

Monitoring Gas Pressure in Vacuum Insulation Panels

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1 Introduction

Besides the thermal conductivity of the fully evacuated core material, gas pressure is the most important indicator of quality of a vacuum insulation panel (VIP). For any core material there is a relation between the thermal conductivity λ and gas pressure p_{gas} , with the typical pressure $p_{1/2}$ as parameter, which depends on the pore size of the core material. In most cases it can be described by the formula:

$$\lambda = \lambda_0 + \lambda_{\text{gas}} / (1 + p_{1/2} / p_{\text{gas}}), \quad (1),$$

with λ_0 being the thermal conductivity at zero gas pressure and λ_{gas} the thermal conductivity of the gas at atmospheric pressure.

Monitoring the change of gas pressure in a VIP with time gives information on the degradation of thermal conductivity and its probable service life time. Both air and water vapour may diffuse through the envelope of a VIP and hence increase the internal gas pressure. The increase of water vapour pressure within the panel also depends on the adsorption properties of the filling material for water vapour. To discriminate between air gas transmission and water vapour, samples may be prepared with getters for water vapour. If outgassing of other gases can be neglected, then essentially only the nitrogen and oxygen transmission originating from the outside atmosphere are responsible for the increase of internal gas pressure.

The gas transmission of section of films can be measured with devices, where the film is in between a volume with high concentration and a volume with low concentration of gas, including a detector for the specific gas (e.g. O_2 , H_2O , He). Such measurements of gas transmission of high barrier films used for vacuum panels often are at the limit of the resolution of the available apparatus. Furthermore, the increase of gas pressure in VIP doesn't only depend on the gas transmission of the plane barrier film, but may additionally be influenced by the gas transmission of the welded seams and deficiencies in the barrier film e.g. at the edges and corners. A direct measurement of gas pressure increase within a VIP thus can be more valuable than the measurement of the plane film properties alone.

With available high barrier films the gas pressure increase may be very low, e.g. 1 hPa per year for a 20 mm panel. Therefore accurate methods are needed to measure such low increases, or methods, which accelerate the gas pressure increase. Means for the acceleration of the gas pressure increase within vacuum panels may be: high storage temperatures, gases with high permeability and thin panels.

For measurement of gas pressure it is possible to use internal sensors, which, however, usually need a connection between the inside and outside of a panel, e.g. cables for electricity input and sensor signal output. A very accurate sensor system is the spinning rotor gauge, which measures the slowing of a rotating steel ball by friction of the residual gas. Here a steel tube, which is closed on one side and contains the steel ball, is connected with its open side to the vacuum device to be tested.

Especially valuable are methods, which may be applied to any vacuum panel without or with only small modification of the panel itself like: 1) testing the lift-off of the panel envelope when the whole panel or part of it is exposed to a gas pressure smaller than the pressure inside the panel, e.g. within a vacuum chamber and 2) measuring the thermal conductivity of the vacuum panel.

The first method is quite common and used for the testing of gas pressure within microporous silica panels. The second method is not applicable in the case of silica panels as there is only a small influence of gas pressure on the thermal conductivity. For core materials with larger pores, however, this method is very useful, as even a gas pressure change of 0,01 hPa may result in a significant change of thermal conductivity of the core material. Envelopes to be tested are filled with such a core material and evacuated to a gas pressure below 0,2 hPa. Then the change of thermal conductivity with time is monitored by e.g. a hot plate apparatus.

A modification of method 2), developed by *va-Q-tec*, uses a thin, open porous fleece, which is put below the surface film of the vacuum panel. In addition a heat sink, e.g. a metal plate, is provided under the fleece on the panel side. With a small measurement head applied from the outside, then the thermal conductivity of the thin inside sample fleece is measured. From the known relation between thermal conductivity of the sample fleece and the gas pressure the gas pressure within the panel is deduced. This method ("*va-Q-check*") does not depend on the kind of core material. It now is routinely used at *va-Q-tec* for quality control, research and development. Samples can be stored under different conditions (temperature, humidity, different gas atmosphere) and the change of the gas pressure be monitored with time. In the following we report some results of these different methods for measuring gas pressure and gas pressure increase.

