Short report
Research initiative: Zukunft Bau, a research program of the Federal Ministry for Transport, Building and Urban Development (BMVBS),
File number: SF – 10.08.18.7-11.2 / II 3
Project period: July 1, 2011 to July 31, 2013

The research project was funded by means of the research initiative Zukunft Bau, of the Federal Institute for Research on Building, Urban Affairs and Spatial Research.
The author is responsible for the content of this report.

In cooperation with FIW (München) and VARIOTEC GmbH & Co. KG (Neumarkt)

Development of a permeation measuring technology for the determination of the critical gas permeability of vacuum insulation panels (VIPs)

Director
Prof. Dr.-Ing. habil. Eckhard Beyer

Project manager:
Dipl. Ing. Harald Beese
Phone: 0351/ 83391 3356
Fax: 0351/ 83391 3300
E-mail: harald.beese@iws.fraunhofer.de

Collaborators:
Dr. Wulf Grählert
Susann Schulz

Fraunhofer-Institut
Werkstoff- und Strahltechnik
Winterbergstraße 28
D-01277 Dresden

June 2013

1 Target of the research task

Vacuum insulation panels represent a novel material class for the thermal insulation of components and are based on the principle of a dewar vessel. An evacuated volume is maintained by a highly porous supporting structure. The separation from the atmosphere is guaranteed by barrier films with extremely high gas barrier properties. To preserve the low inner pressure, the barrier properties of the film and their seals must not deteriorate over the complete lifespan of the component (~ 20 – 50 years).

For the selection and quality control of barrier films, suitable for VIP applications, a reliable test is necessary. This test, however, must be performed without extra costs creating a new VIP. The duration of such a test must be shorter than that for the determination of the heat conductivity increase at a VIP and must consider significant transport processes and gases.
Water vapor is the most critical gas, which may significantly influence the efficiency of VIPs. The penetrating water vapor increases the inner pressure but, additionally, it increases solid state heat conductivity, due to the adsorption at the porous supporting structure.
Water vapor, apart from nitrogen and oxygen, is the most common existing gas in the atmosphere, a fact which guarantees a high partial pressure difference between the inner and the outer side of the barrier film. Additionally, the available barrier films are more permeable for water vapor than for inert gases.

Within this research task the water vapor transmission rates of different commercially available and suitable (for VIP applications) barrier films were determined. The measuring technology for the determination of the water vapor transmission rate of barrier films is based on laser diode spectroscopy and was developed by the IWS scientists. The IWS technology offers a significantly lower detection limit (water vapor transmission rates WVTR < 10^{-4} g m^{-2} d^{-1}), when compared to commercially available measuring technologies.
A further task was to determine the aging behavior of barrier films. For this purpose the selected barrier films aged at an accelerated rate and periodically measured.
During the treatment of barrier films and during the application of the VIPs, the films are mechanically stressed. Due to damages, the water vapor transmission rates can differ from those of the basic films. This is the reason why the water vapor transmission rate of row films as well as stressed films was examined.

The project task focusses on the characteristics of the sealed seam with respect to the water vapor transmission rate. The standard technology is just suitable for flat substrates and thus an appropriate measuring adapter had to be designed and tested.

2 Implementation of the research task

There are several measuring methods available to determine water vapor transmission rates. Derived from ambient conditions (temperature and pressure) the Absolute Pressure Method seems to be the most suitable, since it best mirrors the conditions at the VIP. It has to be taken into account that the measured increase of water vapor concentration in the gas phase causes a 2-35 folds underestimated water vapor transmission rate [1], [2]. This is caused

by the water vapor adsorption at the measuring system as well as at the sample. This is true for the Absolute Pressure Method as well as for the quasi-isostactic methods (nearly same absolute pressure at both sides, different water vapor partial pressure).
The isostatic carrier gas method determines the amount of water vapor truly, which permeates through the barrier film. Due to the use of a laser spectroscopic sensor instead of a coulometric sensor (DIN EN ISO 15106-3) the lower detection limit can be decreased to 9 x 10^{-5} g m^{-2} d^{-1}. Even lower water vapor transmission rates can reliably be verified by a diffusion method [4] and a combined process of gas phase accumulation and mass determination during the subsequent purge phase. What all measuring modes have in common is that a reliable determination of the water vapor transmission rate is only possible in a steady state. That means, first, a constant water vapor gradient must be formed in the examined barrier film. Secondly, a steady state must be established between the water vapor adsorbed at the surfaces of the measuring cells and samples and the water vapor concentration in the gas phase.
Considering the demands such as low head conduction over edges, commercial availability, laminateblity, etc., we tested the following metallized barrier films with respect to their water vapor transmission rates:

