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Development of the Measurement Apparatus for the Effective Thermal Conductivity of Core Material

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Abstract—A measurement apparatus is designed and fabricated to measure the effective thermal conductivity (k_{eff}) of a VIP (vacuum insulation panel) core specimen under various vacuum states and external loads. The apparatus consists of part for measuring k_{eff} , and parts for controlling external load and vacuum condition. Uncertainty of the apparatus is validated by measuring the standard reference material and comparing with commercial devices with VIP samples. Assessed uncertainty is maximum 2.5 % in case of the standard reference material, 10 % in case of VIP samples. Using the apparatus, k_{eff} of glass paper under various vacuum levels is examined.

Keywords—Effective thermal conductivity, guarded hot plate method, vacuum insulation panel

I. INTRODUCTION

THE vacuum insulation panel (VIP) has approximately 1/10 of effective thermal conductivity k_{eff} compared with conventional insulation materials. Usually, a VIP comprises an envelope, a core and a gas absorbent [Fig. 1].



Heat transfer in the VIP mainly occurs across the core by solid conduction, residual gas conduction and radiation. These are denoted by the conductivities k_s , k_g and k_r , respectively. Consequently, k_{eff} of the core is the sum of k_s , k_g and k_r but they are usually coupled with each other. The contribution of k_g to k_{eff} can be eliminated by making a perfect vacuum. However, k_s and k_r persist even in vacuum so that the minimum k_{eff} is k_s+k_r . There are theoretical models to investigate k_s [1-3], k_r [4], and k_g [5]. However, to apply those theoretical models for VIP core, they have to be validated experimentally under 'special conditions' to which core of the VIP is exposed, i.e., the inner

vacuum condition, and latm of the external load. Some experimental studies are reported [6-8] but they do not meet both of conditions. The objective of this study is to design and fabricate a measurement apparatus to measure k_{eff} of material under vacuum condition and external load. This apparatus also enables one to measure k_{eff} of various cores without having to finish all the process of VIP to vacuum sealing for the measurement sample.

II. MEASUREMENT APPARATUS

A. Requirements and Parts of the Measurement Apparatus

Principal requirement of the apparatus is to measure the thermal conductivity of a specimen. There are several measurement methods according to the range of k_{eff} . In case of vacuum insulation which has very low k_{eff} , heat flow meter (HFM) and guarded hot plate (GHP) methods can be applied and GHP is known to be more precise than HFM [9]. Another requirement is to maintain vacuum during the measurement. For this purpose, vacuum components such as vacuum chamber, pumps and gauges are needed. The last requirement is to apply pressure to the specimen during the measurement. When the VIP is in atmospheric condition, it is always pressed at 1atm. It is well-known that the external load can heavily influence to k_s of a specimen. However, if the VIP is placed in a vacuum chamber and the chamber is evacuated to vacuum condition, there is no more external load on the VIP. A pressing tool is thus necessary to exert external load on the specimen. [Fig. 2] shows the whole composition of the apparatus.



Fig. 2 Schematic diagram of measurement apparatus

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B. Parts for Measuring Thermal Conductivity

This part (GHP) consists of a cold plate, a hot plate, a heater block, a guard and an auxiliary insulator [Fig. 3]. The heater block is surrounded by the guard across a narrow gap. The auxiliary insulator is placed under the heater block and the guard. A Pyrogel® core VIP is used as the auxiliary insulator. Its thickness is 17.5 mm and thermal conductivity is 5 mW/m·K. The hot plate is placed under the auxiliary insulator and the cold plate is placed over the heater block and the guard. A specimen is sandwiched between the cold plate and the heater block/guard.



If temperature of the cold plate is lower than that of the heater block and temperatures of the guard and the hot plate are same as the heater block, all of the heat which is electrically generated in the heater block is transferred to the cold plate via the specimen. Then, effective thermal conductivity k_{eff} of the specimen can be derived as

