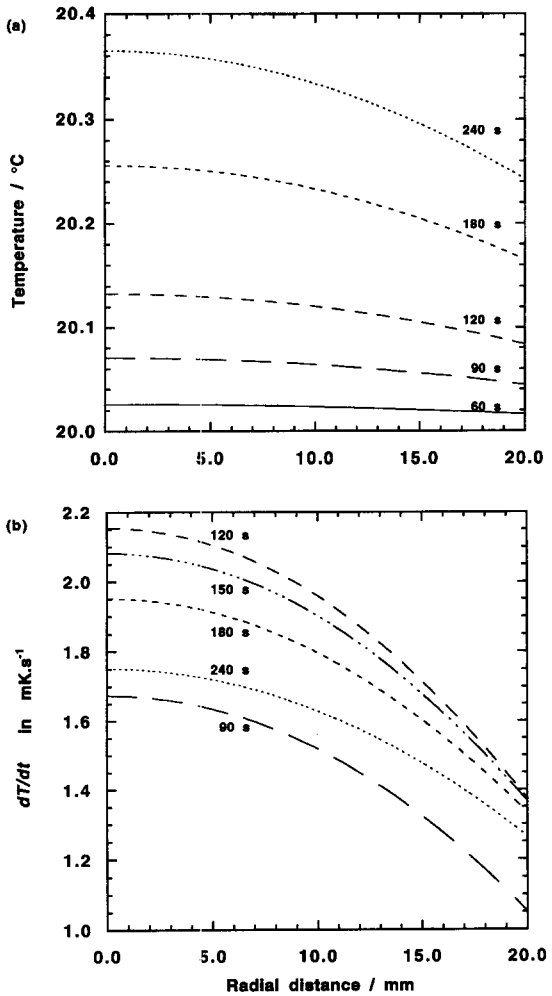

Fig. 3. Finite element calculations of the radial profile of (a) the temperature of the external surface of the cold glass sheet and (b) the rate of change of temperature of this surface. These calculations were made for a sample of vacuum glazing with doped SnO_2 internal surfaces. The time after applying power to the copper disc is indicated on both graphs.

ting close to room temperature. This apparatus has itself been calibrated by comparing measurements and calculations of radiative heat transfer between uncoated glass surfaces [12]. The absolute value of the measurements using the guarded hot plate has been shown to be accurate to better than $\pm 2\%$. Repeated measurements were made on samples with (the three combinations of) uncoated internal surfaces and doped SnO_2 coated (K-glass) internal surfaces, in both the transient and guarded hot plate apparatus.

Values of thermal conductance for the reference samples at temperatures above room temperature were obtained by calculating the radiative heat flow using the wavelength, angular and temperature dependent emittance of both uncoated and doped SnO_2 coated glass. For uncoated glass, this was done in the same way as for the calibration of the guarded hot plate apparatus using accurate values of the optical properties of glass, and the standard Fresnel relations [12]. For the coated glass, this emittance was calculated using a Drude model. Briefly, for the doped SnO_2 surface, three parameters in the Drude model have been determined by Zhang *et al.* [12], through a comparison of the calculated normal reflectance with near-normal measurements over a wavelength range of about 3–20 μm (at room temperature). These calculations have been validated by comparing them with experimental measurements of reflectance as a function of wavelength over a much wider range of wavelengths, and at angles other than normal incidence. Further validation of the optical data and the method of calculating radiative heat transfer was obtained by comparing calculated rates of heat transfer for parallel, facing surfaces with high accuracy measurements of radiation heat transfer made on dynamically pumped samples. These measurements were made with a guarded hot plate apparatus for which the area is accurately known. To calculate accurately the radiative conductance over a range of temperatures, it is necessary to include explicitly the wavelength and angular dependence of emittance in the relevant integrals [12].