Table 1: Excamined selected barrier films

Film number	Producer	Title	Film structure
1	Hanita	V07421	2-times coated with aluminum
2	Hanita	V08621	3-times coated with aluminum
3	Rexor	Rexotherm CIV 8 C 90	3-times coated with aluminum
4	Braun	Vakuumfolie	2-times coated with aluminum
5	Reuther	VIP 270730	unknown

3 Summary of the results

3.1 WVTR comparison of selected films

At first the WVTR of the selected films (see data sheet above) were tested. Three samples of each film type were measured for 48 h at 38 ° and 90 % R. H. The determined transmission rates are all lower than the WVTR-values, given in the data sheet.

Table 2: Comparison of measured WVTR with data sheet values

film number	manufacturer	name	WVTR / g m^{-2} d^{-1} datasheet	WVTR ° / g m^{-2} d^{-1} HiBarSens	standard dev. g m^{-2} d^{-1} HiBarSens
1	Hanita	V07421	< 0.02**	0,0141 *	0,0020
2	Hanita	V08621	< 0.01**	0,0086*	0,0001
3	Rexor	Rexotherm CIV 8 C 90	< 0.03*	0,0137*	0,0019
4	Braun	Vakuumfolie		0,3000*	0,0128
5	Reuther	VIP 270730	< 0.1*	0,0083*	0,0004

* 38°C 90 % R. H.
** 38°C 100 % R. H.
° 48 h mesurement time

In order to define the dynamical behavior, the breakthrough time of a barrier film (Hanita V08621) at 38 °C, was determined to be 95 hours after an increase of the relative humidity from 0 % R.H. to 90 %R.H. 240 hours are already required to reach 90 % of the WVTR value, determined in a steady state. A reliable WVTR measurement is only possible after a complete breakthrough and an establishment of all steady states. Before that, the WVTR value is primarily determined by the outgassing process of the vapor, bounded in the barrier film as well as the evacuation speed of the vapor from the surrounding gas phase. The outgassing process is further dependent upon how the barrier film was stored before the measuring process. If the barrier film is stored at a low relative humidity, the measuring curve will show a flat minimum after reaching the steady state level. This can lead to a false interpretation of a stationary condition and thus to an underdetermination of the actual WVTR by early ending the measurement. After a test duration of more than 10 days the WVTR of the barrier film (Hanita V08621) is 68 %(1.68×10^{-2} g m^{-2} d^{-1}) above the data sheet specified WVTR and 98% over the 48 h WVTR rate. Measuring times of more than 48 h are not compatible with a screening of different thermal and mechanical stresses. Therefore only 48 h samples tests in laboratory conditions were performed. This method does not determine the actual water vapor transmission rate, but significant damages can quickly and reliably be detected.

3.2 Thermal aging

The barrier films were thermally aged according to the BIBt (Deutsches Institut für Bautechnik) method. This procedure mirrors a real aging process of approximately 5 – 10 years. Three barrier films of each film type were tested after different aging periods. The 48 h rapid test showed no significant influences on the water vapor transmission rate of the barrier films due to aging processes. (inside the margin of error).