2 Measurement of gas pressure with spinning rotor gauge

The accuracy of gas pressure measurement with a spinning rotor gauge (SRG) is relative high, but the pressure range usually has to be below 10 Pa. A VIP sample was prepared containing a special open porous material and a metallized barrier film as envelope. It contained a dryer material. The tube with the spinning ball was attached through the seam to the inner side of the sample. First the sample was stored at room temperature (ca. 25 °C) and the gas pressure measured online by the measurement unit for five days. After two more weeks the sample was placed into a freezer at a temperature of - 30 °C and the gas pressure measured regularly. As can be seen in Fig. 1 the measurement started with a gas pressure as low as 0,4 Pa. After 2 days a steady increase of gas pressure of 0,2 Pa/day or 72 Pa/year is reached. At - 30 °C storage according to Fig. 2 the gas pressure increase is reduced to only 0,6 Pa/year. In both cases this is an upper limit for the gas transmission as the possible additional gas leak due to the tube connection is unknown. From the measurements at the two different temperatures an Arrhenius factor for the gas diffusion through the film of 50 kJ/mol can be estimated.

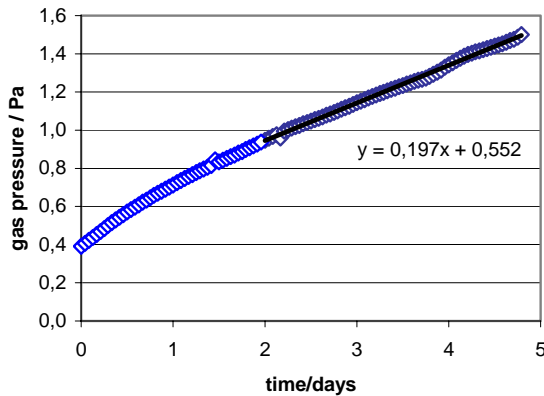


Fig. 1: Pressure increase with SRG at 25 °C

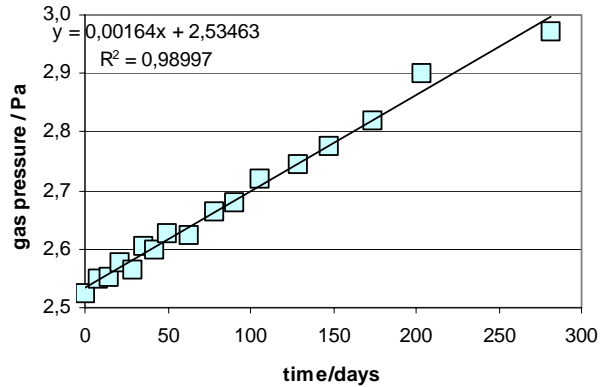


Fig. 2: Pressure increase with SRG at - 30 °C

3 Gas pressure measurements with a vacuum chamber

In order to evaluate the gas pressure increase of silica VIPs stored at room temperature a batch of 15 VIPs with the size 500 x 450 x 20 mm³ was measured regularly by the vacuum chamber method. The mean values are shown in Fig. 3. The increase of gas pressure here is about 1 hPa/year. Silica VIPs permanently stored within a climate chamber of 80 °C temperature have a larger increase of about 5 hPa/year as can be seen in Fig. 4 (mean values of 12 samples with size 200 mm x 200 mm x 20 mm). The increase of gas transmission with temperature is smaller than expected if the Arrhenius factor derived in section 2 is taken into account. This is due to the relative large amount of water vapour transmission at room temperature. It may contribute up to two thirds to the pressure increase of 1 hPa/year.

