NORWEGIAN UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF CIVIL AND TRANSPORT ENGINEERING

| Title: Ageing Effects on Thermal Properties and Service Life of Vacuum Insulation Panels | Date: 09.06.2010<br>No. of pages (w/ appendixes): 88 | | | |
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| | Master thesis | X | Project Work | |
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Abstract:

Vacuum insulation panels (VIPs) represent a high performance thermal insulation material solution offering an alternative to thick wall sections and large amounts of traditional insulation in modern buildings. Thermal performance over time is one of the most important properties of VIPs to be addressed, and thus the ageing effects on the thermal properties have been explored in this work.

Laboratory studies of ageing effects are conducted over a relatively limited time frame. To be able to effectively evaluate ageing effects on thermal conductivity, accelerated ageing experiments are necessary. As of today, no complete standardized methods for accelerated ageing of VIPs exist. By studying the theoretical relationships between VIP properties and external environmental exposures, various possible factors for accelerated ageing are proposed. The factors that are found theoretically to contribute most to ageing of VIPs are elevated temperature, moisture and pressure. By varying these factors it is assumed that a substantial accelerated ageing of VIPs can be achieved.

Four different accelerated ageing experiments have been performed to study whether the theoretical relationship may be replicated in practice. To evaluate the thermal performance of VIPs, thermal conductivity measurements have been applied.

The different experiments gave a varying degree of ageing effects. Generally the changes in thermal performance were small. Results indicated that the acceleration effect was within what could be expected from theoretical relationships, but any definite conclusion is difficult to draw due to the small changes. Some physical changes were observed on the VIPs, i.e. swelling and curving. This might be an effect of the severe conditions experienced by the VIPs during testing, and too much emphasis on these should be avoided.

Keywords:

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| 1. VIP |
| 2. Vacuum insulation panels |
| 3. Accelerated ageing |
| 4. Building insulation |

(sign.)

# Preface

This work is part of the compulsory activities required of graduate students at the Norwegian University of Science and Technology. The work is delivered under the Department of Civil and Transport Engineering.

Results reported in this work have been the basis for a scientific article, which has been submitted to the board of the Research Centre of Zero Emission Buildings (ZEB) before submission to *Journal of Building Physics*.

Working with Vacuum Insulation Panels, as a relatively new and innovative material, has been both interesting and challenging. It has given me the possibility to utilize the knowledge I have acquired during my studies, and at the same time provided new insights into the field of building and material technology. I would like to extend my gratitude to all the staff at the Department of Civil and Environmental Engineering at NTNU and the Department of Building Materials and Structures at SINTEF that has supported this work in different ways. Special thanks are given to Bjørn Petter Jelle at SINTEF and NTNU for his contribution as supervisor.

Erlend Wegger

# Sammendrag

Vakuumisolasjonspaneler (VIP) er en høyisolerende materialløsning som kan være et alternativ til tradisjonell bygningsisolasjon. På grunn av god isolasjonsevne kan man ved bruk av VIP redusere veggtykkelsen og fortsatt tilfredsstille energikravene som stilles til moderne bygninger. En av de viktigste egenskapene for VIP er evnen til å bevare høy termisk ytelse over tid. I den sammenheng har aldringseffekter for VIP blitt undersøkt.

Siden laboratoriestudier av aldringseffekter gjøres i løpet av et relativt kort tidsrom, er akselerert aldring nødvendig for å få evaluert termiske egenskaper over tid. Det finnes pr. i dag ingen standardisert metode for akselerert aldring av VIP. Det finnes likevel flere studier av sammenheng mellom klimaforhold og VIP egenskaper. Spesielt er gass og fuktdiffusjon inn i panelet behandlet grundig i litteraturen. Basert på dette er det foreslått flere mulige faktorer for aldring av VIP. De faktorene som er funnet å bidra mest til aldring av VIP er temperatur, fuktinnhold i lufta og utvendig lufttrykk. Ved å variere disse faktorene er fire forskjellige aldringsforsøk beskrevet og gjennomført. Konduktivitetsmålinger er blitt brukt som et mål på de termiske egenskapene til de testede VIPene.

De forskjellige forsøkene viste forskjellig grad av aldringseffekt. Generelt var endringen i konduktivitetsverdier liten. Resultatene indikerer at akselerasjonseffekten var innenfor hva som kan forutsies fra de teoretiske sammenhengene. Likevel er det vanskelig å trekke noen definitive konklusjoner, både siden endringen var så liten, og fordi få paneler ble brukt i forsøkene.

Noen fysiske endringer ble observert under forsøkene. Blant annet este et av panelene noe ut, mens et annet bøyde seg permanent. Man burde likevel ikke legge for mye vekt på disse effektene, siden de kan skyldes de relativt ekstreme testforholdene.

# Contents

## Structure of the Master Thesis

The main part of this master thesis consists of a scientific article concerning accelerated ageing of VIPs. A somewhat abridged version of this article is submitted to the board of the Research Centre of Zero Emission Buildings (ZEB) before submission to *Journal of Building Physics.* Chapters 5, 6, 7.1, 9.2 and 9.6.3 is not included in the abridged version, otherwise the articles are identical. The reason for the abridgement is that the topics covered in the omitted chapters is considered to extensive and detailed for a scientific journal.

In addition the appendixes include supplementary work and details on some of the performed experiments, which were not reported in the article. The following work is included in the appendix:

Appendix A – Details on how thermal conductivity measurements were performed on small vacuum insulation panels

Appendix B – Details on temperature measurements on the vacuum insulation panel protected by a wall construction and placed in a vertical climate simulator

Appendix C – Analytical solution of a differential equation for the internal gas pressure increase of VIPs, with respect to external pressure.

Appendix D – E. Wegger, B.P. Jelle, E. Sveipe, S. Grynning, R. Baetens, J.V. Thue, ”Ageing Effects on Thermal Properties and Service Life of Vacuum Insulation Panels”, Proceedings of the *Building Enclosure Science & Technology (BEST 2 - 2010),* Portland, Oregon, U.S.A., 12-14 April, 2010.

Appendix E – Abstract submitted for the proceedings of the Renewable Energy Research Conference, Trondheim, June 2010. Focuses on the experimental part of the master thesis

Appendix F – E. Wegger, B.P. Jelle, E. Sveipe, S. Grynning, A. Gustavsen, J.V. Thue, “Accelerated Ageing of Vacuum Insulation Panels”, Proceedings of the *Renewable Energy Research Conference*, Trondheim, Norway, June 2010

Appendix G – Abstract submitted for the 12$^{th}$ International Conference on Durability of Building Materials and Components, Porto, Portugal, April 2011

Appendix H – Presentation given at the Renewable Energy Research Conference, Trondheim, June 2010. Focuses on the experimental part of the master thesis.

# Ageing Effects on Thermal Properties and Service Life of Vacuum Insulation Panels

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## Abstract

Vacuum insulation panels (VIPs) represent a high performance thermal insulation material solution offering an alternative to thick wall sections and large amounts of traditional insulation in modern buildings. Thermal performance over time is one of the most important properties of VIPs to be addressed, and thus the ageing effects on the thermal properties have been explored in this work.

Laboratory studies of ageing effects are conducted over a relatively limited time frame. To be able to effectively evaluate ageing effects on thermal conductivity, accelerated ageing experiments are necessary. As of today, no complete standardized methods for accelerated ageing of VIPs exist. By studying the theoretical relationships between VIP properties and external environmental exposures, various possible factors for accelerated ageing are proposed. The factors that are found theoretically to contribute most to ageing of VIPs are elevated temperature, moisture and pressure. By varying these factors it is assumed that a substantial accelerated ageing of VIPs can be achieved.

Four different accelerated ageing experiments have been performed to study whether the theoretical relationship may be replicated in practice. To evaluate the thermal performance of VIPs, thermal conductivity measurements have been applied.

The different experiments gave a varying degree of ageing effects. Generally the changes in thermal performance were small. Results indicated that the acceleration effect was within what could be expected from theoretical relationships, but any definite conclusion is difficult to draw due to the small changes. Some physical changes were observed on the VIPs, i.e. swelling and curving. This might be an effect of the severe conditions experienced by the VIPs during testing, and too much emphasis on these should be avoided.

**Keywords:** *Vacuum insulation panel, VIP, Building insulation, Service life prediction, Ageing properties, Accelerated ageing*

# 1 Introduction

Use of thermal insulation in buildings has experienced an enormous increase since the 1970s. Although most insulation materials were developed prior to 1960, it was only after the 1973 oil crisis that thermal insulation became the preferred way to improve a building's energy efficiency. Since then the required energy efficiency has increased steadily. In Norway the requirement of a wall construction in 2010 is an U-value of 0.18 W/(m$^2$K), which is equivalent to 250 mm mineral wool insulation. Future requirements in order to obtain zero emission standards may require wall thicknesses up to 500 mm filled with mineral wool. Obviously these kinds of wall thicknesses and amounts of insulation are a challenge both for architects and engineers in building aesthetically, economically and in accordance with building physical principles.

Vacuum insulation panels (VIP) may offer a solution to this problem. VIPs consist of a solid, porous core which is sealed with an air- and watertight foil while there is a vacuum in the core. It has thermal conductivities that are 5-10 times lower than for traditional thermal insulation. It will thus be possible to reduce the thickness of the walls, but retain, or even increase, the thermal resistance. So far VIPs have been used mostly in refrigerators and cold-shipping boxes. In recent years, however, a lot of research has been put into introducing VIPs on the building market.

Germany and Switzerland were some of the first countries to support this kind of research. The largest research and development effort so far has been within the International Energy Agency (IEA) Implementing Agreement; Energy Conservation in Buildings and Community Systems (ECBS) (IEA/ECBCS 2005a).

In the last decade extensive studies have been performed to assess the thermal properties of VIPs over time. These properties are vital to the evaluation of the service life of VIPs. Several studies have been conducted under the IEA/ECBCS project (2005a).

The most important features for evaluating service life of VIPs are the permeation of gases and water vapour through the barrier foil, and the response in the core material to these alterations. Permeation rates for different envelopes and different temperature and moisture conditions have been evaluated experimentally by Schwab et al. (2005a,b). Simmler and Brunner (2005a,b) have studied internal pressure increase over time for varying temperature and moisture content. The effect of absorbed water in the core material on the total thermal conductivity has been investigated by Heinemann (2008). Morel et al. (2007, 2009) did extensive studies on the moisture effects on physical properties of the silica core material. Based on results from all these studies models for service life prediction and for the increase in internal pressure and moisture content have been proposed, among others by Schwab et al. (2005a,b,c) and Tenpierik (2009). There have also been some studies into the in situ performance of VIPs (Brunner and Simmler 2008). An account of the results and progress so far can be found in the IEA/ECBS, Annex 39, Subtask A (IEA/ECBCS 2005a) and in Baetens et al. (2010a).

There have been few studies, however, into accelerated ageing of VIPs. Currently there exists no common understanding of how a realistic accelerated ageing experiment should be conducted. Some effort was put into this study by Simmler & Brunner (2005a,b), where a strong correlation between severe hygro-thermal conditions (high moisture content and high temperature) and internal pressure increase was found. In addition, results found by Schwab et al. (2005 a,b,c) provide valuable insight into the physics of vacuum insulation panels, which could be developed analytically to evaluate the effect of accelerated ageing. This article presents the theoretical background for ageing of VIPs and the formulas and plots relevant for predicting service life of VIPs and the acceleration factors for various procedures. The background for and how accelerated ageing of VIPs may be carried out is discussed. Finally, a variety of ageing experiments are presented to evaluate the theoretical predictions, and to increase the understanding on the effect of various accelerated ageing procedures on VIPs.

# 2 VIP Buildup

VIPs consist of a porous core wrapped in an air- and vapourtight envelope. Various different materials and solutions exist for both core material and envelope.

## 2.1 Core

Several materials have been applied as core materials for VIP. Examples of possible materials are polyurethane, extruded polystyrene (XPS) and various forms of silica. The common characteristics that are needed from a core material are:

- Low thermal conductivity
- Ability to withstand atmospheric pressure
- An open pore structure for easy evacuation of air from the material

The core material might have a great impact upon the thermal performance of VIPs.

## 2.2 Envelope

The main purpose of the envelope is to conserve vacuum in the VIP by preventing water vapour and other air gases from entering it. Various material solutions have been applied for this purpose. In addition to providing a vapour barrier, the envelope must have sufficiently low thermal conductivity to avoid thermal bridges at the panel edges. Experiments show that in most cases the edge effect of the VIP on the thermal conductivity cannot be neglected (Ghazi Wakili et al. 2004, Willems et al. 2005).

The most common envelopes consist of a number of metalized polymer films, alternatively thin metal sheets. Generally, the metal sheets provide the best barrier against air and vapour penetration, but the large thermal conductivity makes them unsuitable for application in VIPs.

The labeling of the most common films used are as follows (Willems et al. 2005):

**Metal Film (AF)** – A central aluminium layer with thickness up to 10 μm is used. This layer is laminated with a polyethylene teraphtalate (PET) layer to provide some mechanical resistance.

**Metalized Films (MF)** – These laminates have up to three layers of aluminum-metalized polyethylene terephthalate (PET) or polypropylene (PP) sheets.

Crossections of four different MF laminates and one AF laminate are shown in Fig. 1. All laminates have an inner polyethylene (PE) layer for sealing purposes. In Fig. 2 a microscopy image of a MF3 laminate is visualized.