This procedure enables the thermal conductance of several reference samples to be determined accurately, over a wide temperature range. The transient technique was calibrated with these samples using only data for which there was no evidence of an increase in gas pressure (as indicated by a slow increase in the measured value of thermal conductance). Finite element calculations—with the conductance of the gap given by the known conductance—were compared with the experimental transient technique measurements, and the room temperature value of the specific heat of soda-lime glass was adjusted in the finite element model until there was agreement between the results of the transient experiment and the calculations of the finite element model, as shown for example in Fig. 2. The temperature dependence of the specific heat was determined by a linear approximation to the data shown by Shand [17]. This yields values of specific heat capacity of soda-lime glass given by $[813 + 1.67T]$ J kg^{-1} K^{-1} (for temperatures T in °C between 0 and 200°C). The value at room temperature is within a few percent of the various values of specific heat of soda-lime glass quoted in the literature [14–17]. The temperature dependence of the thermal conductivity of glass, obtained from the literature [17], was also included in the calculations, although this has a much smaller effect compared with that of the specific heat.

While the procedure described above effectively calibrates the transient technique against known values of thermal conductance, a simple and straightforward means of estimating the thermal conductance for all samples of vacuum glazing also involves the application of the lumped parameter model. This has the advantage of avoiding repeated finite element calculations and associated comparisons of temperature curves. In order to make such estimates, the output

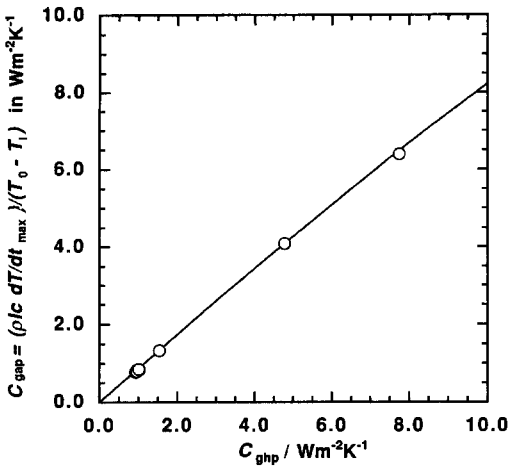

Fig. 4. Thermal conductance measured by the transient technique C_{gap} (using equation (1) in the text) vs the thermal conductance measured on the guarded hot plate apparatus C_{ghp}. The circles are experimental data. The solid line shows a fit through the results of the finite element simulations (individual results are not shown) which have also been analysed using equation (1).

of the finite element model was analysed in a manner identical to the experimental measurements, and equation (1) used to give values of thermal conductance C_{gap}. Using curve fitting techniques, a quadratic relation was then found between C_{gap} and the known thermal conductance measured with the guarded hot plate apparatus C_{ghp}. This relation is shown in Fig. 4 (the solid line), as well as the experimental data for the reference samples (data points). Further modelling has shown that this approach to calibration is valid for values of sample conductance up to more than 30 W m^{-2} K^{-1}.

The experimental data in Fig. 4 were measured at room temperature, except for the point at the highest value of thermal conductance. For this data point, the sample was measured using the transient technique at room temperature and at about 70°C. The known thermal conductance was calculated (using the optical properties of glass) from the increase in radiative conductance of the sample with uncoated internal surfaces. The temperatures of the internal surfaces (of all samples) were determined from measurements made on the exterior of the sample while using the transient technique, allowing for the temperature difference across the glass due to the heat flow.

We note that, as expected, the thermal conductance is significantly under-estimated (by ~20% relative to the known conductance of the gap) by the lumped parameter model. The non-linearity of the calibration, which is evident from the curve and experimental data in Fig. 4, arises because for more conducting samples the maximum rate of temperature rise on the cold glass side occurs earlier in time and hence closer to the period during which the temperature of the hot glass sheet is still increasing. Despite the shortcomings of the lumped parameter model, the curve through the data of the finite element model provides a convenient means of determining the thermal conductance of the gap in any sample of vacuum glazing: initial estimates are made using the lumped parameter analysis (that is, using equation (1), including the temperature dependent specific heat capacity of soda-lime glass) and are subsequently corrected using the quadratic fit to the finite element results shown in Fig. 4.