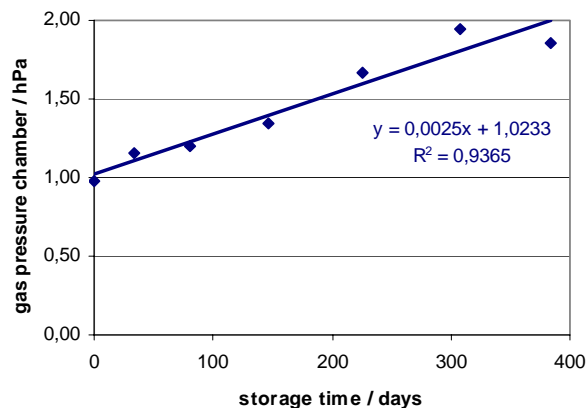


Fig. 3: Gas pressure increase at room temperature

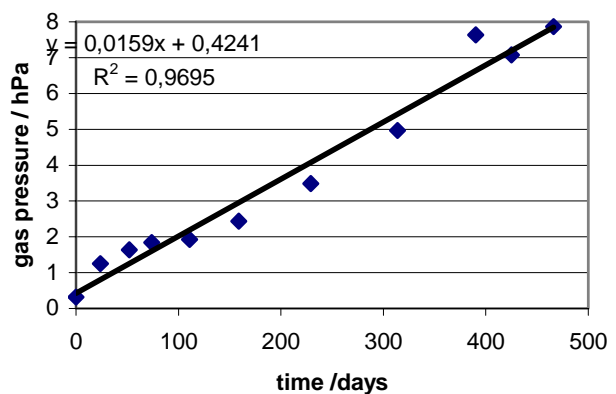


Fig. 4: Gas pressure increase - storage at 80 °C

4 Gas pressure measurements with "va-Q-check"

Measurement time of the *va-Q-check* device usually is only 10 seconds. Gas pressures in the range between 0,02 hPa and 10 hPa can be measured. Devices have been built for production control with an attached computer, which stores the VIP product number and the measured gas pressure. Mobile devices with somewhat less accuracy have also been constructed (Fig. 5). They may be used for control of gas pressure during installation of VIPs e.g. in a building. A typical calibration curve for a mobile *va-Q-check* according to eq. (1) is shown in Fig. 6.



Fig. 5: Mobile *va-Q-check* apparatus

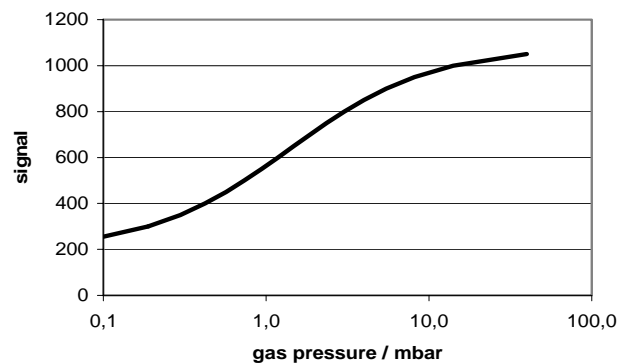


Fig. 6: Calibration curve for a mobile *va-Q-check* with a coarse fleece

4.1 Measurement of gas pressure increase

A VIP sample with 5 mm thickness was prepared using a metallized film. The sample was equipped with the sensor disk and stored at room temperature (during summer time) without temperature and humidity control.

In Fig. 7 the variation of gas pressure as measured with the *va-Q-check* system is depicted. Already after two weeks an increase of gas pressure of about 2 hPa per year can be deduced from the data. This corresponds to a dry gas transmission rate of 10 hPa liter per m² of panel and year.