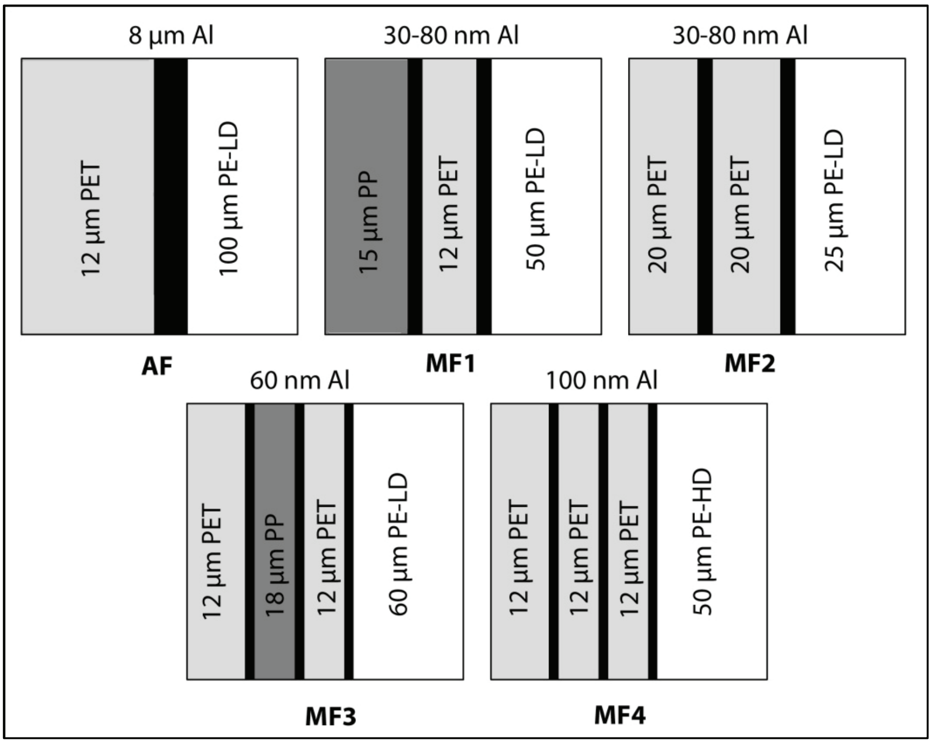


**Fig. 1 Crossections of various envelope solutions for application in VIPs. The laminates and the various layers are not drawn to scale. The names and buildup of the laminates are consistent with what is reported in IEA/ECBCS Annex39. The thickness of the Al-layer denotes thickness of each separate layer (i.e. 60 nm for MF3).**

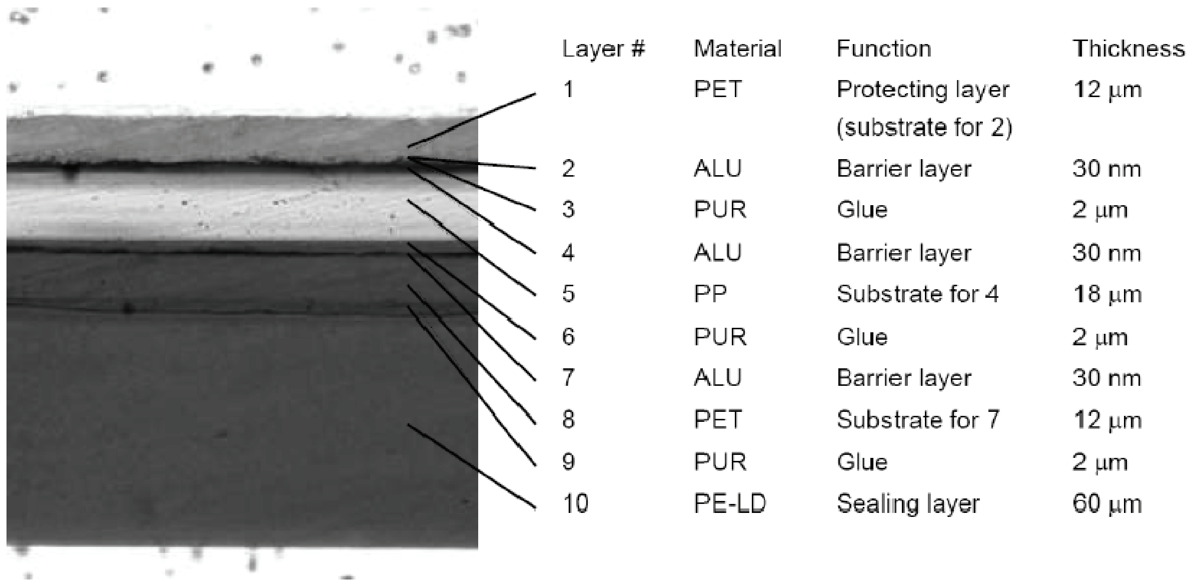


**Fig. 2 Visualization of a multilayer laminate for use as a VIP envelope. (From Simmler and Brunner 2005a).**

A weakness of the MF laminates, compared to the more massive AF laminates is the moisture permeance. However, service lives of several decades are still achievable with the use of MF laminates in normal building application (Simmler and Brunner 2005b).

# 3 Thermal Conductivity of VIPs

The thermal conductivity of VIPs is dependent on several factors, both internal and external. The theoretical relationships governing this and the necessary background for predicting ageing effects will be explored here.

The thermal conductivity ( $\lambda_{tot}$ ) in a material with coherent internal structure (i.e. no coupling effect) can be described as (Brodt 1995):

$$\lambda_{tot} = \lambda_{cd} + \lambda_g + \lambda_r + \lambda_{cv} \tag{1}$$

where

- $\lambda_{cd}$ = solid conduction within material skeleton (W/(mK))
- $\lambda_g$ = gas conduction within the material pores (W/(mK))
- $\lambda_r$ = radiation heat transfer between internal pore surfaces (W/(mK))
- $\lambda_{cv}$ = air and moisture convection within pores (W/(mK))

In addition, a coupling term can be included to account for the interaction between the gas molecules and the pore walls. The coupling effect can be quite complex and will be neglected in the rest of this article. Most theoretical approaches to thermal performance of VIPs, assumes the coupling effect to be negligible.

The high thermal performance of VIPs is mostly due to the effect of reduced gas conduction ( $\lambda_g$ ) as pressure decreases in the core material of the VIP. The most effective reduction is achieved at total vacuum, when $\lambda_g$ would approach zero. This is a result of the Knudsen effect. The Knudsen effect, relates gas conductivity to the pore size of a material and the number of gas molecules. As the pressure decrease, the mean free path length of the gas molecules increases. When the mean free path length becomes longer than the average pore size of the surrounding material, only elastic collisions between gas molecules and the pore surface are assumed to occur. As these collisions don't transfer any significant energy, the gas conduction may be reduced towards zero as the pressure decreases.

The influence on gas conductivity from the Knudsen effect can be found from the following relationship (IEA/ECBCS 2005a):

$$\lambda_g = \frac{\lambda_{g0}}{1+2\beta Kn} \tag{2}$$

Where Kn is the Knudsen number,

$$Kn = \frac{l_{mean}}{\delta} \quad \text{and} \quad l_{mean} = \frac{k_B T}{\sqrt{2}\pi d_g^2 P_g} \tag{3}$$

and

$\lambda_{g0}$ = Free air conductivity (W/(mK))

$\beta$ = Constant characterizing the energy transfer efficiency between the gas molecules and the solid state pore walls (between 1.5 and 2.0))

$l_{mean}$ = Mean free path of air (m)

$\delta$ = Characteristic size of pores, e.g. pore diameter (m)

$k_B$ = Boltzmann's constant (J/K)

$T$ = Temperature (K)

$d_g$ = Diameter of the gas molecule (m)

$P_g$ = Gas pressure (Pa)

Equations (2) and (3) are used to obtain Eq. (4), indicating the three main parameters that influence gaseous heat conduction in porous media: Gas pressure, characteristic pore size and temperature (Baetens et al. 2010a).

$$\lambda_g = \frac{\lambda_{g,0}(T)}{1+C\dfrac{T}{\delta P_g}} = \frac{\lambda_{g,0}(T)}{1+\dfrac{P_{1/2,g}}{P_g}} \tag{4}$$

where $P_{1/2,g}$ is the pressure at which thermal conductivity reaches one half the value of $\lambda_{g,0}$ and C is a constant defined as $2\beta k_b \big/ (\sqrt{2}\pi d_g^2)$.

From these relationships it is evident that the choice of core material for VIPs is of vital importance to achieve the desired thermal performance over time, also for increasing pressures. The thermal conductivity versus gas pressure is shown for a range of materials in Fig. 3.

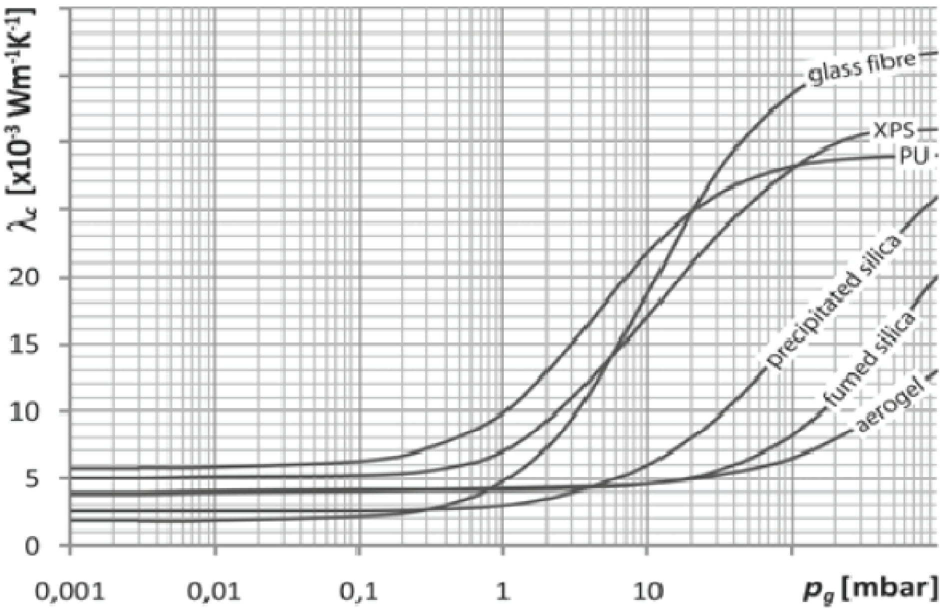


**Fig. 3** **Thermal conductivity versus gas pressure for a variety of materials (From Tenpierik 2009).**

As can be seen, fumed silica and aerogel have reduced conductivity even at atmospheric pressures. In comparison with materials such as mineral wool that would require gas pressures in the range of 0.1 mbar to reduce gas conductivity, these silica based materials are highly suitable for application in VIPs (Caps et al. 2001).

In Fig. 4 the relationship between pore size, gas pressure and thermal conductivity is drawn. From this graphical 3D-plot, the Knudsen effect is apparent.

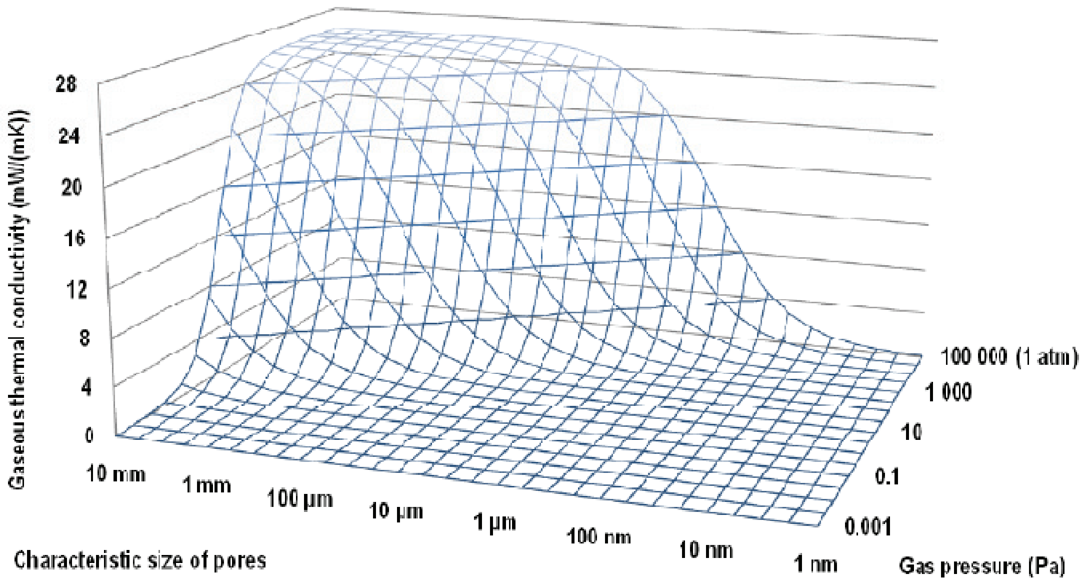


**Fig. 4** **Gaseous thermal conductivity of air (mW/(mK)) as a function of characteristic pore size and gaseous pressure at a temperature of 300 K. Derived from Eqs. (2) and (3) (From Baetens et al. 2010).**

# 4 Ageing of VIPs

As the thermal performance of VIPs are highly dependent on conservation of the vacuum in the panels, all gases that permeate through the envelope will contribute to the reduction of thermal properties of the VIP. Apart from extraordinary mechanical stresses and production failures, gas and moisture transport into the VIPs are considered the most important ageing mechanism to consider when evaluating the performance of VIPs over time.

The means of molecular transport through VIP envelopes depends on the size and properties of the various gas molecules. For oxygen, and other air gases, the transport mainly happens at macroscopic defects in the envelope material in the order of 0.1-1.0 µm$^2$. For the permeation of water vapour the main transport is dissolution of molecules in the polymers, and the condensation in capillaries. Generally it can thus be said that for oxygen, the macrostructure of the envelope barrier is vital, while for water vapour also the microstructure of the envelope is important (IEA/ECBCS Annex 39).

## 4.1 Gas Transport

The envelope of the VIPs consists of several different layers depending on the type of laminate. As a result of this it is difficult to specify a permeance for the envelope. Instead an empirical value is employed. This value is referred to as the Gas Transmission Rate (GTR) or the Air Transmission Rate (ATR). The GTR specifies how much of a given gas permeates the VIP envelope during a given time. The ATR is the amount of permeated gas when the VIP is exposed to a mixture of air gases. The total gas transmission rate is defined as (Schwab et al. 2005a)

$$GTR_{tot} = GTR_A(T,\varphi)\cdot A + GTR_L(T,\varphi)\cdot L \tag{5}$$

where

- $GTR_A$ = the surface gas transmission rate of the laminate cover per panel area (m$^3$/(m$^2$s)
- A = total surface area of the VIP with front and rear sides (m$^2$)
- $GTR_L$ = the length related gas transmission rate along the circumference of the panel (m$^3$/(m s)
- L = length of panel circumference (m)

The GTR relates to the laminate permeance ( $Q_{gas,tot}$ ) as (Schwab et al. 2005a)

$$Q_{gas,tot} \equiv \frac{GTR_{tot}}{\Delta p_{gas}} \tag{6}$$

where

- $\Delta p_{gas}$ = pressure difference across laminate barrier

As a result of the gas permeation, a pressure increase occurs inside the panel. This pressure increase depends on the GTR and can be found from Schwab et al. (2005a) to be

$$\frac{dp_{gas}}{dt} = \frac{Q_{gas,tot}\Delta p_{gas}}{V_{eff}}\left(\frac{T_m p_0}{T_0}\right) = \frac{GTR_{tot}}{V_{eff}}\left(\frac{T_m p_0}{T_0}\right) \tag{7}$$

where

$\left(\frac{T_m p_0}{T_0}\right)$ = conversion factor from standard (index 0) to measurement conditions (index m)

$V_{eff}$ = effective pore volume in the VIP ($m^3$)

For service life predictions it is usually assumed that $\Delta p_{gas}$ initially equals atmospheric pressure $\left(p_{atm}\right)$ as the internal pressure is negligible. Then, a linear increase in pressure over time results (Schwab et al. 2005a):

$$p(t) = \frac{Q_{air,tot}\,p_{atm}}{V_{eff}}\left(\frac{T_m p_0}{T_0}\right)t = \frac{GTR_{tot}}{V_{eff}}\left(\frac{T_m p_0}{T_0}\right)t \tag{8}$$

Eq. (7) can also be solved analytically to give an expression for internal pressure as a function of time and external pressure

$$p(t) = p_{app} - (p_{app} - p_{init})e^{\frac{-T_m p_0 Q_{gas,tot}}{T_0 V_{eff}}t} \tag{9}$$

where

$p_{app}$ = applied external pressure (Pa)

$p_{init}$ = initial internal gas pressure of VIP (Pa)

## 4.2 Moisture Transport

Schwab et al. (2005a,b) and Simmler and Brunner (2005b) have performed several experiments to determine the rate at which water vapour permeates through various barrier laminates. This rate is found to vary some with size of panel and measurement conditions.