Figure 5 shows experimental measurements (points) in the transient apparatus of the thermal conductance due to radiation for glazings with uncoated (NN) and SnO_2 coated (KK) internal surfaces. The data have been plotted against the cube of the average temperature of the two surfaces $\langle T \rangle$ (T in Kelvin) since for a small temperature difference across the surfaces ($\Delta T \ll T$) the radiative thermal conductance is proportional to $\langle T \rangle^3$ and the emittance. Measurements and calculations [12] indicate that the effective emittance of the combination of the two surfaces in these samples increases by only about 2% over the temperature range (300–360 K) of these data. This figure also shows calculated values (lines) of thermal conductance for these samples, as determined from measurements at room temperature and the procedure described above, including the temperature dependent emittance. The very good agreement between the experimental and calculated data validates the procedure, described above, to calibrate the transient measurements.

3.4. *Measurement uncertainty*

Both systematic and random uncertainties are associated with the physical and thermal constants related to the glass sheets. It is known, for example, that the thickness of the glass sheets (nominally 4.0 mm) may vary by up to several percent. This has been confirmed by direct measurement and by measuring the mass and area of several representative samples of vacuum glazing. It is also likely that there is a systematic error in the specific heat of glass used in these calculations. This is to be expected given the lack of accurate data available in the literature. This potential systematic error is corrected implicitly using the finite element calibration as described above for conductances up to at least 30 W m^{-2} K^{-1} and temperatures up to about 200°C. At higher temperatures and/or conductances, we would expect the errors to increase since the specific heat is not known as accurately, and the rate of rise of temperature on the cold side of the sample is very large. Fortunately, such conductances only occur in samples which are highly degraded and for which it is less important to know the conductance accurately.

In measuring the temperature step applied to the hot glass sheet, there is an uncertainty of about 1% associated with fluctuations in the thermocouple readings. Effects associated with the non-ideal form of the step temperature increase of the copper block have been corrected implicitly by adopting in the finite element model the same averaging procedure as used

in the experiment for estimating the initial and final steady-state temperatures.

Other significant errors can arise from uncertainties associated with the measurements of the rate of change of temperature on the cold side. It is necessary to calibrate the thermistor accurately, including small departures from the conventional exponential dependence of resistance on inverse temperature. As noted above, it is also necessary to consider the effects of self-heating of the thermistor in the Wheatstone bridge.

The total systematic and random uncertainties in the measurement of thermal conductance have been shown to be less than $\pm 5\%$ for temperatures and sample conductances up to 200°C and at least 30 W $m^{-2} K^{-1}$, respectively. Analysis of repeated measurements for glazings under constant conditions shows that the uncertainty due to random sources is about $\pm 2\%$.

3.5. *Gas pressure in vacuum glazing*

It has been shown [8] that, during high temperature ageing, the thermal conductance of vacuum glazing only changes due to an increase in internal pressure. The emittance of the internal surfaces is never observed to change during such ageing. Estimates of the thermal conductance associated with residual gas within samples of vacuum glazing were therefore made by measuring the total thermal conductance of the samples and subtracting the thermal conductance due to radiative heat transfer. The radiative component was calculated from a best fit to experimental measurements, such as those shown in Fig. 5, for glazing temperatures below those for which any significant increase occurred in gas pressure within the glazing. In addition to the obvious increase of thermal conductance with time, the presence of gas in the gap can also be established by the observation of a significant deviation of the experimental measurements from the calculated radiative heat flow (derived from the optical properties of the internal surfaces, as described in Section 3.3). The best fit to these data—determined for each glazing—is a third order polynomial in T^3 which is constrained to pass through the origin. The coefficients for the terms in the second and third powers of T^3 were determined by a fit to the calculated data and kept constant, while the coefficient for the linear term (in T^3) was found from the fit to the experimental data. This procedure satisfactorily accounts for the point-to-point variations in the emittance of the SnO_2 internal coating of K-glass.

At low pressure where the mean free path of the gas molecules is significantly greater than the spacing between the glass sheets, the thermal conductance due to gas conduction C_{gas} is proportional to gas pressure p [19]:

$$C_{gas} = \alpha\left(\frac{\gamma+1}{\gamma-1}\right)\sqrt{\frac{N_A k}{8\pi M T}}p \qquad (2)$$