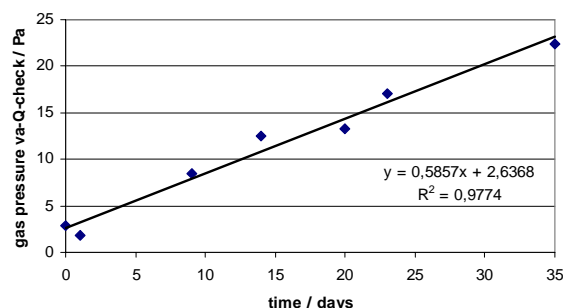


Fig. 7: Increase of gas pressure as measured by *va-Q-check*

4.2 Production control

Main purpose of the development of the *va-Q-check* device was a fast and reliable, 100% production control of VIPs. An example of the variation of gas pressure within a production lot of VIPs containing a fumed silica core is shown in Fig. 8. The panels had a size of 1000 mm x 500 mm x 20 mm. Panels with gas pressures above 3 hPa are rejected. As can be seen continuous improvement of the production process has decreased the starting pressure to about 0,4 - 1 hPa. Thus increases of 1 hPa within two weeks storage times could be detected easily. This corresponds to a gas pressure increase of 25 hPa/year or a gas transmission of 500 hPa liter/m² year.

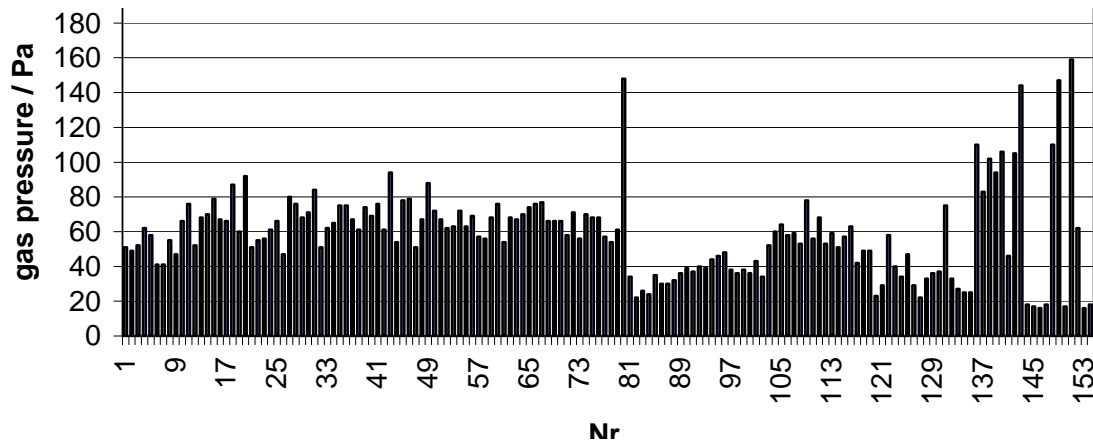


Fig. 8: Variation of gas pressure of VIPs with microporous silica as measured by va-Q-check

The *va-Q-check* sensor can also be used to monitor the gas pressure of VIPs made from fiber material. Here a gas pressure below 0,1 hPa should be achieved after production. In a measurement campaign the reliability of the *va-Q-check* system for these low gas pressures was tested. 58 vacuum panels with fiber filling were produced and sent to the customer. At the customer site three persons measured the panels twice within two days. From the six data sets the mean value of the gas pressure and the standard deviations were calculated.

In Fig. 9 the mean values of all the samples plus/minus the standard variations are depicted. All mean values of the gas pressure are well below 10 Pa. The standard variation for the individual panel measurement was 1 Pa in the average.

Three samples of the same batch had a gas pressure above 30 Pa and thus are far beyond the limit of a "good" panel. They also had been measured with the same procedure as above. Even at this higher gas pressure range the variation of the six measurements is in the order of only 10 Pa as can be seen from Fig. 10.