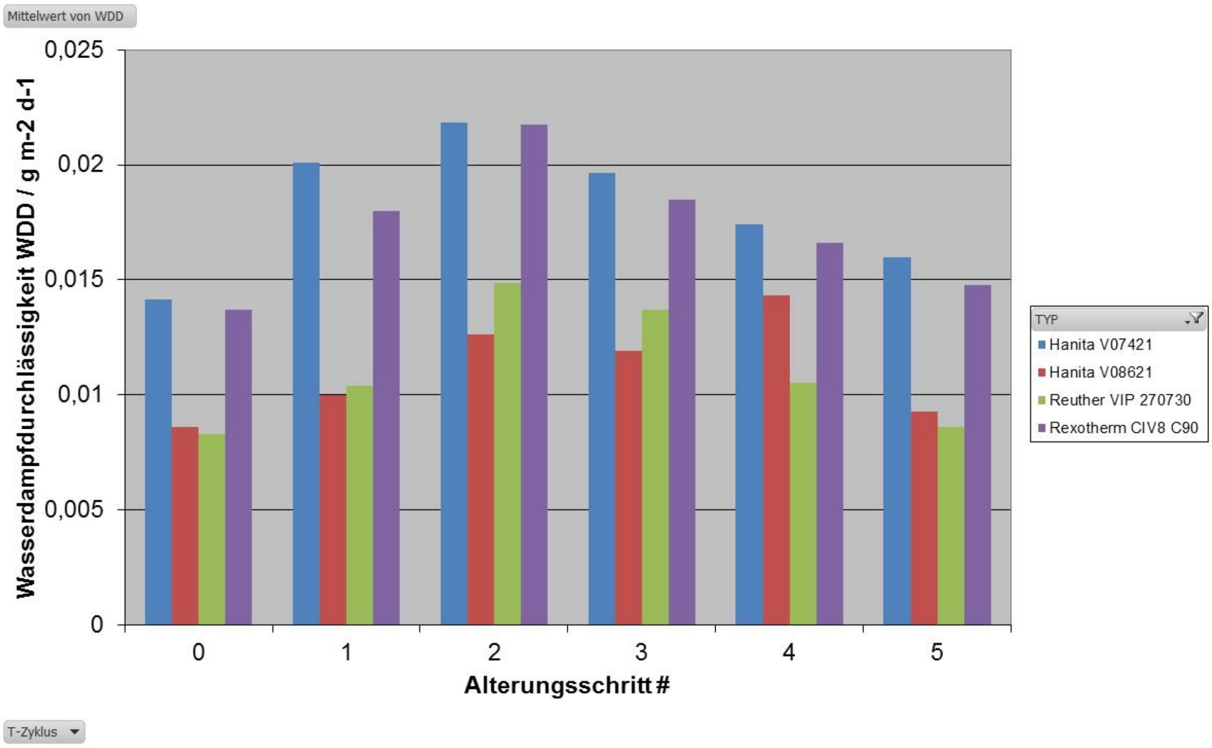

Figure 1: measured WVTR of barrier films after aging step 0-5 (38 °C, 90 % R.H..)

The storage conditions are solely responsible for the typical noticed progression of WVTR during increasing aging processes. Here the WVTR values approach those of untreated samples after a maximum value. The untreated samples were stored during winter time at low humidity. Thus they dissolved less water than those samples being stored in summer at high humidity. The test after the 5th aging step was again performed during winter, so that the sample was again exposed to only a low humidity.

3.3 Mechanical stress

To examine the mechanical stress upon the sample`s WVTR a bending test and a mechanically alternating load test with 164,000 cycles were performed. The bending test was done with 90° and 180° foldings without and with kinks (crossing point of the folds). Only the test with 180° foldings verified an increased WVTR of 20 – 40 % (compared to raw films). If this higher permeability is related to the kink length, the WVTR will only be 2.6 % higher for a VIP with a size of 500 x 500 x 20 mm³. This result shows that the selected barrier films are sufficiently resistant against the loads, to which VIPs are exposed.

3.4 Sealed seam measurements

A sealed seam is the area, where two barrier films are bonded to each other or laminated. There is no protecting metallization and thus there is a path through which vapor or other gases can enter. This process is called lateral diffusion. The water vapor transmission rate at the joining zone is determined by the material used, their geometrical dimensions (height and width) and their processing (cleanliness, temperature, surface pressure). With respect to measuring, the analysis is a genuine challenge, since seams have different shapes and geometries and sealing measurement flanges is very difficult.

To judge if or if not a sealed seam is leak-proof (a decisive criterion of VIPs), a measuring adapter was developed. This adapter measures the WVTR of samples with different sealed seams and relations between seal length and film surface.

Figure 2: Testing sample with seal seam adapter in a climate chamber

First tests with a seal measuring adaptor showed a 30 % higher water vapor transmission rate for samples with sealed seams compared with barrier films without seals. Using the barrier film Hanita V08621, a VIP of the size 500 x 500 x 20 mm³ shows a water vapor transmission rate increase of merely 4.4 %. Further systematic analyses with different barrier films and seal parameters are desirable.