where the combined accommodation coefficient

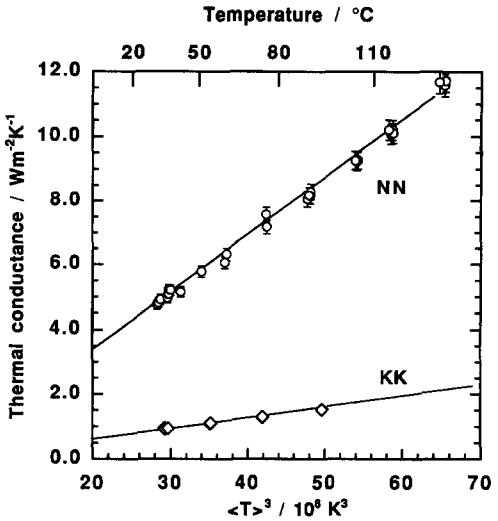

Fig. 5. Thermal conductance due to radiative heat transfer between the interior surfaces of a glazing with uncoated, NN, glass surfaces (circles), and two doped SnO_2 coatings, KK, (diamonds). The data are presented as a function of the cube of the average temperature $\langle T \rangle$, measured in K, of the two inner surfaces of the glazing. The experimental data (points) were measured using the transient technique (using the temperature dependent specific heat for soda-lime glass and the calibration against the guarded hot plate apparatus shown in Fig. 4). The theoretical results (solid lines) were calculated from the optical properties of the uncoated and coated surfaces.

$\alpha = \alpha_1\alpha_2/[\alpha_2+\alpha_1(1-\alpha_2)]$, α_1 and α_2 are the accommodation coefficients of the gas molecules with the two internal surfaces, γ is the specific heat ratio of the gas, M is the molar mass of the gas, T is a temperature intermediate between that of the two surfaces, k is Boltzmann's constant and N_A is Avogadro's number. For a gas like water vapour at room temperature, and assuming a combined accommodation coefficient of 0.5 and $\gamma = 1.3$ (typical for triatomic molecules), this gives $C_{gas} \approx 0.95p$, where p is measured in Pa and C_{gas} is in W $m^{-2} K^{-1}$. The choice of $\alpha = 0.5$ is simply representative of that for molecules which interact reasonably strongly with the surface. Using a UHV system equipped with a mass spectrometer, we have confirmed that, as expected [20, 21], water vapour is the dominant ($\sim 99\%$) component of the outgassing of soda-lime glass and solder glass, and of the gas released from degraded samples of vacuum glazing when the gap has been (destructively) exposed to the UHV system [22]. We are at present planning to measure the accommodation coefficient of this gas on the internal surfaces.

At high gas pressures, where the mean free path is much less than the glass separation, the thermal conductivity is approximately independent of pressure. For water vapour in a vacuum glazing with a gap of 0.15 mm, the high pressure thermal conductance between the internal glass surfaces is about 160 W $m^{-2} K^{-1}$ at room temperature [16]. It has been found empirically [23] that the total gaseous thermal con-

ductance C_{Tot} at any pressure can be written as a combination of the thermal conductance in the molecular regime C_{gas} and the thermal conductance in the viscous regime C_{vis}:

$$\frac{1}{C_{Tot}} = \frac{1}{C_{gas}} + \frac{1}{C_{vis}}. \quad (3)$$

This equation is quite accurate for both polyatomic and monatomic gases, and where the heat transfer occurs over one characteristic dimension (in this case, the gap between the glass sheets). For water vapour at room temperature, the mean free path is approximately equal to the separation of the internal glass surfaces (~0.15 mm) at a pressure of about 30 Pa (~230 mtorr). At this relatively high gas pressure an estimate of the gas pressure based only on free molecular transport (using equation (2) without first using equation (3)) is low by only about 18%.

4. SAMPLE RESULTS

As mentioned above, the purpose of developing the transient technique for measuring thermal conductance was to study ageing effects and, in particular, high temperature vacuum degradation and recovery in samples of vacuum glazing. To illustrate this, the chronological ageing history of a vacuum glazing sample with two internal doped SnO_2 coated surfaces (sample KK403) is shown in Fig. 6(a), where the gas pressure has been calculated from the measured thermal conductance (using equation (2), assuming the gas to be water, and $\alpha = 0.5$) as described above, including the correction for conductance at high pressures (using equation (3)). The sequence of temperature changes used to degrade this sample is shown in Fig. 6(b). Following the first heating sequence, the sample was held at progressively higher temperatures for four periods. For each period, the gas pressure was observed to increase steadily with time. Between high temperature periods, the sample was rapidly cooled to room temperature and the gas pressure was measured as the temperature was increased progressively. The gas pressure in the sample at room temperature, after each constant temperature ageing period, was found to be measurably greater than that for the pristine sample, and to increase markedly with temperature, as shown in Fig. 7.