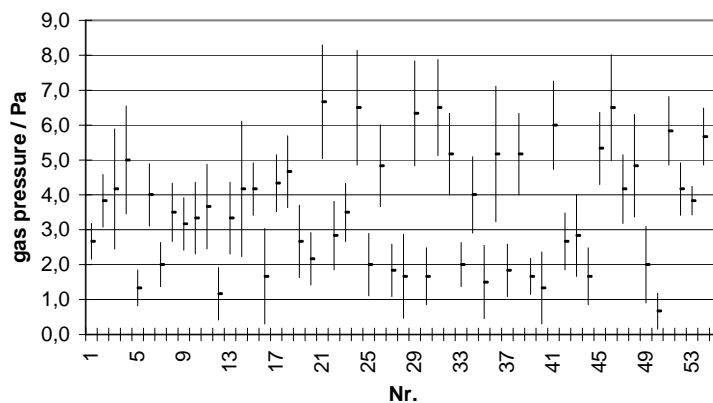


Fig. 9: Mean values and standard variation of panel batch

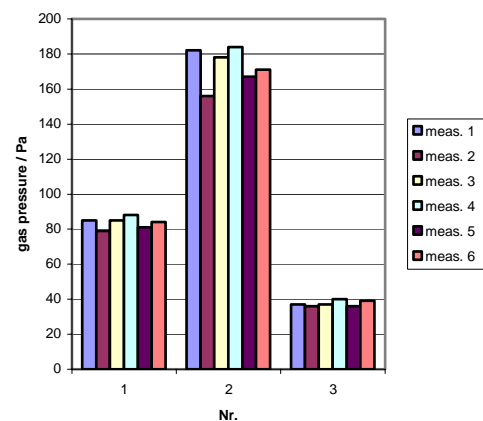


Fig. 10: Individual measurement results

4.3 Storage in helium



Fig. 11: VIPs with flaps (left) and accurately folded corners (right)

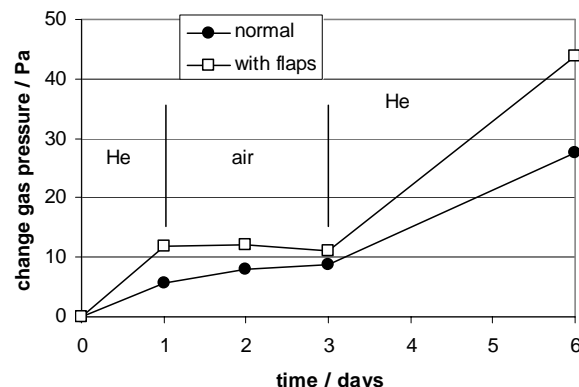


Fig. 12: Effective gas pressure change of samples in He and air (starting pressure set to zero)

Additionally tests have been performed with silica VIPs stored in an atmosphere of helium. Helium has a high permeability in comparison to nitrogen and oxygen. Due to the high gas conductivity the *va-Q-check* signal is also enhanced. Helium gas transmission measurements of plane film indicate that there is a good correlation to the transmission of water vapour [EMPA 2005]. Defects of the envelope should be detected with helium as well. Thus if an envelope is stressed by cracks and folds it should be possible to detect a change of the *va-Q-check* signal within a short time.

50 VIPs with two different folding techniques (Fig. 11) were stored one day in helium, two days in air and again three days in helium at room temperature. Starting pressure was around 0,3 - 0,5 hPa. The size of the vacuum panels was 1000 mm x 500 mm x 20 mm. Fig. 12 shows the mean value of the effective gas pressure change as measured by *va-Q-check* during the storage time of six days (the gas pressure was not calibrated to a mixture of air and helium gas). As can be clearly seen the increase of the effective gas pressure during He exposure is far higher compared to the exposure to ambient air. It is remarkable, that the effective increase of gas pressure due to He (or the change of thermal conductivity of the test fleece) is almost a factor of two higher for the vacuum panels with flaps in comparison to vacuum panels with the special folding technique as it is used by *va-Q-tec*. The higher helium transmission of the panels with tucked flaps may be attributed to the increased flaws in the foil due to the folding stress.