Because the envelope consists of several materials in various layers, it is difficult to determine a definite permeance for the material. Instead an empirical value called the water vapour transmission rate (WVTR) is employed. The WVTR is defined as (Schwab et al. 2005a)

$$WVTR = \frac{dm_w}{dt} = Q_{wv,tot}\Delta p_{wv} \tag{10}$$

where

$WVTR = \frac{dm_w}{dt}$ = mass increase with time (kg/s)

$Q_{wv,tot}$ = total water vapour permeance (kg/(s Pa)

$\Delta p_{wv}$ = water vapour pressure difference across foil (Pa)

A theoretical relationship can be developed for the increase in water content with time using Eq.(10) and the partial water vapour pressure. The partial vapour pressure can be calculated applying the inverse function of the sorption isotherm $\left(\varphi(X_w)\right)$, according to Eq.(11) (Schwab et al. 2005a)

$$p_{wv} = \varphi(X_w)\, p_{wv,sat}(T) \tag{11}$$

where

$p_{wv}$ = water vapour partial pressure (Pa)

$p_{wv,sat}(T)$ = water vapour saturation pressure depending on temperature (Pa)

$\varphi(X_w)$ = relative humidity depending on water content (-)

The change in water content with time can then be described by (Schwab et al. 2005a)

$$\frac{dX_w}{dt} = \frac{Q_{wv,tot}}{m_{VIP,dry}}\left(p_{wv,out} - p_{wv,in}\right) = \frac{Q_{wv,tot}}{m_{VIP,dry}}\, p_{wv,sat}\left(\varphi_{out} - \varphi_{in}(X_w)\right) \tag{12}$$

where

$m_{VIP,dry}$ = dry mass of the VIP (kg)

$p_{wv,out}$, $p_{wv,in}$ = the water vapour pressure outside and inside the VIP respectively (Pa)

$\varphi_{out}$, $\varphi_{in}$ = the relative humidity outside and inside the VIP respectively (-)

By approximating the sorption isotherm to a linear relationship $X_w = k\varphi$, eq. (12) was solved analytically by Schwab et al. (2005a):

$$X_w(t) = k\varphi_{out}\left(1 - e^{\frac{-Q_{wv,tot}\, p_{wv,sat}(T)}{m_{VIP,dry}\, k}t}\right) \tag{13}$$

where k is a constant representing the slope of the sorption isotherm.

As can be seen from the relationship in Eq.(13), both temperature and relative humidity are factors in determining moisture transport through VIPs. With increasing temperature, the saturation pressure increases exponentially. Combined with an increased RH, this will increase the water vapour pressure difference, and hence the driving force for moisture transport, substantially. From the sorption isotherm of silica the proportionality constant k can be estimated at approximately 0.08 mass% per percent of relative humidity up to 60 % RH.

# 5 Moisture Effects on Core Material of VIPs

As has been seen, various materials might be employed as core material for VIPs. One of the most common material is what is referred to as *pressed fumed silica* or *pyrogenic silica.* Pyrogenic silica is made with a high-temperature combustion process that produces extremely fine particle sized silica (Hannebauer and Menzel 2003).

The reaction of silica on moisture is highly dependent on the surface characteristics of the silica particles. The sorption capacity is to a large extent governed by the presence of two groups of molecules at the surface of the material, siloxanes (Si-O-Si) and silanoles (Si-OH) (Morel et al. 2009). It is especially the silanoles that give silica its hydrophilic character. The OH groups act as a center for molecular adsorption and several layers of water molecules can be adsorbed to the surface. This is the so called “physically adsorbed water” (Zhuralev 2000)

Silanoles are formed through hydroxylation of siloxanes when water is present. This is shown in Fig. 5. The water absorbed in this process is the “chemisorbed water”. Temperatures higher than 200°C are required to reverse the process in what is called dehydroxylation (Morel et al. 2007).

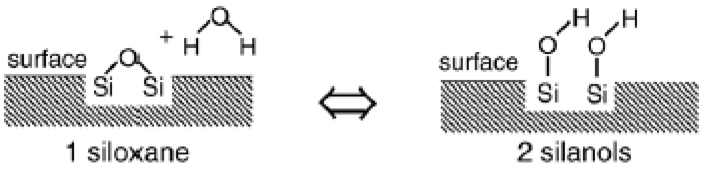


**Fig. 5. Hydroxylation of a siloxane to form two silanoles. This process is the source of the high adsorption capacity of silica. From Morel et al. (2009).**

For pyrogenic silica, which is made under a high-temperature process, Zhuravlev (2000) predicts that the surface will originally be composed mainly of siloxanes. As the presence of water increases, however, a hydroxylation process occurs and the water adsorption can be quite substantial.

As the water adsorption increases several other physical alterations occur in the silica. These alterations were studied extensively by Morel et al. (2007, 2009) for various temperatures and humidities.

**Mass Uptake**

The results from the study showed that for increasing humidity at constant temperature, the mass uptake increased. From this it can be concluded that moisture has a direct impact on mass uptake, which is in accordance with what is predicted among other by Zhuravlev (2000). The mass uptake was quite fast, and was mainly accomplished in 15 days. The same effect was seen during pretreatment of silica samples. Dry samples stored under a $N_2$ stream for 12 hours, and then placed at 20°C and 44 % RH experienced 50 % of its mass uptake within 6 minutes (Morel et al. 2007, 2009).

**Specific Surface Area**

The specific surface area decreased when exposed to high temperature and moisture conditions. The samples at 20°C did not show any alterations, independent of humidity. For 60°C, however, the decrease of specific surface area was bigger for increasing humidity (Morel et al. 2007, 2009).

**Rigidity/ Young’s Modulus**

The results show two trends concerning Young’s modulus. At constant temperature,increasing moisture increases rigidity. And at constant humidity, increasing temperature increases rigidity.

All these effects can possibly be explained in terms of microstructural changes in the silica. Figure Fig. 6 shows how the adsorbed water contributes to the reduced surface area and Fig. 7 depicts how the interfaces between silica particles are assumed to change (Morel et al. 2007, 2009).

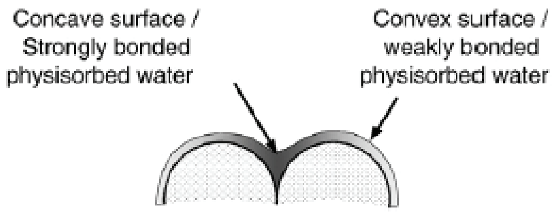


**Fig. 6. Impact on surface from the physically adsorbed water layers on the silica molecules. From Morel et al. (2009).**

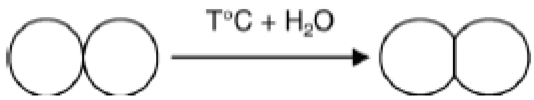


**Fig. 7. Assumed change of interface between silica particles exposed to high temperature and relative humidity. From Morel et al. (2009).**

Note that different silica might have different response to the temperature and humidity conditions. Some tests were performed by Morel et al. (2007) on various silicas, but the results are few, and it is difficult to draw any definite conclusions. However, the various silica showed different response to the same climatic conditions.

During tests on VIPs with different water contents and drying cycles, both Heinemann (2008) and Beck et al. (2007) found a reduction of thickness on the moist panels. One possible explanation of this might be shrinkage due to high capillary forces at high humidity, but this has not been verified experimentally.

# 6 Moisture Effects on Thermal Performance of VIPs

Thermal conductivity for dry VIPs ( $\lambda_{dry}$ ) consists of solid conduction, radiation, convection and gas conduction. Assuming the various parts are independent of each other, the thermal conductivity can be calculated as shown in Eq. 1 above.

Beck et al. (2007) argues that as water vapour infiltrates the VIP, additional possibilities for heat transport occurs. The thermal conductivity for a moist VIP ( $\lambda_{moist}$ ) should therefore be calculated as:

$$\lambda_{moist} = \lambda_r + \lambda_{cd} + \lambda_g + \lambda_{cv} + \lambda_{ad} + \lambda_{wv} \tag{14}$$

Where

$\lambda_{ad}$ = thermal conductivity in adsorbed water in the core

$\lambda_{wv}$ = thermal conductivity due to conduction in water vapour

The effects of moisture on the thermal properties of VIPs have been the subject of many studies. It is generally accepted that the most common heat transport processes involving moisture are (Schwab et al. 2005c, Heinemann 2008 and Beck et al. 2007):

- Thermal conduction of water vapour
- Thermal conduction of adsorbed water and liquid water at particle surfaces
- Thermal transfer by a heat-pipe effect, where water evaporates at the warm side of a VIP and condenses on the cold side

These effects will be discussed separately.

## 6.1 Thermal Heat Conduction of Water Vapour

The amount of water vapour present inside the VIP is highly dependent upon the sorption characteristic of the core material, and the temperature of the panel. With changing temperature, the core material adsorbs or desorbs moisture according to the sorption isotherm. The sorption isotherms for a typical silica based core material, as found by Schwab et al. (2005b) are shown in Fig. 8.

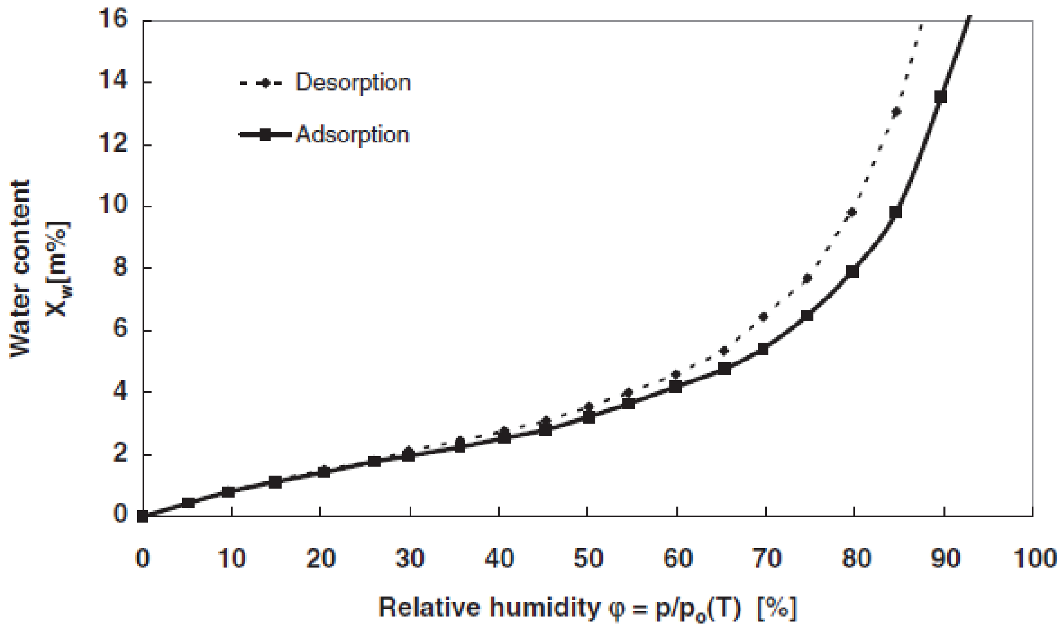


**Fig. 8. Sorption isotherms for a typical silica based core material derived at 23°C. From Schwab et al. (2005b).**

As can be seen from the adsorption isotherm in Fig. 8, the adsorption is almost linear up to about 65 % RH.

The gaseous thermal conductivity for VIPs, depending on the gas pressure can be described by the following relationship (Fricke et al. 2006):

$$\lambda_g(p_g) = \frac{\lambda_{g,0}}{1 + p_{1/2}/p_g} \tag{15}$$

where

$\lambda_{g,0}$ = gas conductivity of free, still gas (W/(mK))

$p_{1/2}$ = gas pressure at which gaseous thermal conductivity is equal to half of $\lambda_{g,0}$ (Pa)

$p_g$ = gas pressure (Pa)

Using this relationship for water vapour, with $\lambda_{g,0}$=0.016 W/(mK) and $p_{1/2}$=120 mbar, the influence on thermal conductivity from water vapour may be calculated.

## 6.2 Thermal Heat Conduction of Liquid Water

Pyrogenic silica as a core material has a high adsorption capacity for high relative humidities. Water adsorbed on the surface of silica contributes to the solid thermal conductivity of the core material. The increase in thermal conductivity depends on the amount of adsorbed water, which again depends on the sorption isotherm, as stated above. The adsorbed water quantity is only about 5 mass% at 60 % RH, but increase substantially up to 90 % RH with about 14 mass%. The large increase from 60 % to 90 % RH is due to capillary condensation in the small pores, according to Quenard and Sallée (2005).

Schwab et al. (2005c) showed that the thermal conductivity of a moist VIP can be described as a function of the water content according to:

$$\lambda(X_w) = \lambda_0 + a_m X_w \tag{16}$$

where

$\lambda_0$ = thermal conductivity of a dry VIP (W/(mK))

$a_m$ = proportionality constant (W/(mK mass%))

$X_w$ = water content of VIP (mass%)

In Schwab et al. (2005c) $a_m$ is found as $5 \cdot 10^{-4}$ W/(mK mass%). This value is, however, not corrected for the presence of water vapour. In Schwab (2004) the value, after correction for water vapour, is found as $2.9 \cdot 10^{-4}$ W/(mK mass%). This corresponds well with values found by Beck et al. (2007) of $5 \cdot 10^{-4}$ W/(mK mass%) without correcting for water vapour, and $2.4 \cdot 10^{-4}$ W/(mK mass%) when correcting for water vapour. Note that these values only apply to the specific measurement conditions. However, they allow rough estimation of the influence of water vapour (Schwab et al. 2005c)

## 6.3 Heat Pipe Effect of Water Circulation

VIPs used as building insulation will naturally be exposed to a temperature difference from one side of the panel to the other. Because of this, the relative humidity will vary with temperature according to Eq.(11), over the panel. As the temperature goes down, saturation vapour pressure will decrease, and the relative humidity will increase. This will increase adsorption in this part of the panel, lowering the vapour pressure. Finally, this will cause a vapour gradient over the panel, which might lead to vapour transport from the warm to the cold side of the panel. This effect was studied by Heinemann (2008), who simulated the moisture distribution for a range of temperatures, moisture contents and temperature gradients. A quite substantial redistribution of moisture from the warm to the cold side of the VIP was found. However, some moisture are assumed to transfer back along the solid core of the material, thus reducing this effect somewhat (Heinemann 2008)

The water circulation in a VIP will thus be as depicted in Fig. 9.

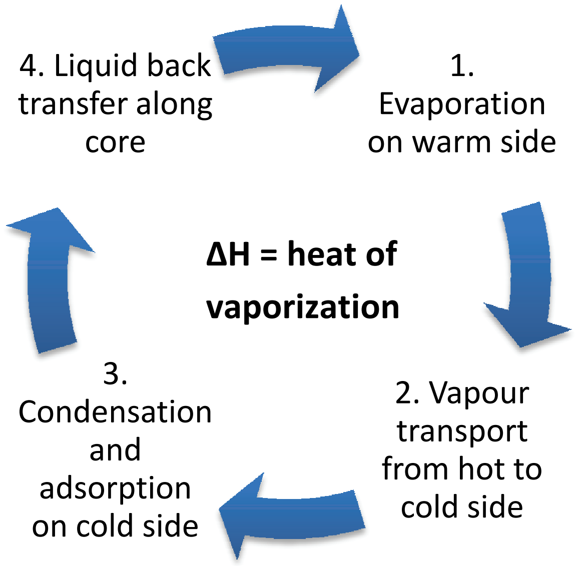


**Fig. 9 Process of water circulation in a VIP with a temperature gradient.**

Due to the process shown in Fig. 9, heat of vaporization will transfer from the warm to the cold side of the VIP, constituting a heat transport mechanism in itself. As an effect of this process it is the coldest side of the VIP that is determining for the maximum water vapour pressure inside the panel.

# 7 Service Life of VIPs

The development of VIPs as a viable alternative to traditional insulation materials is highly dependent upon its ability to retain its high thermal performance over time. Models to predict the ageing effects on VIPs have been developed by Tenpierik (2009) and Schwab et al. (2005a), which can be used to estimate the service life of VIPs. Some studies have been performed on the in situ performance of VIPs in various applications in building insulation. These relevant models and studies will be outlined below.