The uncertainty for each pressure measurement, shown by the error bars in Fig. 7, was taken to be the same as the uncertainty in the corresponding measurement of gas conductance, and represents the reproducibility of the data. The uncertainty of the gas con-

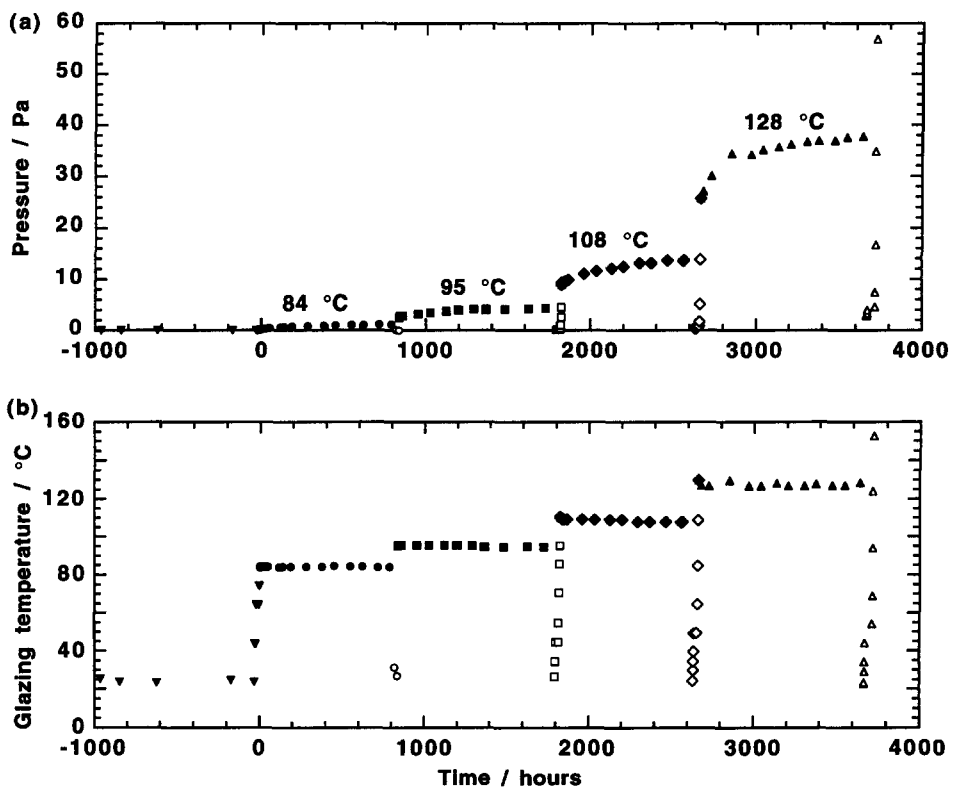

Fig. 6. (a) Chronological history of the gas pressure, estimated from the measured thermal conductance and equations (2) and (3), for a sample (KK403) of vacuum glazing with two doped SnO_2 coated surfaces; (b) the sequence of temperature changes used to degrade this sample. Time is measured from the beginning of ageing at 84°C, when the first indication of degradation was observed. For clarity, solid symbols indicate ageing measurements at a specific constant temperature and the correspondingly shaped hollow symbols indicate measurements following the completion of the ageing period at this temperature.