4.4 Storage in high humidity atmosphere

In order to get information on the amount of water vapour which penetrates the envelope, VIPs with different films have been stored at 30 °C in a high humidity atmosphere (90 - 100 %). The test panels contained a filler material with low water adsorption. In contrast to the gas transmission measurements they were not equipped with a dryer. The gas pressure was measured regularly with the *va-Q-check* device for 30 days. In Fig. 13 the increase of the gas pressure with time is shown for different kind of films. The calibration of the gas pressure does not include the different thermal properties of water vapour and air. Thus the derived gas pressures are "effective" values as in Ch. 4.3. It can clearly be seen that different films yield quite different changes of gas pressure with time. To reach a certain level of gas pressure, e.g. 1 hPa, it takes 18 days with film A and only 5 days with film D and E. Using this method the transmission of water vapour can be assessed relatively to a standard film (e.g. film A). Usually at least two weeks are needed until the transmission of water vapour through the film envelope is about stationary.

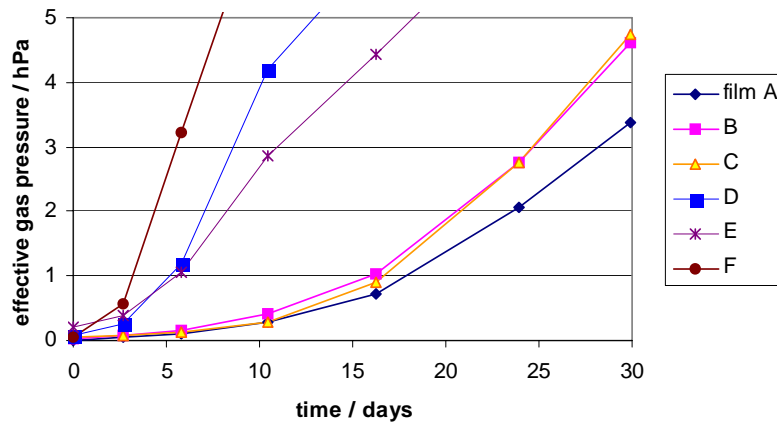


Fig. 13: Increase of effective gas pressure within VIPs stored in a high humidity atmosphere

4.5 Permanent measurement head

The surface of VIPs which are installed e.g. in a house wall or technical device often are not accessible. The *va-Q-check* measurement head then can not be applied any more. To solve this problem, a permanent measurement head was constructed. This measurement head is permanently fixed at the panel surface above the internal sensor chip. It has a thickness of only 1 mm (Fig. 14). Eight wires are needed to supply the measurement head with electrical power, control the temperatures and monitor the signal. The measurement head is heated to operating temperature within some seconds, then the usual *va-Q-check* measurement routine is started. The signal at the end of the measurement is converted to the gas pressure by a calibration curve. The calibration curve in Fig. 15 is related to a fine fleece, which in comparison to the fleece used in Fig. 6 has a higher measurement range up to 100 hPa. Currently some permanent measurement heads have been installed for long term observation of selected VIPs in different building objects.