3.5 Conclusions for the pressure increase of VIPs and the heat conductivity

Within the scope of this project the WVTR temperature dependence of barrier films was examined. These data allow for the calculation of the pressure increase in the VIP due to actual annual temperature patterns. Here, water vapor adsorption is of decisive importance for the calculation. Due to an adsorption isotherm the influence of the adsorption upon the inner pressure could be considered.

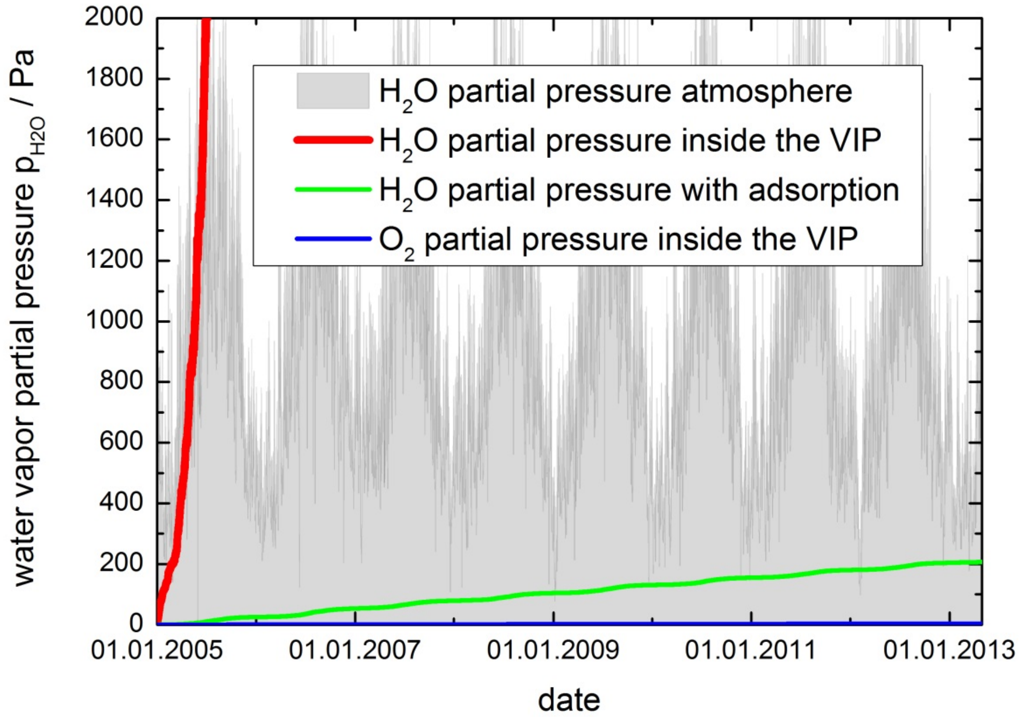

Figure 1: Simulation of pressure increase in a VIP

With the help of the demonstrated approach it is possible to predict the lifespan of VIPs with sizes and supporting structures. Considering the temperature dependence of the WVTR of the barrier film, the water vapor adsorption isotherm of the supporting surface structure as well as the actual temperature pattern, when using Hanita V08621, a simulation shows a change of the heat conductivity of 0.03 x 10^{-3} W $^{m-1}$ K^{-1} a^{-1}.

In comparison: Sprengard ([5] figure 27) measured a heat conductivity increase, due to the aging process, of 0.1 x 10^{-3} W m^{-1} K^{-1} a^{-1} .Theses measurements were done at 23° and 80 % R.H. The three-fold difference can plausibly be explained by different film material.

To summarize: the pressure increase in the VIP is decisively determined by the water vapor transmission rate. The WVTR and thus the inner pressure in the VIP increases exponentially with higher temperatures and higher atmospheric humidity concentrations. With the help of water vapor transmission rate determinations it is possible to evaluate the suitability of a barrier film for vacuum insulation panels. The lifespan of a VIP can be estimated even after a few days testing. A permeation test should be performed right at the beginning of every production series, since the WVTR of the barrier films may vary due to production.

4 Literature reference

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