## 7.1 Service Life Definitions

In ISO 6707-1 service life is defined as "period of time after installation during which a building or its part meet or exceed the performance requirement(s)".

In development and application of ageing models, Tenpierik (2009) employs the following definition, which will also be used for this article:

**Service life definition –** The time elapsed from the moment of manufacturing (t=0) until the moment the effective thermal conductivity of the vacuum insulation panel ( $\lambda_{eff}$ ) has exceeded a certain critical value ( $\lambda_{cr}$ , ($t=t_{sl}$)). In other words, the service life has expired if the following condition has been reached:

$$\lambda_{eff}\Big|_{t=t_{sl}} = \lambda_{cr} \tag{17}$$

Note that this definition uses the effective thermal conductivity of the VIP, which means that edge effects such as thermal bridging are included. In most literature, however, the center of panel thermal conductivity is used, neglecting edge effects. This will also be done in this article, assuming that edge effects are constant over the VIP lifetime and negligible. This is a somewhat imprecise assumption, but is considered sufficient for a theoretical study of ageing effects. Any eventual edge effects can be incorporated as (Ghazi-Wakili et al. 2004):

$$\lambda_{eff,aged} = \lambda_{cop,aged} + \Delta_{edge} \tag{18}$$

Where

$\lambda_{eff,aged}$ = effective thermal conductivity of aged VIP (W/(mK))

$\lambda_{cop,aged}$ = center of panel thermal conductivity of aged VIP (W/(mK))

$\Delta_{edge}$ = change in thermal conductivity relating to edge effects (W/(mK))

For a population of VIPs, a bathtube curve as seen in Fig. 10 can be used to describe the service life.

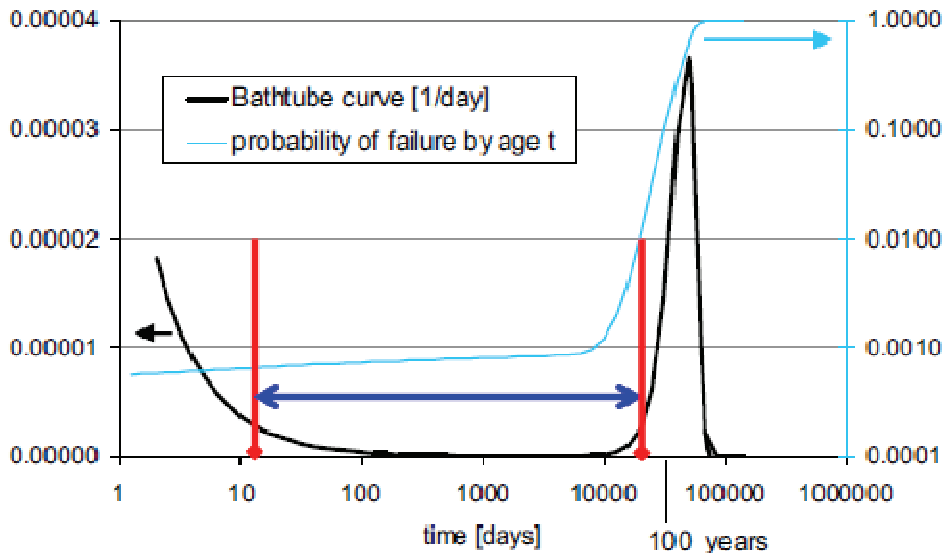


**Fig. 10. Example of a bathtub curve to describe the service life of a population. (From Simmler and Brunner 2005a).**

The bathtube curve show an infant mortality period, where production failures and failures during handling or installation occur, one extended “normal period” with low failure rate, and eventually a wear out period with increasing failure rate.

Initially the infant mortality for VIPs was quite high, but experience and refinement in production and handling have decreased this rate substantially (Simmler and Brunner 2005a).

## 7.2 Prediction Models

Assuming that gas pressure and water content can be treated as thermal resistances in parallel, Schwab et al. (2005a) propose that thermal conductivity as a function of time can be written as:

$$\lambda(t) = \lambda_{evac} + \frac{\lambda_{air,0}}{1 + p_{1/2,air} / p_{air}(t)} + bX_w(t) \tag{19}$$

where

$\lambda_{evac}$ = Thermal conductivity in evacuated state (W/(mK))

$\lambda_{air,0}$ = Thermal conductivity of free and still air (W/(mK))

$p_{1/2,air}$ = The pressure at which thermal conductivity of the gas equals half of $\lambda_{air,0}$ (Pa)

$p_{air}$ = Pressure inside VIP (Pa)

$b$ = Constant dependent on the sorption isotherm (W/(mK mass%)

$X_w(t)$ = Moisture content (mass%)

In this model, the effect of water vapour is not included in a separate term, but is incorporated into the term for dependence on water content.

Based on the function in Eq. (19) and results from Simmler and Brunner (2005b) and Schwab et al. (2004, 2005a) Tenpierik (2009) propose the following model:

$$\Delta\lambda_c = \frac{\partial\lambda_c}{\partial p_g}\Delta p_g + \frac{\partial\lambda_c}{\partial p_{wv}}\Delta p_{wv} + \frac{\partial\lambda_c}{\partial u}\Delta u$$
$$\approx \frac{\partial\lambda_c}{\partial p_g} p_{g,e}(1-e^{-(t-t_{get})/\tau_g}) + \frac{\partial\lambda_c}{\partial p_{wv}} p_{wv,e}(1-e^{-(t-t_{des})/\tau_w}) + \frac{\partial\lambda_c}{\partial u}\frac{du}{d\varphi}\varphi_e(1-e^{-(t-t_{des})/\tau_w}) \tag{20}$$

where

$p_g$ = Pore gas pressure (Pa)

$p_{g,e}$ = Atmospheric gas pressure (Pa)

$p_{wv,e}$ =Partial water vapour pressure outside the VIP (Pa)

$\varphi_e$ = Relative humidity of the air outside the VIP (-)

*u* = Water content of the core material (-)

*t* = Time (days)

$t_{get}$ and $t_{des}$ = Time shifts due to getters and desiccants (s)

$\tau_g$ and $\tau_w$ are time constants according to:

$$\tau_g = \frac{\varepsilon V}{GTR(T,\varphi)} \cdot \frac{T_0}{p_o T} \tag{21}$$

$$\tau_w = \frac{\rho_{dry} V}{WVTR(T,\varphi)} \cdot \frac{1}{p_{sat}(T)} \frac{du}{d\varphi} \tag{22}$$

In this model the effect of moisture is split in separate terms for adsorbed water and water vapour.

## 7.3 Prediction Curves

Based on the models in Eqs. (19) and (20), plots can be made that show how thermal conductivity of a VIP changes over time at constant climatic conditions. Since thermal conductivity is a direct result of increased gas pressure and moisture content in the VIP, curves for moisture content and gas pressures over time can also be provided, enabling the prediction of various VIP parameters. This is shown for five different laminates types (AF, MF1-MF4) for panels with size 100 cm x 100 cm x 2 cm in Fig. 12 to Fig. 13 for a period of 100 years. When drawing the plots, it is assumed that all contributions to thermal conductivity can be treated as thermal resistances in parallel, and total thermal conductivity over time, $\lambda_c(t)$, is based on the equation:

$$\lambda_c(t) = \lambda_{evac} + \lambda_g(t) + \lambda_{wv}(t) + \lambda_w(t) \tag{23}$$

Where

$\lambda_{evac}$ = initial thermal conductivity of dry and evacuated panel. Assumed to be 4.0 mW/(mK)

$\lambda_g(t)$ = conduction due to permeation of air gases over time (W/(mK))

$\lambda_{wv}(t)$ = conduction due to permeation of water vapour over time (W/(mK))

$\lambda_w(t)$ = conduction due to absorbed water in the core over time (W/(mK))

These factors are further calculated as shown in Eqs.(24)-(26).

$$\lambda_g(t) = \frac{\lambda_{g,0}}{1 + p_{1/2,g} / p_g(t)} \quad (24)$$

$$\lambda_w(t) = \frac{\partial \lambda_c}{\partial u} \frac{du}{d\varphi} \varphi_e (1 - e^{-t/\tau_w}) \quad (25)$$

$$\lambda_{wv}(t) = \frac{\lambda_{wv,0}}{1 + p_{1/2,wv} / p_{wv}(t)} \quad (26)$$

Where, from Eq.(20),

$$p_{wv} = p_{wv,e}(1 - e^{-t/\tau_w})$$

$$p_g = p_{g,e}(1 - e^{-t/\tau_g})$$

and where $\tau_g$ and $\tau_w$ can be found in Eqs. (21) and (22) respectively.

Input parameters for these curves are found in Table 1.

**Table 1 Input parameters for VIP calculations. ATR and WVTR values are normalized for 23°C, 50% RH and 1 bar.**

| **Properties** | **Barrier envelope materials** | | | | | **Source** |
|---|---|---|---|---|---|---|
| | **AF** | **MF1** | **MF2** | **MF3** | **MF4** | |
| $ATR_A$ ($cm^3/(m^2d)$) | - | 0.016 | -[1] | 0.0034 | 0.0088 | (IEA/ECBCS Annex 39) |
| $ATR_L$ ($cm^3/(md)$) | 0.0018 | 0.0080 | 0.0039 | 0.0091 | 0.0018 | (IEA/ECBCS Annex 39) |
| $WVTR_A$ ($g/(m^2d)$) | 0.0006 | 0.0233 | 0.0057 | 0.003 | 0.0048 | (IEA/ECBCS Annex 39) |
| $WVTR_L$ (g/(m d)) | - | - | - | 0.0008 | 0.0006 | (IEA/ECBCS Annex 39) |
| Activation energy ($E_a$) (kJ/mol) | 26 | 40 | 28 | - | - | Schwab et al. (2005b) |
| Porosity | 90 % | | | | | Quenard and Sallée (2005) |
| Dry core density | 200 kg/$m^3$ | | | | | Quenard and Sallée (2005) |
| $du/d\varphi$ | 0.08 | | | | | Heinemann (2008) |
| $\partial \lambda_c / \partial u$ | 0.29 m*W/(mK)* | | | | | Schwab (2004) |
| $p_{sat}$ | 2775 Pa | | | | | (Calculation example) |
| RH $\varphi$ | 50 % | | | | | (Calculation example) |
| $\lambda_{wv,0}$ | 16 mW/(mK) | | | | | Fricke et al. (2006) |
| $p_{1/2,wv}$ | 120 mbar | | | | | Fricke et al. (2006) |
| $\lambda_{air,0}$ | 25,7 mW/(mK) | | | | | Schwab et al. (2005a) |
| $P_{1/2,air}$ | 593 Pa | | | | | Schwab et al. (2005a) |

[1] Note that an $ATR_A$ value for MF2 was not resolvable because tested on limited panel size. This does not mean that an $ATR_A$ value does not exist for MF2. It can be expected to lie somewhere between the values MF1 and MF3. As an effect of this, the thermal performance for VIPs with MF2 over time is expected to be slightly overestimated.

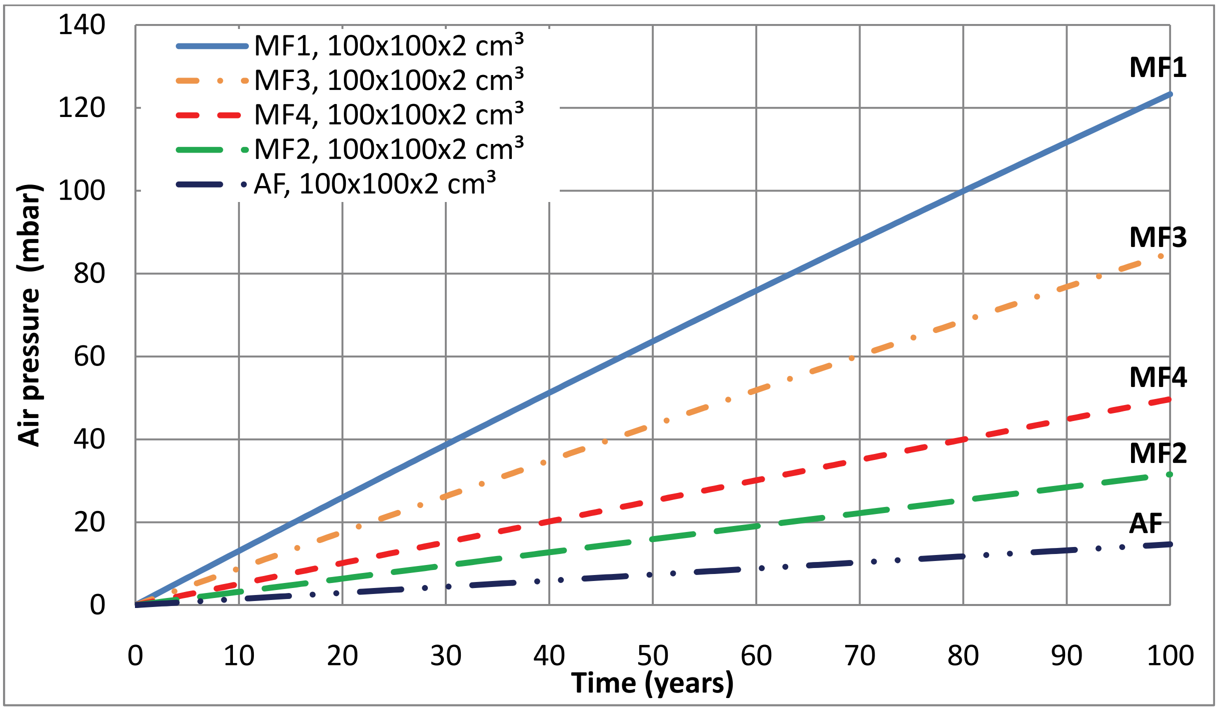


**Fig. 11. Air pressure for various laminate types. The inner air pressure is assumed to be zero at $t_0$=0. It is assumed that laminate properties remains the same during the entire period. No getters and desiccants have been taken into account.**