Fig. 14: Permanent measurement head on VIP

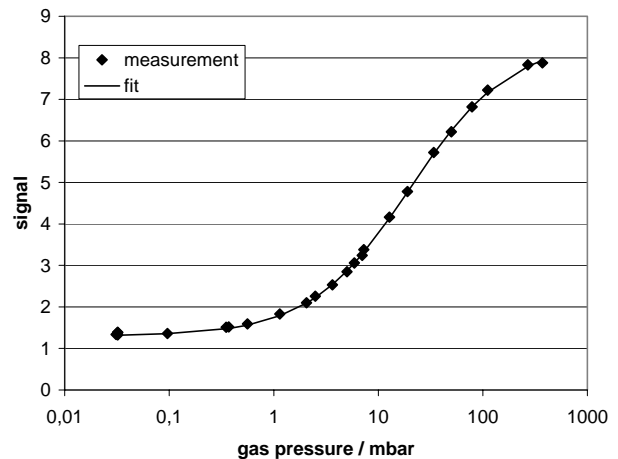


Fig. 15: Calibration curve with fine sensor fleece

5 Internal thermal sensor for gas pressure measurement

For higher sensitivity at low gas pressures also an internal sensor based on a thermal sensor principle was constructed and tested. It consists of a heating foil suspended within a small metal casing. The foil quickly is heated to a temperature of about 80 °C, whereas the metal casing stays at room temperature. As there is a small gap between the heating foil and the metal casing, after some seconds

the power input into the foil corresponds to the heat flux through the gap. This heat flux again depends on the gas pressure. The relation between gas pressure and sensor signal can be deduced by measurements with known gas pressure similar to the routine used for the *va-Q-check* system.

The sensor casing with a typical diameter of 44 mm and 5 mm height is mounted into a vacuum panel. The wires for heat input and temperature sensor are led through the seam of the panel. In order to test whether this wire connection causes gas leaks, a sample vacuum panel was equipped with an aluminium foil and the sensor. It then was evacuated and sealed. The gas pressure obtained by the internal sensor and the gas pressure measured by the *va-Q-check* system were measured as function of time. The results are compared in Fig. 16. Whereas both methods give about the same increase of gas pressure with time, the gas pressure deduced by the internal sensor has a far lower variation of the signal. From the thickness of the panel and the gas pressure increase a gas transmission of 1,5 hPa liter/m² year can be deduced. The leakage of the wire connection from the internal sensor to the outside thus must be lower than this value.

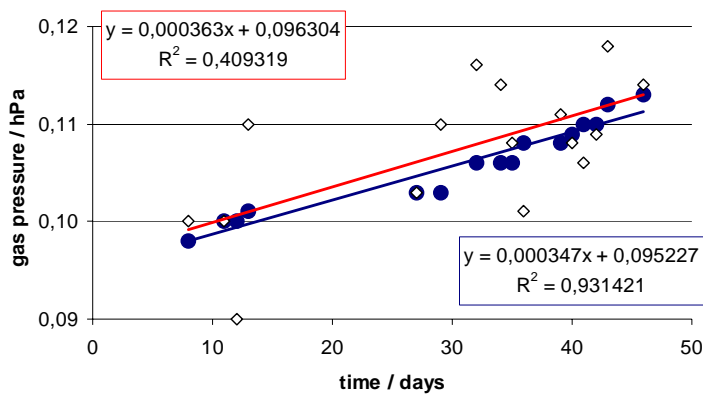


Fig. 16: Comparison of gas pressure increase measured by internal sensor (dots) and by *va-Q-check* (rhombs)

6 Gas pressure increase by thermal conductivity measurements

6.1 Air transmission

For monitoring the increase of gas pressure thermal conductivity measurements of VIPs are also possible, if materials with large pore diameters are used. Within a short time from such measurements the increase of gas pressure and the corresponding gas transmission of the barrier film envelope can be deduced. A sample measured in a heat flow meter apparatus is shown in Fig. 17.