$$k_{eff} = \frac{Q \cdot t}{A_{eff} \cdot (\Delta T)} \tag{1}$$

where Q is the generated heat at the heater block, t is thickness of the specimen, and ΔT is temperature difference of top and bottom surface of the specimen. Effective area $A_{e\!f\!f}$ covers the surface area of heater block and half of the gap area. The heater block has hollow space to insert an electric heater. At a steady state, it usually generates below 1 W. For uniform temperature distribution, residual space between the electric heater and the heater block is filled with thermal grease. Also heater block is made of 99.99 % pure copper for the same purpose. Heat from the heater block should be ideally transferred to cold plate in a one-dimensional manner. Therefore, heat loss through sides and bottom should be minimized. The guard and the hot plate prevent heat loss through sides and bottom surface respectively by maintaining the temperature same as the heater block. Temperature of the guard and the hot plate is controlled by hot circulating water. Material of the guard is 99.99 % pure copper and that of the hot plate is 99.99 % pure aluminum. The cold plate has exactly the same shape as the hot plate and its temperature is controlled by circulating water, too. A linear variable differential transformer (LVDT) is attached on the cold plate and the guard and it measures the distance change between the cold plate and the

Vol:5, **Noard 20h4** T-type thermocouple (TC) which is known to be most precise in the given temperature range [10] is employed to measure temperatures of each parts. For a better measurement result, temperature difference between (1) the heater block and the guard and (2) the heater block and the hot plate have to be measured very precisely. Those temperature differences are so small that voltage signal from TC wire is very small. To amplify the voltage signal, 5-pairs of thermopiles are attached.

C. Parts for External Load

The pressure pad is designed to give an external load to the specimen. The external load is equivalent to the external pressure acting on the VIP. The pressure pad is in cylindrical shape and comprised of a moving part, spacers and walls [Fig. 4]. When compressed air flow into the pressure pad, the moving part moves upward. Since a specimen is sandwiched between the pressure pad and the vacuum chamber lid, the external load presses the specimen as the moving part goes up. To accommodate various specimen thicknesses, several dummy plates may be inserted between the cold plate and the vacuum chamber. If there is no external load from the pressure pad, only pressure from weight of the cold plate (1 kPa) exerts on the specimen.



D.Parts for Vacuum

The vacuum chamber has a cylindrical shape with 510 mm of diameter, 375 mm of height and it is made of stainless steel. It is evacuated using a cascade of a diffusion pump and a rotary pump. The diffusion pump has 0.57 m^3 /s of pumping rate, 10^{-5} Pa of the ultimate pressure. Back pressure of the diffusion pump is made by the rotary pump which has 0.03 m^3 /s of pumping rate and 10^{-2} Pa of the ultimate pressure. Pressure in chamber is measured by ion vacuum gauge.

E. Validation and Uncertainty Analysis

A standard reference material (SRM) is used to calibrate the measurement apparatus. It is made of glass fiber and has dimension of 30 cm x 30 cm x 2.53 cm. Measurement of the SRM is conducted in atmospheric pressure with 20 % of relative humidity and the result is shown at Fig. 5. In Fig. 5, solid line is the reference value, dots are the measured values and error bar means ± 1.5 % relative error. Maximum relative error is 2.6 % and average relative error is 1.5 %. This result shows the validity of current device for conventional insulation

materials



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Vol:5, No:	1, 2011 TABLE UNCERTAINTY OF MEASURE	K_{EFF} of a VIP Sample	
red	Parameter	Uncertainty	
	Q	2.14 %	
	$A_{e\!f\!f}$	0.03 %	
	t	0.22 %	
	ΔT	3.12 %	
	Total uncertainty	5.51 %	

TABLEI

F. Validation of Theoretical Heat Transfer Model

The measurement apparatus enables us to validate a theoretical heat transfer model of the core material. As an example, glass papers are measured under 23 °C ambient temperature, 25-30 % relative humidity, 25 °C mean temperature of a specimen and different vacuum levels. The specimen is manufactured by Hankuk Carbon and has dimension of 270 mm x 270 mm x 0.8 mm and density of 157 kg/m^3 . Diameter and span of the fiber string are estimated to 6 μm, 150 μm from SEM micrograph. Young's modulus, Poisson's ratio and thermal conductivity of the fiber string are 72.4 GPa, 0.2 and 1.3 W/m·K, respectively. The mass specific extinction coefficient er is assumed to be 53 m²/kg [12]. Fig. 6 is showing the dependency f k_{eff} on the residual gas pressure. The curve shows typical tendency of k_{eff} [13]. Plotted together is a theoretical prediction [3]. Due to page limitation, the detailed theory is referred to [3]. Suffice it to mention that it is based on rarefied gas conduction, diffusion radiation heat transfer and Hertz contact theory [14] assuming uniform contact between crossing fiber. Dependency on the residual gas pressure is in good agreement between the measurement and the theory, which shows the validity of theory of k_g .