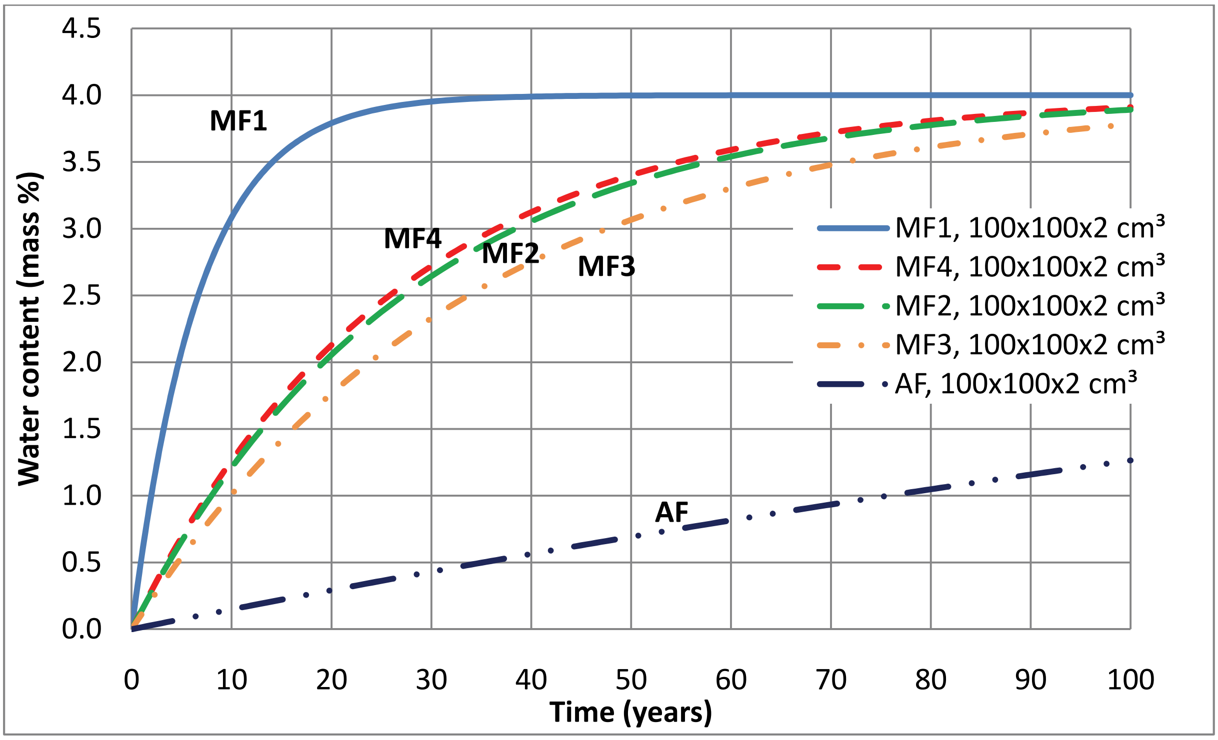


**Fig. 12. Water content for various laminate types. It is assumed laminate properties remain the same during the entire period. No getters and dessicants have been taken into account.**

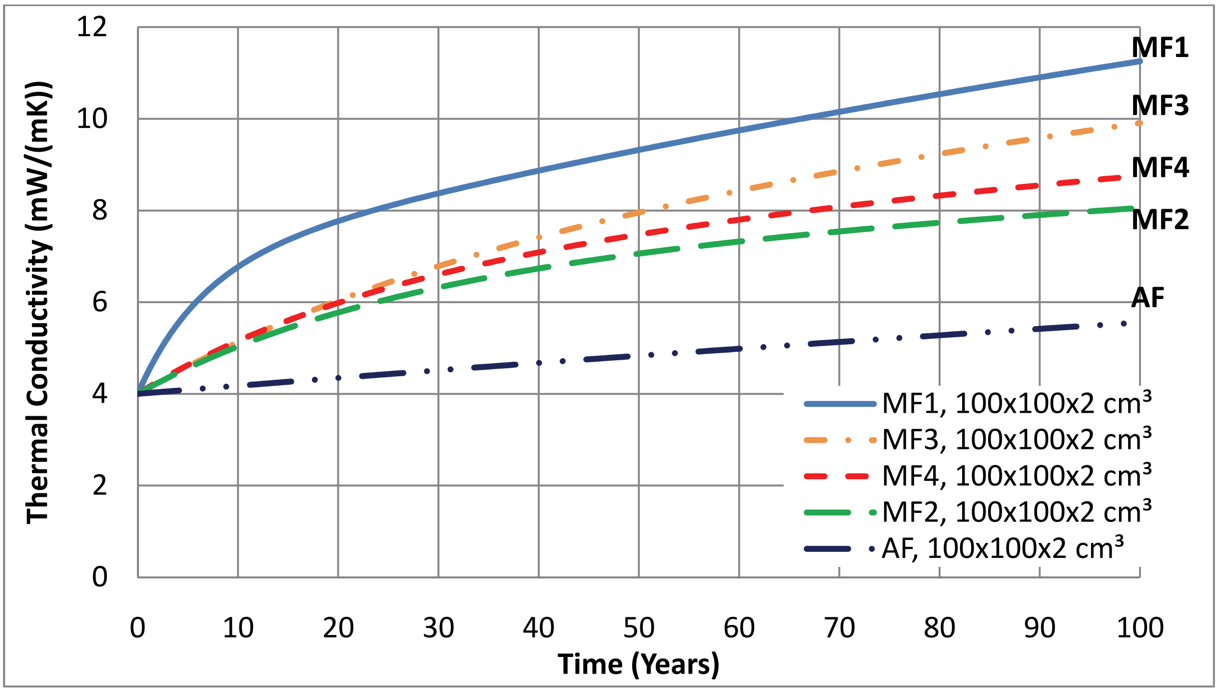


**Fig. 13. Total thermal conductivity for various laminate types. The inner air pressure is assumed to be zero at $t_0$=0. It is assumed that laminate properties remains the same during the entire period. No getters and desiccants have been taken into account.**

To evaluate the influence of panel size on thermal conductivity, the 100 year thermal conductivities of 100 cm x 100 cm x 2 cm panels is compared to those of 50 cm x 50 cm x 1 cm panels for VIPs with MF3 and MF4 laminates in Fig. 14.

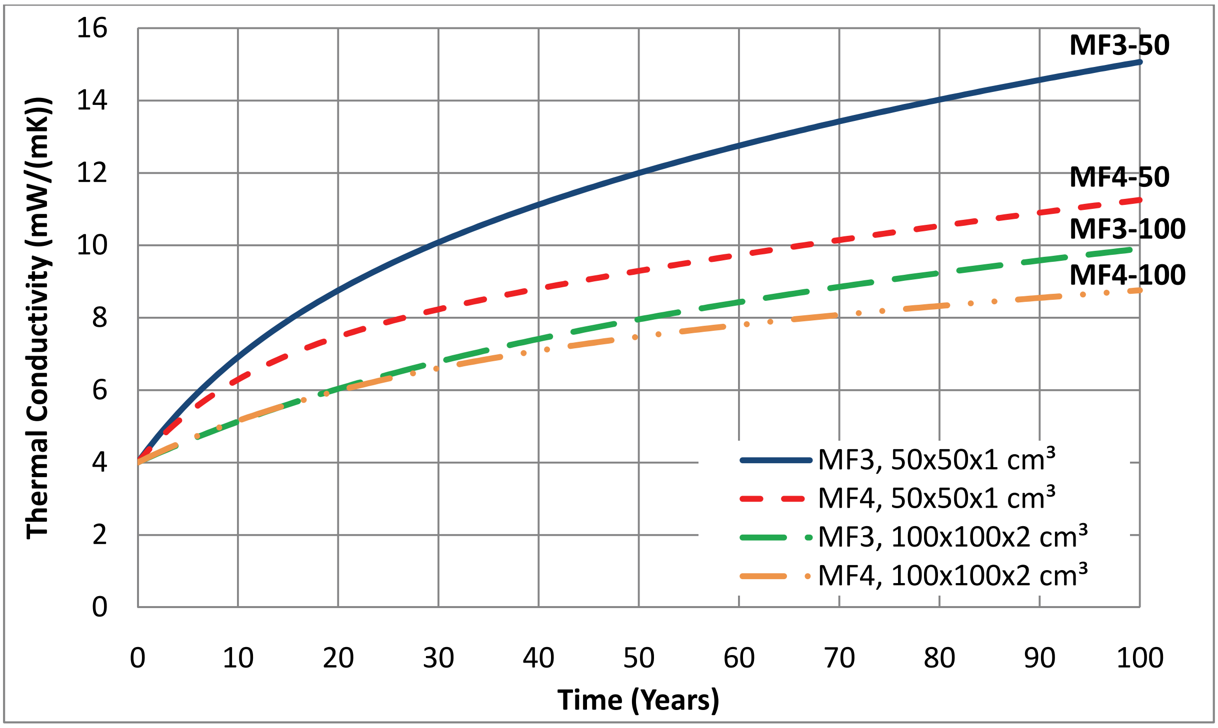


**Fig. 14 Total thermal conductivity for two panel sizes and two different barrier laminates. The inner air pressure is assumed to be zero at $t_0$=0. It is assumed that laminate properties remains the same during the entire period. No getters and desiccants have been taken into account.**

For these plots, constant climatic conditions during the entire period are used. In addition, the same conditions are used for both sides of the VIP. For VIPs in actual building applications the climatic conditions can vary greatly between outer and inner surface.

To evaluate how varying climate might affect service life of VIPs Baetens et al. (2010b) applied a dynamic model for simulation. Results from the dynamic simulations proved to be somewhat similar to those of the steady state predictions above. However, since 23°C and 50% RH as used in the static simulation represents a high average temperature and moisture content, the dynamic simulation showed a somewhat slower gas pressure increase and lower moisture content.

Dynamic simulations for the climate of several European locations showed that the deviations across the various locations were quite small. For 100 year simulations the center of panel thermal conductivity for VIPs with MF1 laminates were found to be 14.7 ± 0.7 mW/(mK) for 50 cm x 50 cm x 1 cm panels and 10.3 ± 0.4 for the 100 cm x 100 cm x 2 cm panels (Baetens et al. 2010b)

# 8 Accelerated Ageing

VIP properties change over time, most notably as air gases and water vapour permeate through the envelope barrier. To be able to evaluate the long term service life of VIPs, and to study the performance of VIPs over time within a limited time-frame, accelerated ageing is necessary. As of today no standardized method exists for the accelerated ageing of VIPs. However, the theoretical relationships presented in previous chapters can be used as a basis for designing accelerated ageing experiments.

The external climate factors that theoretically contribute to the ageing of VIPs are temperature, moisture and pressure. In addition, several other elements such as pollutants or acidity in surroundings might give a physical degradation of either envelope or core material, but that is not within the scope of this study.

There is a complex relationship between external factors and pressure increase in the VIPs. For the sake of simplicity the different factors will be treated separately, but it is important to remember that in a real-life situation it is difficult to separate the effect of each single factor

## 8.1 Temperature

Generally, temperature effects on gas and water vapour diffusion can be assumed to follow an Arrhenius relation (Schwab et al. 2005b):

$$Q(T) = Q(T_0)\,\mathrm{e}^{\frac{E_a}{R}\left(\frac{1}{T_0} - \frac{1}{T}\right)} \tag{27}$$

where

Q = Permeance of envelope ($cm^3$/ ($m^2$s Pa))

$E_a$ = Activation energy (J/mol)

R = Gas Constant 8.31 (J/K mol)

This relationship for air gases was confirmed by Schwab et al. (2005b). Results from this study are summarized in Table 2 below.

**Table 2. Factor exp($-E_a/(RT) + E_a/(RT_0)$) for different laminatess (AF, MF1 and MF2) and increasing temperature. $T_0$=25$^0$C (Reproduced from Schwab et al. 2005b)**

| Temperature (°C) | Laminate AF | Laminate MF1 | Laminate MF2 |
|---|---|---|---|
| 0 | 0.39 | 0.23 | 0.35 |
| 10 | 0.58 | 0.43 | 0.47 |
| 25 | 1.0 | 1.0 | 1.0 |
| 45 | 1.9 | 2.7 | 2.0 |
| 65 | 3.4 | 6.7 | 3.8 |
| 80 | 4.9 | 12.2 | 5.8 |

For water vapor permeance, the temperature dependence is more complex because of the interaction between temperature, water vapour pressure and relative humidity. Ambient moisture is a very important factor as will be seen below, but tests performed by Schwab et al. (2005b) suggests that a temperature dependence can also be found. The exception is for aluminum-coated laminates (AF), were no temperature effect could be detected. One possible reason for this is that the activation energy for PET for water vapour is quite low, rendering the temperature influence negligible (Schwab et al. 2005b). In addition, the complete process of water vapour diffusion through VIP laminates is not sufficiently known to estimate the temperature dependence exactly.

Simmler and Brunner (2005b) suggest the use of a parameterized Arrhenius function to account for the combined effect of moisture and temperature.

## 8.2 Moisture

From Eq. (13) it can be concluded that the ambient water vapour pressure is important for the moisture increase of the VIP. The saturation vapour pressure shows an almost exponential dependence on temperature, according to (Heinemann 2008):

$$p_{sat}(T) = 611 e^{\left(17.08 \frac{T-273.15}{T-39}\right)} \qquad (28)$$

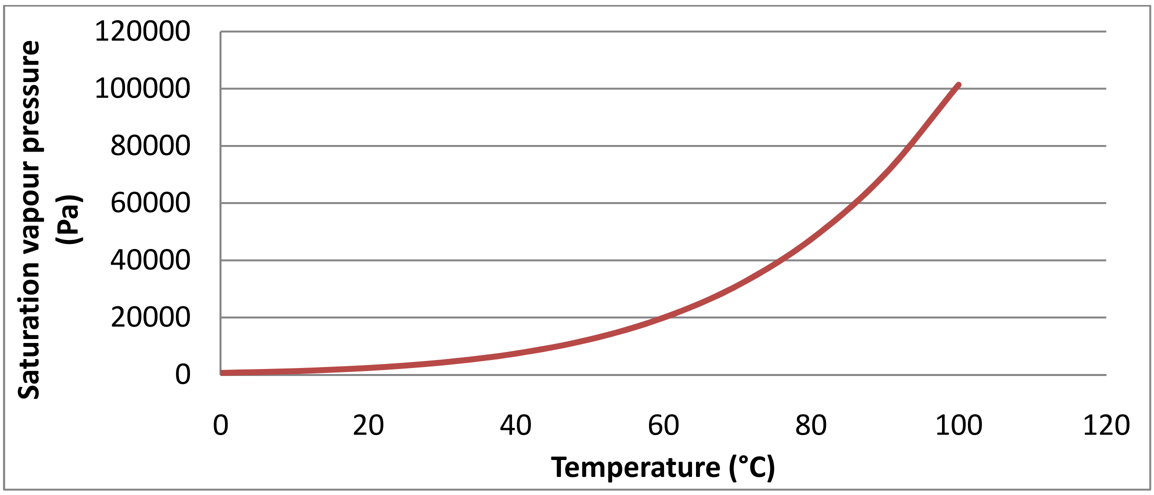


**Fig. 15 Saturation water vapour pressure for increasing temperatures. Based on Eq. 28.**

Based on this, it could be assumed that high temperature in combination with high RH would greatly accelerate the ageing effects on a VIP. This will increase the water vapour pressure difference across the envelope, and thus the driving pressure. In addition, it could be assumed that the high temperature will increase the WVTR somewhat, according to the Arrhenius relation in Eq.(27), as discussed above.

## 8.3 Pressure

Pressure is a factor in all formulas used for calculating the increase in thermal conductivity of VIPs, either directly through the external atmospheric pressure of the VIPs or indirectly through the saturation water vapour pressure. Based on this it is natural to assume that increased external pressure might give a substantial accelerating effect for the ageing of VIPs.

To evaluate the acceleration effect of increase pressure, plots are made for the increase of water content, gas pressure and thermal conductivity of VIPs over time for increasing external pressure. For these curves it is assumed that the panels are pressurized using air with constant temperature and water vapour content, leading to a constant RH for increasing pressures, but an increasing water vapour pressure. It is assumed that the relationship in Eq. 29 holds for pressures in the range used for these curves.

$$\phi_2 = \phi_1 \frac{p_{sat1} P_{a2}}{p_{sat2} P_{a1}} \tag{29}$$

Where

$\phi_1, \phi_2$ = Relative humidity for state 1 and state 2 respectively

$p_{sat1}, p_{sat2}$ = Saturation water vapour pressure for state 1 and state 2 respectively

$P_{a1}, P_{a2}$ = Pressure in state 1 and state 2 respectively

As the air/water vapour mixture is pressurized, the number of molecules will increase and the water molecules will possibly be pressed together. The dipole-binding of the $H_2O$ molecule might affect the attraction between water molecules. This compression of water molecules might lead to changes in the saturation water vapour pressure. However, this is not studied more extensively in this work. For the plots in Fig. 17 and Fig. 18 the saturation water vapour pressure is assumed to be proportional to the total pressure for constant temperature.