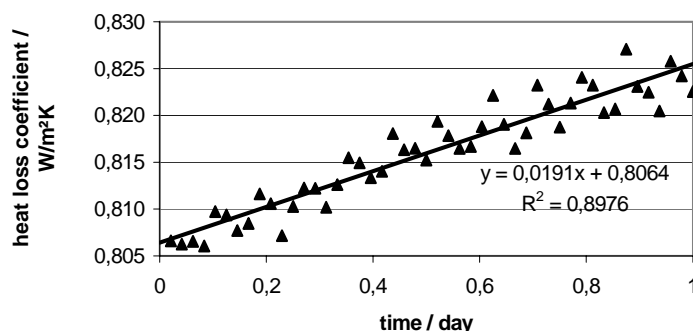


Fig. 17: Heat flux through VIP with metallized film in air

The sample was the same as used for the gas pressure measurements with *va-Q-check* in Fig. 7. Within one day the heat loss coefficient increased from 0,805 W/m²K to 0,825 W/m²K. The variation of the measurement signal is well below 1%, thus the change of the heat loss coefficient can be determined with good accuracy. The applied temperatures of the sample were 37 °C at the hot side and 20 °C at the cold side. From the properties of the core material and sample thickness a gas transmission of 14 hPa liter/m² year is derived.

6.2 Helium transmission

A similar VIP as above but with an aluminium foil as envelope was exposed to a helium atmosphere during the measurement. The increase of heat loss coefficient is 0,03 W/m²K per day as shown in Fig. 19. Thus even panels with lowest transmission values can be evaluated in short time if exposed to He.

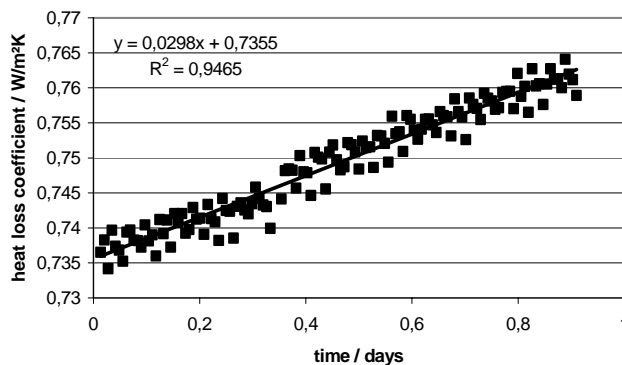


Fig. 18: Heat flux through VIP with aluminium foil in helium

7 Conclusions

va-Q-tec has developed and tested several new and fast methods for measurement of gas pressure and gas pressure increase. These methods are the key for high quality VIPs. They allow a simple and quick, 100% production control of gas pressure and a continuous improvement of panel quality. It is even possible to evaluate the gas tightness of silica VIPs after production within one or two days, if they are stored in a helium atmosphere and measured with the *va-Q-check* device.

Of similar importance as the test of produced panels is the test of the delivered film material before it is used for production. Here several tests have been developed, which allow to evaluate high barrier films within short time. In Fig. 19 the measured relative gas transmission of three different type of films A, B and C is depicted. As can be seen clearly there are not only different properties of the three films, but there may also be a variation of film quality, depending on different delivered charges (e.g. A1 and A2). This emphasizes the importance of quality control of the high barrier films.

va-Q-tec does not restrict the reported methods to the production of microporous silica VIPs but applies them also to products with alternative core materials like open porous foam and glass fibre. Better and controlled VIP products for any application - building, cold service equipment, shipping container and refrigeration - are the consequence.

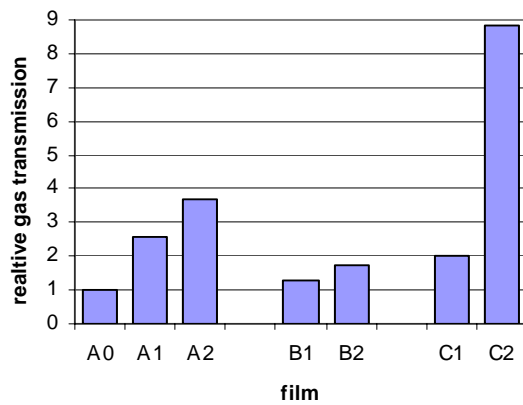


Fig. 19: Measured relative gas transmission of different metallized high barrier films (A, B, C) from different deliveries (0, 1, 2).

8 Reference:

[EMPA 2005]: *Vacuum Insulation Panels* Subtask A, Report HiPTI - IEA/ECBCS Annex 39

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