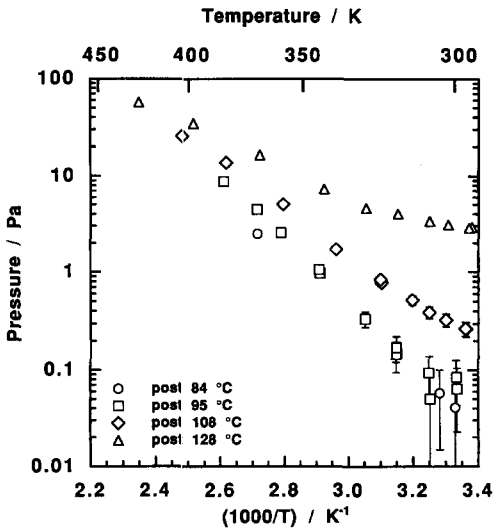

Fig. 7. Arrhenius plot of the gas pressure for the data of Fig. 6. The measurements were made after the glazing had been cooled to room temperature following extended baking at the temperatures shown. The data symbols correspond to those used in Fig. 6.

ductance was determined by addition of the absolute uncertainties in both the total measured gas conductance, and the estimated conductance due to radiation. For both of these conductances the relative uncertainty was taken to be $\pm 2\%$, corresponding to the reproducibility of each measurement. Therefore, when the dominant heat transfer is by gas conduction, the relative uncertainty in the pressure is about $\pm 2\%$; it can be much greater when there is little contribution from gas. This manner of estimating the uncertainty in the gas pressure ignores any systematic uncertainty related to the conversion of conductance to gas pressure using equations (2) and (3). Owing to the imprecise knowledge of the constants used in equation (2), the absolute values given for the pressure measurements cannot be considered accurate to better than a factor of about two. Considerably more accurate data will be obtained when the accommodation coefficient for water vapour on the internal surfaces is known. It is also possible that the accommodation coefficient may depend on the pressure within the sample, and on temperature.

Some information about the relative numbers of adsorbed molecules and molecules in the gaseous phase is revealed by the measurements made during the relatively rapid temperature changes after each period of constant temperature ageing. For these measurements, additional ageing of the sample is negligible, since the time taken to make a measurement and raise the temperature of the oven and glazing between measurements is relatively short (between 1 and 2 h). This has been confirmed on several samples by immediately repeating the measurements, with no detectable change in the results. The increase in gas pressure, immediately following that in temperature, is interpreted as being due to the desorption of molecules from the internal surfaces. The results from such measurements have been replotted in the Arrhenius plot of Fig. 7, where the gas pressure is displayed on a logarithmic scale against $1/T$ on a linear scale. The data are consistent with thermally activated desorption ($p \propto \exp(-E_s/kT)$) of the gas from the surface, and the slope of the linear portion of the graphs in Fig. 7 can be used to determine the activation energy E_s. For example, $E_s \approx 0.7$ eV for the desorption measurements shown in Fig. 7 following the 95°C ageing. A full study of the degradation of vacuum glazing, including the numerical modelling of desorption processes, and of the physical processes which result in gas evolution from the internal surfaces, is currently in progress [24].

5. CONCLUSIONS

A technique has been developed for measuring the thermal conductance of vacuum glazing due to radiation and gas conduction between room temperature and 200°C. The technique involves establishing a rapid temperature change on one side of a sample of vacuum glazing and making accurate measurements of the slow rate of temperature increase on the other side. The reproducibility of measurements by this transient technique is $\pm 2\%$. The technique has been calibrated using samples of vacuum glazing which have been measured with a guarded hot plate; the accuracy of the technique is estimated at $\pm 5\%$. Such a calibration process yields a value of specific heat capacity of soda lime glass of $[813 + 1.67T]$ J kg^{-1} K^{-1}, for temperature T in °C between 0 and 200°C.

The gaseous conductance in as-produced samples of vacuum glazing is negligible. Measurements on such samples at room and slightly elevated temperatures therefore yield an accurate estimate of radiative conductances over the entire temperature range of interest. Using such estimates, the gaseous conductance can be determined as the internal vacuum degrades during high temperature ageing. Such measurements are being used to identify and quantify the physical processes which are responsible for degradation of vacuum in these devices.

Although the transient technique gives very reproducible data for gas conductance in a degraded sample of vacuum glazing, the internal pressure is at present only know to within a factor of approximately two. Significant improvements in the absolute accuracy of estimates of internal pressure require a knowledge of the accommodation coefficients for the interaction between the gas species, water vapour, and the internal surfaces.

The transient technique could also be used to study vacuum glazing at even higher temperatures, or to obtain information about the heat transport processes in conventional double glazing. In both cases this would require further finite element modelling, or calibration measurements against samples having known thermal conductance.

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