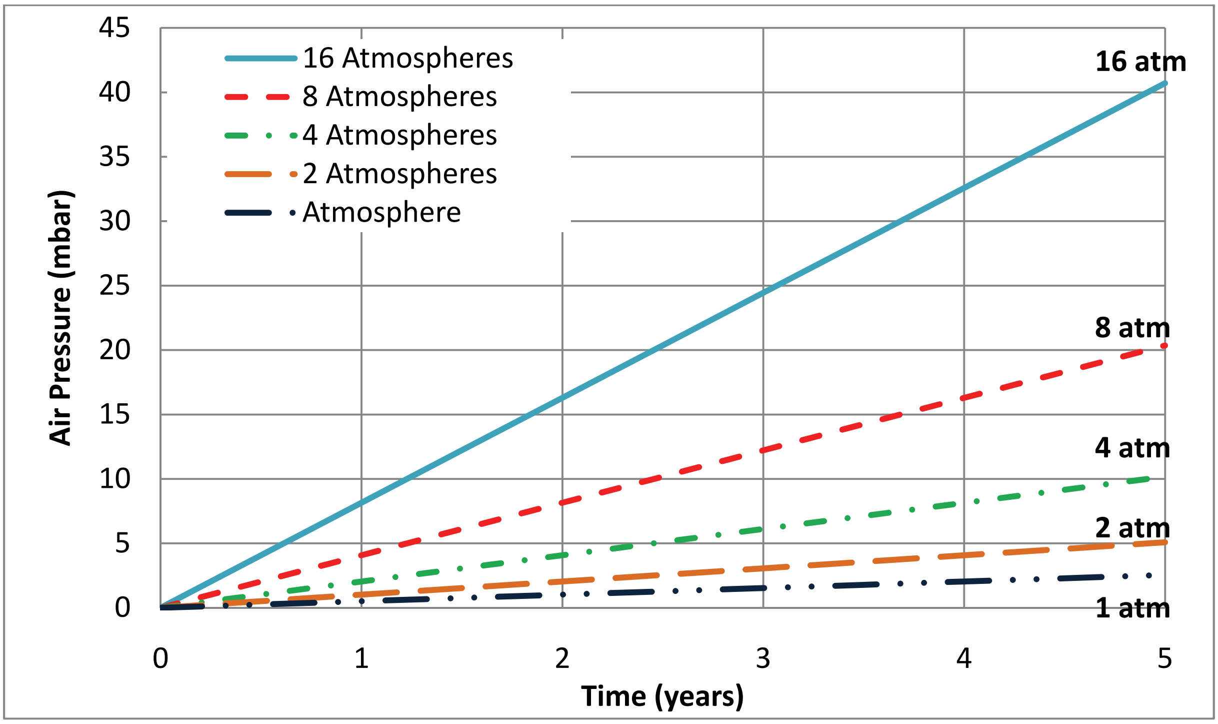


**Fig. 16** Internal pressure as a function of time and external pressure. Values for MF4 panels have been used to calculate pressure increase. Panel size is set as 100 cm x 100 cm x 2 cm.

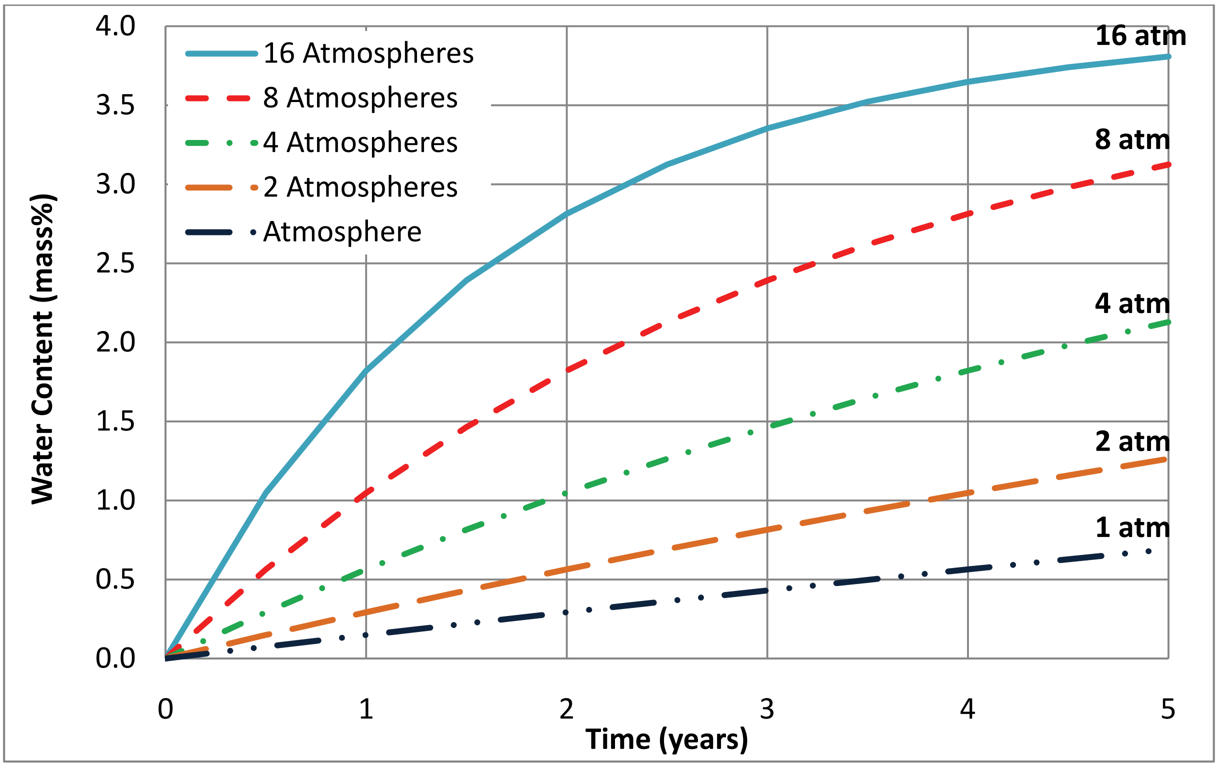


**Fig. 17** Water content as a function of time and external pressure. Values for MF4 panels have been used to calculate the water content. Panel size is set as 100 cm x 100 cm x 2 cm.

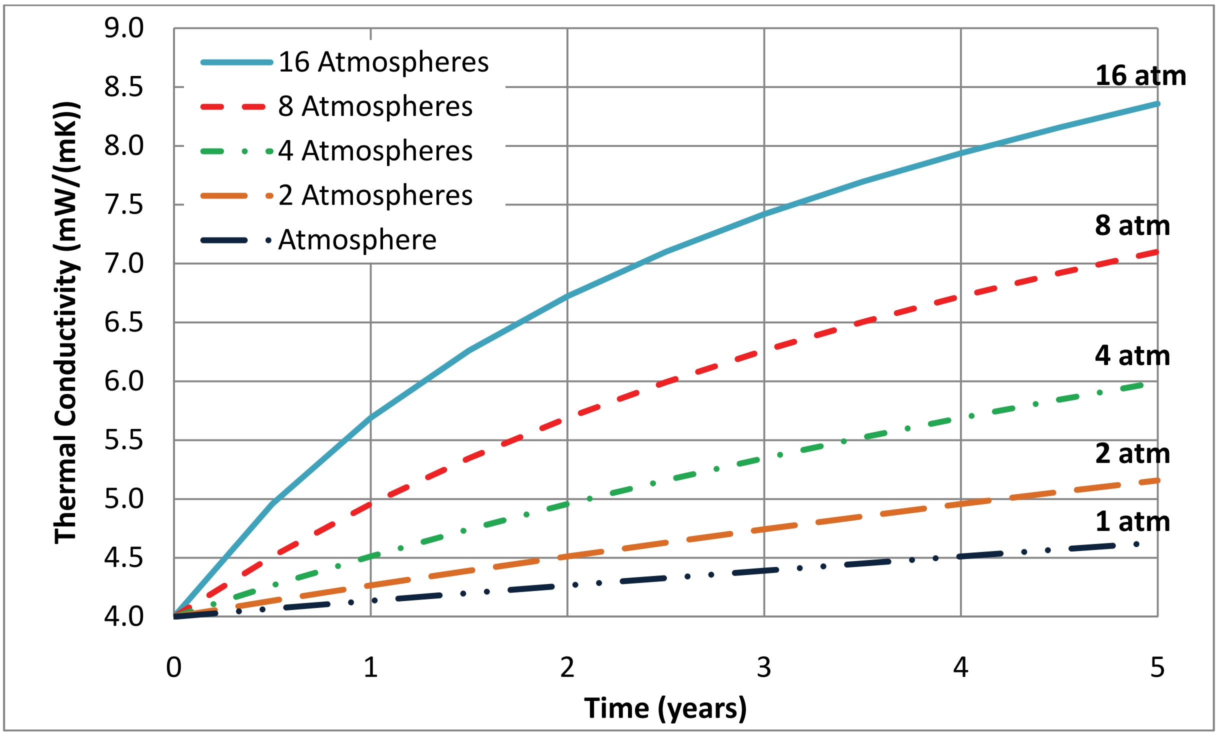


**Fig. 18 Thermal conductivity as a function of time and external pressure. Values for MF4 panels have been used to calculate the resulting thermal conductivity. Panel size is set as 100 cm x 100 cm x 2 cm.**

As increases in internal air pressure, water vapour pressure and water content are all accelerated by increased pressure, the total acceleration effect of panel ageing can be quite large, as can be seen from Fig. 18 above. The actual acceleration effect of increased pressure can be seen from Fig. 19 where the ageing time is plotted versus the accelerated age of the VIP.

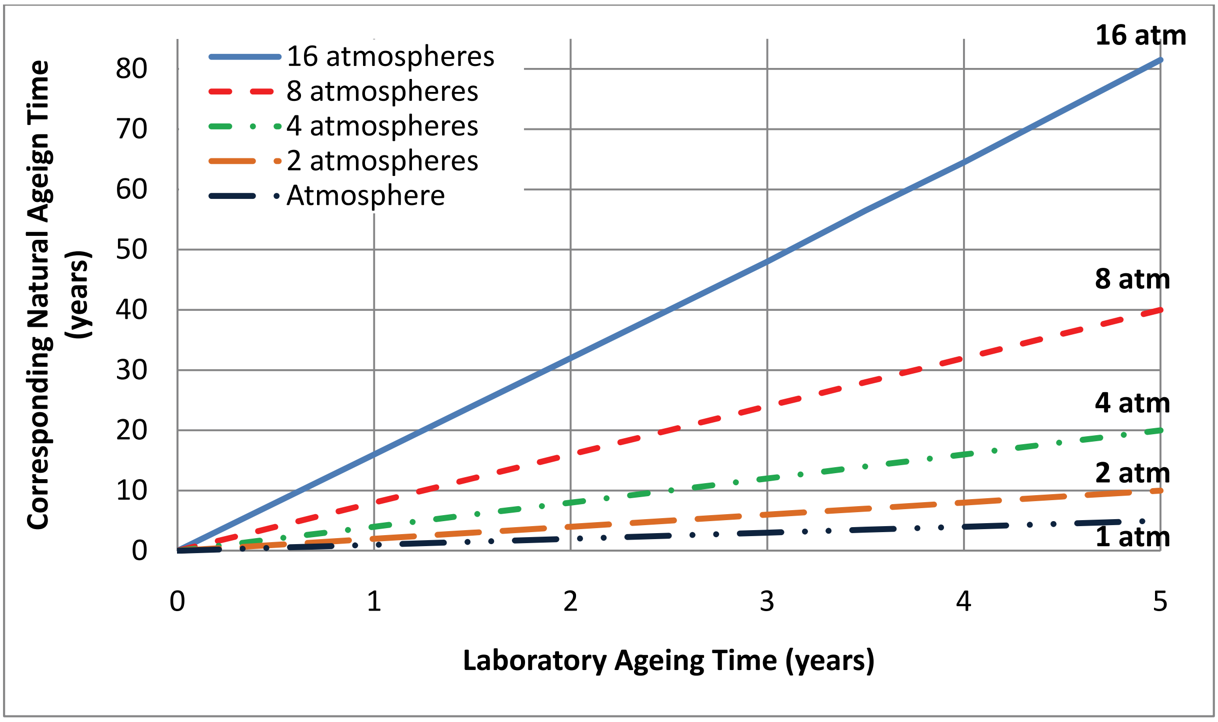


**Fig. 19 Acceleration effect of increased pressure, plotted for ageing times up to 5 years. Panel size is set as 100 cm x 100 cm x 2 cm.**

The natural ageing time is found by comparing the calculated values for thermal conductivity for each elevated pressure with the thermal conductivity for atmospheric pressure, based on values found from Eq. (23) and Fig. 13.

Based on these results it can be concluded that increasing external pressure is a valid acceleration method, at least theoretically. It can also be concluded that the higher the external pressure, the higher the acceleration factor. One issue in pressure ageing of VIPs is which pressures the VIPs can withstand without changes to the physical properties of the core or the panel.

# 9 Ageing Experiments Performed on Vacuum Insulation Panels

To evaluate the actual ageing effects on the thermal conductivity of VIPs, different ageing experiments have been performed. These experiments are conducted both to verify the theoretical relationships presented in the previous chapters and to evaluate the resistance of VIPs to severe climatic strains.

## 9.1 Vacuum Insulation Panels Used in Experiments

The VIPs employed for the experiments presented in this thesis are of the type va-Q-vipB from the producer Va-Q-tec (2009). Va-Q-vipB consists of a core of amorphous silicon dioxide and an inorganic opacifier. The panel is sealed with a high barrier laminate which is again covered on the exterior with a black protection fleece. The high barrier laminate consists of three layers of metalized PET with PE as a sealing layer on the inside. This is equivalent to a MF4 barrier laminate. Total thickness of the laminate is approximately 100 µm. The VIPs have dimensions 100 cm x 60 cm x 2 cm.

## 9.2 Thermal Conductivity by Heat Flow Meter

To evaluate the change in thermal conductivity of VIPs, a Heat Flow Meter (HFM) has been used. The setup of this apparatus and factors that has to be considered will be discussed. All measurements are performed in accordance with current versions of ISO 8301 and NS-EN 12667.

### 9.2.1 Principle

The principle with the HFM apparatus is to establish a unidirectional uniform density of heat flow through the specimen. By measuring the heat flow through the specimen, the density of heat flow rate (q) can be calculated when the metering area (A) and temperature difference (ΔT) is known. The temperature difference is kept constant with heating and cooling plates at opposite sides of the specimen, and temperature sensors on both sides of the specimen.

The thermal resistance (R) is calculated from the knowledge of q, A and ΔT. When the thickness of the specimen is known, the thermal conductivity ($\lambda$) can be calculated.

The apparatus is calibrated using a standard specimen of known thermal resistance.

### 9.2.2 Apparatus and Setup

Several different configurations of heat flow meters are possible. The one used in these tests is a single-specimen symmetrical configuration according to ISO 8301. This apparatus consists of heating and cooling plates above and below the specimen respectively, as well as heat flow meters on both sides of the specimen. This kind of apparatus is commonly called a hot-plate apparatus. The setup is shown in Fig. 20.