However, general VIP has much smaller k_{eff} than the SRM. Since there is no SRM which has such small k_{eff} , additional validation is carried out by comparing to other commercial instruments. Two VIP samples made in a laboratory are used as the specimens. One sample is compared with NETZSCH GHP Titan 456, and the other one is compared with NETZSCH HFM 436. Comparison of the first VIP shows 9.6 % relative error and the other comparison shows 0.5 % relative error. This roughly shows that the current device has a deviation of about 10 %, although it is not certain which device is responsible for the error. For a more quantitatively precise evaluation of the uncertainty, an uncertainty analysis is performed as follows. The uncertainty of k_{eff} is expressed as [11]

$$dk_{eff} = \sqrt{\left(\frac{\partial k_{eff}}{\partial Q} dQ\right)^2 + \left(\frac{\partial k_{eff}}{\partial A_{eff}} dA_{eff}\right)^2 + \left(\frac{\partial k_{eff}}{\partial t} dt\right)^2 + \left(\frac{\partial k_{eff}}{\partial (\Delta T)} d(\Delta T)\right)^2}$$
(2)

As the reference case, uncertainty is calculated for the first VIP sample. The magnitude of each term in (2) is summarized in Table I. The uncertainty of ΔT has the largest portion due to precision of T-type TC and it can be reduced by increasing ΔT . Uncertainties of A_{eff} and t come from error of the measured area and thickness of the specimen, respectively. The uncertainty of Q is due to the error of the power supply. Some portion of the Qis lost to the guard mainly by gap convection and to the auxiliary insulator by conduction. Therefore, those heat losses have to be compensated from the total heat transfer rate.



However, error between them is clearly seen when the external load is changed while maintaining the residual gas pressure at 1 atm. See Table II for k_{eff} . When external load increases from 0.001 MPa to 0.1 MPa, k_{eff} increases by 5.54

mW/m·K, and when it increases from 0.1 MPa to 0.2 MPa, k_{eff}

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increases by 4.26 mW/m·K. Since k_r and k_g remain constants, No:1, 2011

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increment of k_{eff} means increment of k_s . Also, increment of k_s can be theoretically calculated [Table III]. External load increase from 0.001 MPa to 0.1 MPa, and then to 0.2 MPa shows 0.24 mW/m·K and 0.06 mW/m·K increments of k_s , respectively. The theoretical heat transfer model predicts a linear relationship between increment of external load and k_s in the calculation; when external load increases x times, k_s increases x/300 times. However, measured increment of k_s is not proportional to the external load increase. This tells that theoretical model may have a fault. It does not consider the changing number of contact points when changing the external load and it may have brought the error between measured and calculated results. This example demonstrates the usefulness of this measurement device in improving the VIP and the related theory.

TABLE II MEASURED κ_{eff} of GLASS PAPER UNDER VARIOUS EXTERNAL LOADS (RESIDIAL GAS PRESSURE=1 ATM)

External load (MPa)	$k_{e\!f\!f} \ (W/m\cdot K)$	Increment of k_{eff} (mW/m·K)
0.001	0.03842	5.54
0.1	0.04396	
0.2	0.04822	4.26

TABLE III

CALCULATED K_S OF GLASS PAPER WITH VARIOUS EXTERNAL LOADS (RESIDUAL GAS PRESSURE=1 ATM)

		/
External load (MPa)	k_s (W/m·K)	Increment of k_s (mW/m·K)
0.001	0.00075	0.24
0.1	0.00099	
0.2	0.00105	0.06

III. CONCLUSION

In this paper, a measurement apparatus for k_{eff} in vacuum is designed and manufactured. Thermal conductivity of specimen is measured by the guarded hot plate method. External load on specimen is exerted by the pressure pad. They are placed in a vacuum chamber in order to control vacuum level during measurement. The uncertainty of measurement apparatus is validated to be 2.5 % in case of normal insulation material, 10 % in case of vacuum insulation panel. The measurement apparatus enables to validate theoretical heat transfer model of a specimen. As an example, effective thermal conductivity k_{eff} of the glass paper according residual gas pressure, external loads are measured. It gives a clue for improving the theory.