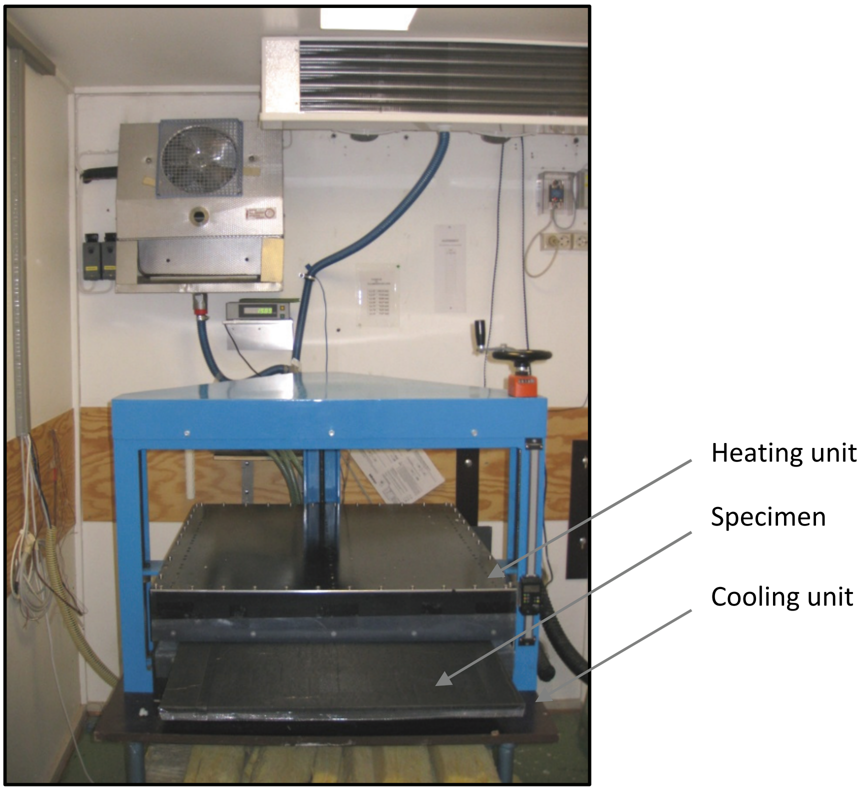


**Fig. 20 Heat Flow Meter Apparatus setup for measurement of thermal conductivity.**

In this specific apparatus the cooling plates are 600 mm x 600 mm and the heat flow meters are 300 mm x 300 mm. The heat flow is vertically downwards.

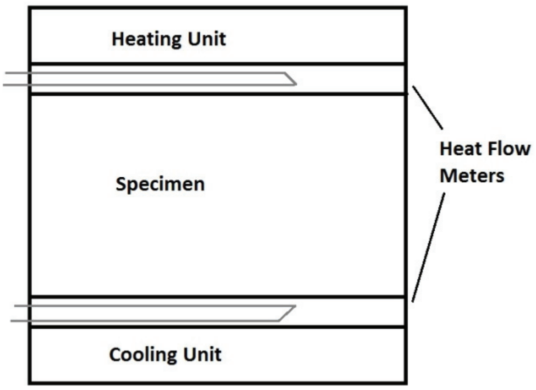


**Fig. 21 Schematical layout of heat flow meter apparatus.**

### 9.2.3 Sources of Error

Special attention must be given to two particular factors when measuring VIPs in a HFM apparatus:

- Possible heat loss from edges of the specimen
- Homogeneity of the specimen

The possibility of heat loss from edges of the specimen becomes especially valid as the measured VIPs (60 cm x 100 cm x 2 cm) are larger than the heating and cooling units of the apparatus. This can be seen from Fig. 20, as the panel is visible outside the apparatus. Tests were performed to evaluate this heat loss, and the possible effect it could have on the measured thermal resistance. It was found, however, that this had little effect on the results of the measurements, and no extra insulation or other measures were considered necessary.

As regards homogeneity, problems especially arise concerning the VIP envelope barrier. The core material is known to be porous and homogenous, but the metal layers in the envelope barrier might transfer some heat along the VIP edges. The thermal bridge of the VIP envelope barrier has been studied extensively in the literature, and it can possible give a great contribution to heat transfer were VIPs are used (Ghazi-Wakili et al. 2004). When thermal conductivity was determined by heat flow meter, however, no substantial effect of the envelope materials was found. Despite this, care should be taken when measurements are performed on smaller VIPs. Then the panel edges will be closer to the heat flow meters and can possibly contribute to the total measured heat flow. For the larger panels tested here, however, no problems arise as the area of the heat flow meters used are 300 x 300 $mm^2$, and only the center of panel thermal conductivity is measured.

The uncertainty in the measurements is estimated to be ± 1.9% with 95% confidence interval for all performed tests. This is based on the uncertainty that is estimated for the apparatus during calibration according to ISO 8301:1991.

## 9.3 Temperature Ageing According to CUAP 12.01/30

One method for testing ageing effects on VIPs is suggested in CUAP 12.01/30. The test is based on severe temperature conditions over an extended period of time. The ageing is supposed to cover a time span of 25 years.

### 9.3.1 Scope

The main scope of the experiment is to verify whether an ageing of 25 years can be achieved by application of this procedure. The procedure has been altered somewhat, to accommodate more measurements than originally specified.

### 9.3.2 Procedure

1. Conditioning at (23 ± 2)°C and (50 ± 5)% RH for at least 72 hours.
2. Determination of initial thermal conductivity
3. Cycling in alternating climate (8 cycles), where one cycle consists of:
    a. 8 hours at (80 ± 3)°C
    b. 16 hours at (-15 ± 3)°C
4. Determination of the thermal conductivity
5. Temperature ageing for 90 days at (80 ± 3)°C
6. Determination of the thermal conductivity
7. Temperature ageing continued for another 90 days at (80 ± 3)°C
8. Final determination of the thermal conductivity

Additional measurements of thermal conductivity were conducted when considered necessary. Alternating climate was achieved by manually transferring the VIP between a heating cabinet and a freezer at the end of each period. The temperature ageing were conducted in a heating cabinet without humidifier, and the ambient moisture content can thus be considered negligible.

## 9.4 Cyclic Climate Ageing According to NT Build 495

The Nordtest Method NT Build 495 is a test method exposing materials in the vertical position to accelerated climate strains.

### 9.4.1 Scope

The scope of this experiment is to evaluate the resistance of VIPs to varying climate strains. This involves the integrity of the panels in addition to the thermal properties. By using two samples, one exposed and one protected by a timber-frame, the durability and robustness of exposed VIPs can be evaluated and compared to that of protected VIPs. The testing of the exposed VIP would especially be interesting for storage and handling of VIPs during the construction phase.

### 9.4.2 Experimental Setup

The test rig consists of the following successive climate strains:

1. UV-radiation (UVA = 33 $W/m^2$, UVB = 2.4 $W/m^2$) and IR-radiation giving a black panel temperature of (63 ± 5)°C
2. Wetting with a spray of water
3. Freezing at -20 ± 5°C
4. Thawing at laboratory climate

The time interval in each of the positions is one hour. The setup of the test rig is shown in Fig. 22.

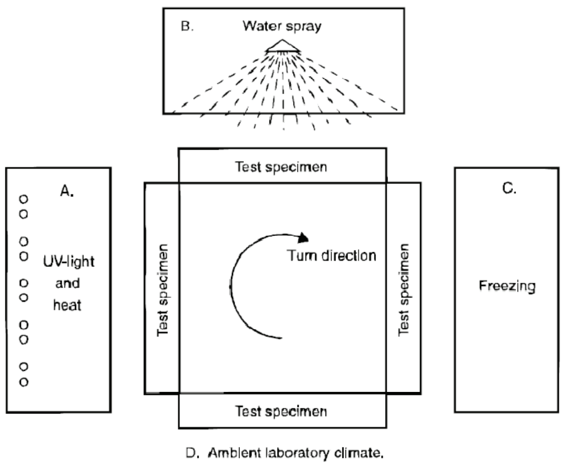


**Fig. 22 Test rig for accelerated climate exposure according to NT Build 495 (2000).**

### 9.4.3 Test Specimen

The test consists of two different specimens. One is a VIP that is directly exposed to the climatic strains. The other specimen is a VIP built into a ventilated timber frame wall. Wall construction details are shown in Fig. 23.

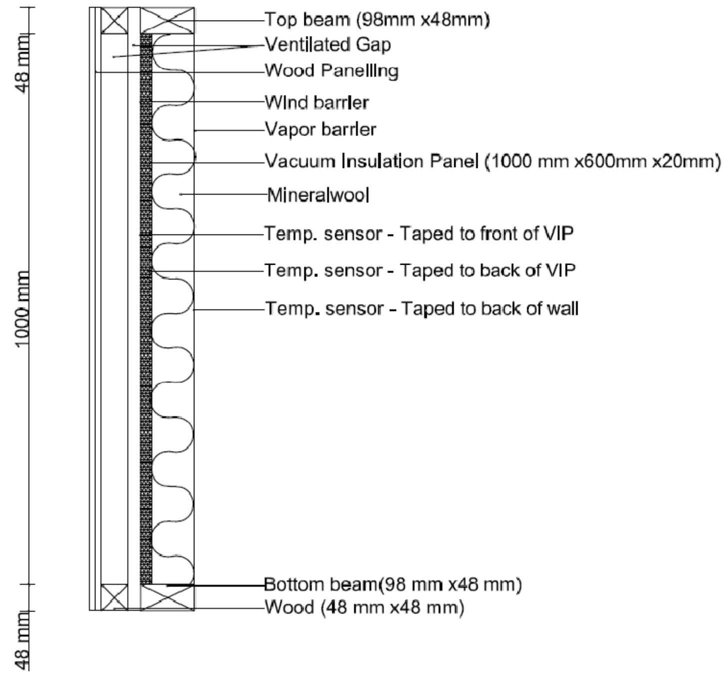


**Fig. 23 Construction detail for wall exposed to accelerated climate strains.**

For the wall construction, special interest is taken in the temperature conditions on both sides of the VIP while exposed to cooling/freezing. Temperature sensors were therefore placed on both sides of the panel, and on the exterior of the wall to be able to study these conditions.

## 9.5 Moisture and Temperature Ageing

To evaluate the effect of severe hygrothermal conditions on VIPs, a test is designed to expose a VIP to high temperature in combination with high moisture pressure.

### 9.5.1 Scope

The scope of the experiment is to evaluate which ageing effect that can be achieved by exposing a VIP to high relative humidity and high temperature simultaneously. Since saturation vapour pressure show an exponential increase with temperature, a very high moisture pressure is achievable when the temperature is increased.

### 9.5.2 Experimental Setup

In this preliminary experiment it is desired to maximize the moisture pressure within the specified temperature limits for the VIP. To facilitate this, the VIP is placed in a sealed envelope together with a container of water. The whole envelope is then placed in a heating cabinet at 70°C.

The following procedure has been employed in the testing:

1. Conditioning at (23 ± 2)°C and (50 ± 5)% RH for at least 72 hours.
2. Determination of initial thermal conductivity
3. Storage in heating (with water container) cabinet for 30 days at 70°C
4. Determination of thermal conductivity
5. Storage in heating cabinet (with water container) for 30 days at 70°C
6. Determination of thermal conductivity

7. Storage in heating cabinet (with water container) for 30 days at 70°C
8. Final determination of thermal conductivity

## 9.6 Pressure Ageing

As has been showed, the pressure gradient across the VIP envelope is a component in the formulas for both gas and moisture transport into the VIP. By increasing the external pressure, it can therefore be predicted that the transport will increase proportionally with the pressure increase.

Because of limitations on testing equipment, smaller VIPs were employed for the pressure tests than for the other ageing experiments. The panels used for pressure ageing were 20 cm x 12 cm x 2 cm. These panels were also provided by the producer va-Q-tec, and were of the type va-Q-vip. These VIPs did not have the black fire protection fleece found on the larger panels.

### 9.6.1 Scope

The scope of the experiment is to test whether these relationships hold for actual accelerated ageing by exposing VIP samples to high pressures in a pressure tank. The procedure and experimental setup is described below.

### 9.6.2 Testing Procedure

A new procedure was developed for the pressure testing of VIPs. Initially it was vital to assess the physical changes on VIP samples exposed to high pressures. To evaluate this, a VIP panel was exposed to increasing pressure, while the panel thickness was measured at intervals. The results from this test can be seen in Fig. 24.

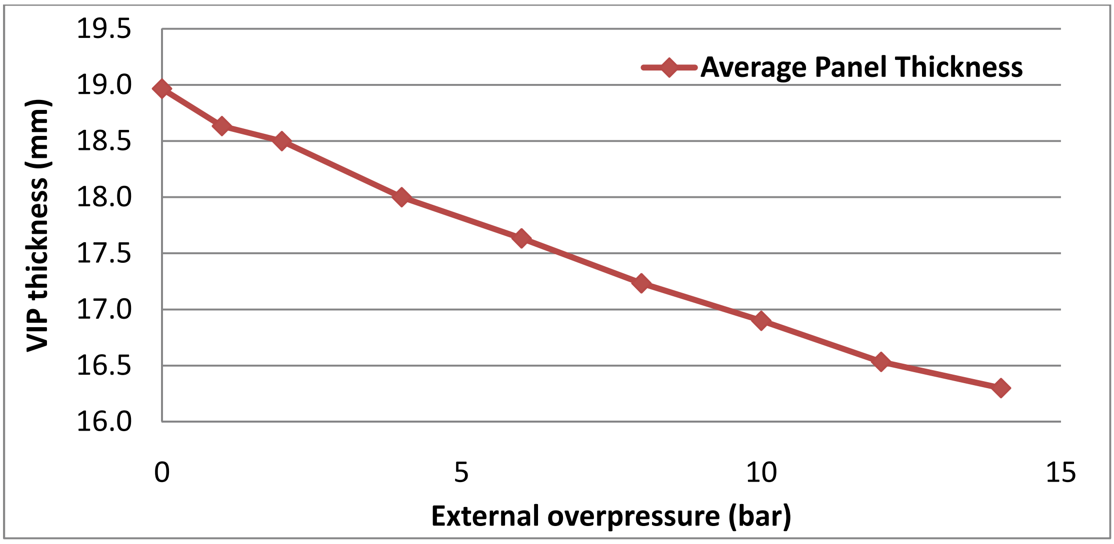


**Fig. 24 Average VIP thickness for increasing air overpressure.**

As can be seen, the increased pressure lead to a permanent deformation of the VIP, and the panel shrank approximately 15% when pressurized with 14 bar overpressure. It is natural to assume that this is an effect from the deformation of the core material. Since this might have a significant effect on the thermal conductivity it became vital for the further pressure test to separate the effects of potential increased air permeation into the VIP from those of changed physical properties of the VIPs or the core material.

The following procedure was employed for testing:

1. Determination of initial thermal conductivity[2]
2. Pressurizing to 8 bar overpressure using pressurized air
    a. 1 panel tested for new thermal conductivity
    b. 2 panels stored for 30 days at 8 bar before determination of new thermal conductivity
3. Comparison between panel pressurized to 8 bar and not stored, and those stored for 30 days to evaluate any relative change of thermal conductivity.

Any significant difference in the relative change of thermal conductivity between the panels stored for 30 days at high pressure and the one not stored would signify some change to the thermal conductivity other than what is caused instantly by the increased pressure. The suitability of this testing method is discussed below.

### 9.6.3 Experimental Setup

For the pressurizing of the VIPs, a pressure tank with an external gas tank was employed. This is shown in Fig. 25. Regular pressurized, dry air was used to increase the pressure in the tank. The maximum pressure capacity for the equipment used was 14 bar for short term exposure and 8 bar for long term exposure.

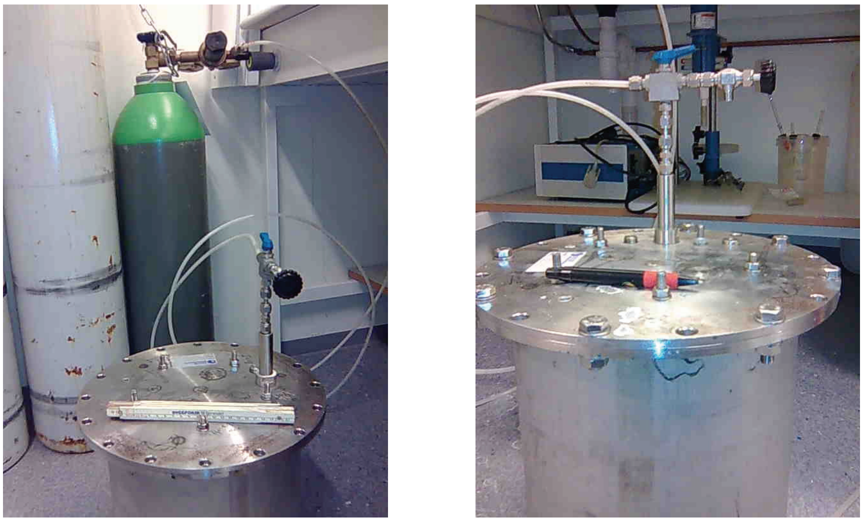

**Fig. 25 Experimental setup for pressure experiments on VIPs.**

[2] Since the VIPs employed for pressure testing were smaller than the panels used for the other experiments, the determination of thermal conductivity were more complicated. A discussion on the thermal conductivity testing procedure can be found in Appendix A.

## 10 Results from Ageing Experiments

When evaluating the ageing of VIPs for various procedures, thermal conductivity was used as a measure for the performance. In addition, any physical changes on the VIPs were registered as they might be interesting for VIP in building applications.

### 10.1 Temperature Ageing According to CUAP 12.01/30

The initial thermal conductivity was measured to be 4.6 ± 0.1 mW/(mK). The panel was then subjected to the ageing procedure as presented in part 9.3.

After the freeze/thaw cycles the outer fleece began to fray at the edges. No change of thermal conductivity was observed at this time.

After less than a week in the heating cabinet at 80°C the outer fleece layer began to lift from the VIP envelope. Large areas of the fleece had loosened from the substrate creating blisters of various shapes and sizes. This effect became more pronounced until approximatelt one month into the experiment. No further changes were observed after this time. Figures Fig. 26 and Fig. 27 show the VIP after 1 week and 1 month respectively, visualizing the change on the fleece layer. No further changes were observed during the rest of the ageing period.

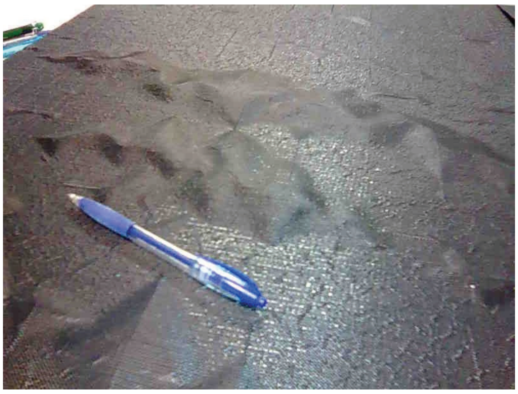

**Fig. 26 Visible delamination of the outer fleece layer of the VIP envelope after exposure at 80°C for 7 days.**

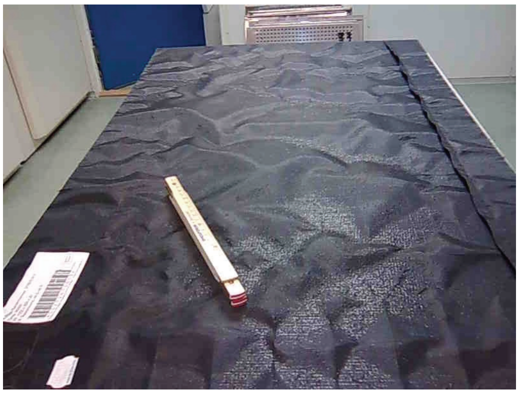

**Fig. 27 Visible delamination of the fleece cover after exposure at 80°C for approximately 1 month. More of the envelope cover has lifed from the substrate than after 7 days. No further changes were observed during the rest of the ageing period.**

Measurements showed that this delamination had no effect on thermal conductivity. It can thus be assumed that delamination was restricted to the outer fleece, as the gas-and vapour barrier remained intact.

When thermal conductivity was measured approximately 100 days into the procedure, it was found that the panel had swelled somewhat. As a result the thickness of the sample was higher than it was when initially tested. The initial thickness used for thermal conductivity measurements were 19.9 mm, while the new thickness after 100 days was 21.0 mm. This increased thickness leads to a slightly higher thermal conductivity than would otherwise be found. The thermal resistance is retained, however, as the increased thickness offsets the increased thermal conductivity.

## 10.2 Cyclic Climate Ageing According to NT Build 495

The initial thermal conductivity was measured to 4.3 ± 0.1 mW/(mK). The panel was then subjected to the ageing procedure as presented in part 9.4. The configuration in the climate simulator can be seen in Fig. 28.


**Fig. 28 Wall section of the climate simulator showing both VIPs. The exposed VIP can be seen in the top right corner, while the protected VIP is behind the weatherboards.**

After less than a day in the climate simulator, the outer fleece layer on the exposed panel began blistering, similar to the thermally aged panel. However, the delamination did not continue, and only small areas blistered. Another pronounced physical effect on the exposed VIP in the climate simulator was that it curved permanently towards the exposed side. The curvature of the panel is visualized in Fig. 29.

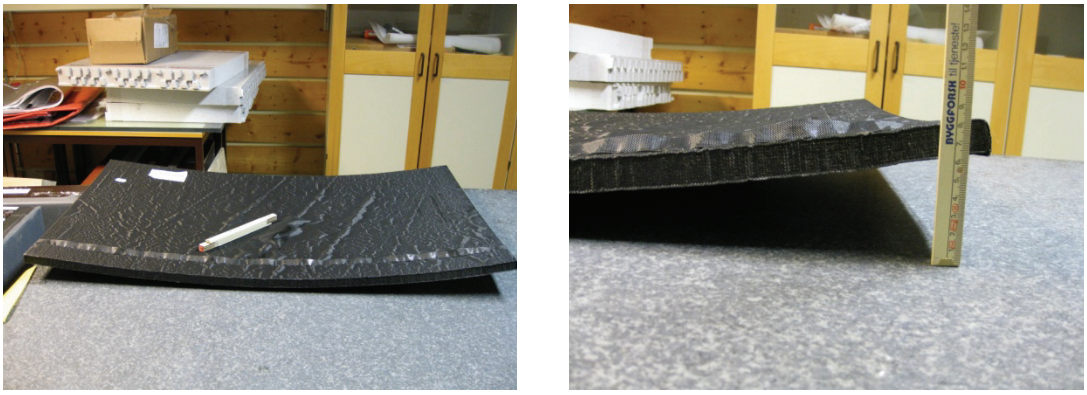

Fig. 29 Exposed panel after exposure to cyclic climate strains in vertical climate simulator for approximately one month. Some delamination of the fleece cover is visible. The panel had curved during exposure. No further changes were observed during the rest of the ageing period.

## 10.3 Moisture and Temperature Ageing

The initial thermal conductivity was measured to 4.4 ± 0.1 mW/(mK). The panel was then subjected to the ageing procedure as presented in part 9.5. When the VIP was tested after 60 days of ageing, the thermal conductivity had increased drastically to 17.9 mW/(mK). This might be best explained by failure of the VIP due to some external factor, such as mechanical damage. The experiment was discontinued. Thermal conductivity measurements are summarized in Fig. 30.

## 10.4 Thermal Conductivity Measurements

To evaluate the relative ageing effect the results from the thermal conductivity measurements for all experiments, except for the pressure experiment, are shown in Fig. 30. The thermal resistance is provided in Fig. 31.

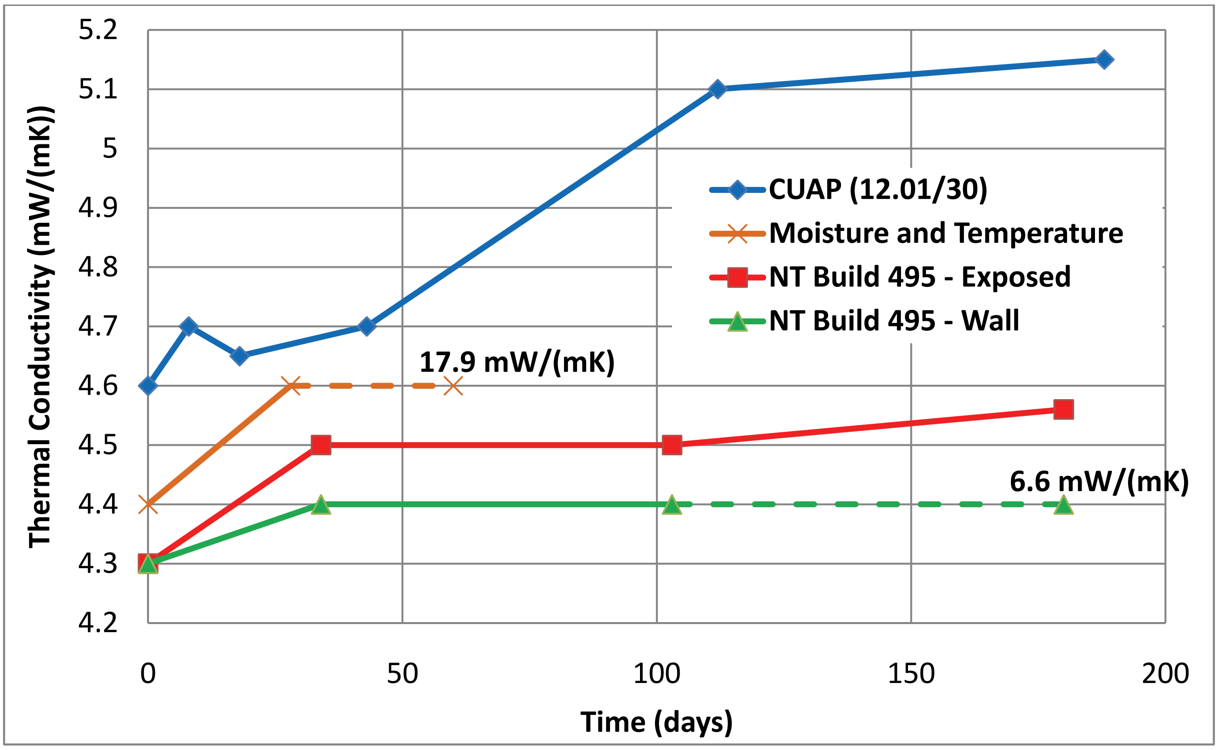


Fig. 30 Thermal Conductivity of VIPs exposed to various ageing experiments. The time periods for total exposure vary somewhat depending on the method.

Note that the initial non-aged thermal conductivity of the VIPs vary by 0.3 mW/(mK) which is approximately 7% of the total conductivity. Due to the relatively low rise in thermal conductivity for the VIPs exposed to ageing procedures, the variation in initial thermal conductivity might have as large or larger impact on thermal performance as the ageing effects. This variation also shows the necessity of confirming results with more extensive testing on several VIP samples.

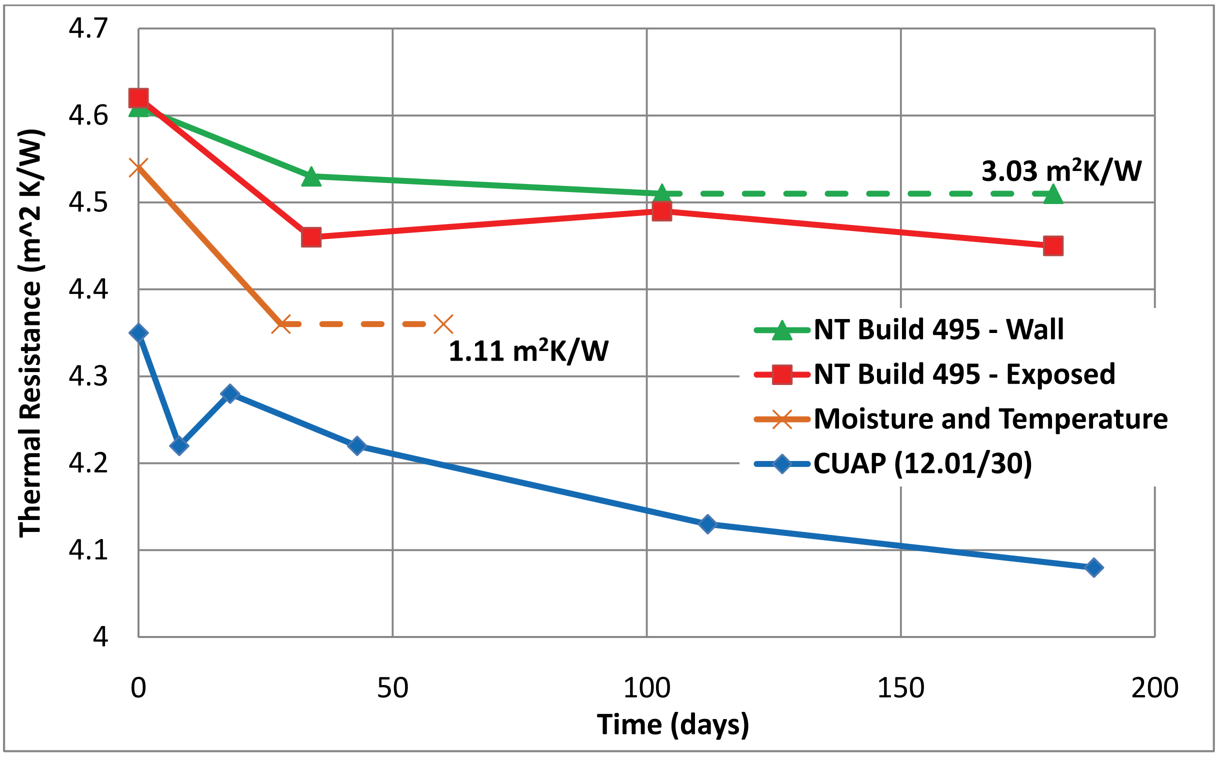


**Fig. 31 Thermal resistance of VIPs exposed to various ageing experiments. The time periods for total exposure vary somewhat depending on the method.**

For the thermally aged VIP (CUAP 12.01/30) it can be seen that the relative change in thermal conductivity is far higher than the change in thermal resistance. This is due to the increased thickness of the VIP that occurred as a result of swelling during the experiment. The insulating capacity is therefore best represented by the thermal resistance, as this value incorporates the geometrical changes of the VIP. For the moisture and temperature aged VIP and the protected VIP in the climate simulator, increases in thermal conductivity was higher than can be explained by ageing effects alone, and some failure must have occurred. This is marked with dotted lines for the relevant VIPs in the above figures.

## 10.5 Pressure Testing

As shown above, the pressurizing of VIPs led to a permanent compression of the panels. The panels shrank in all directions as can be seen from Fig. 32.

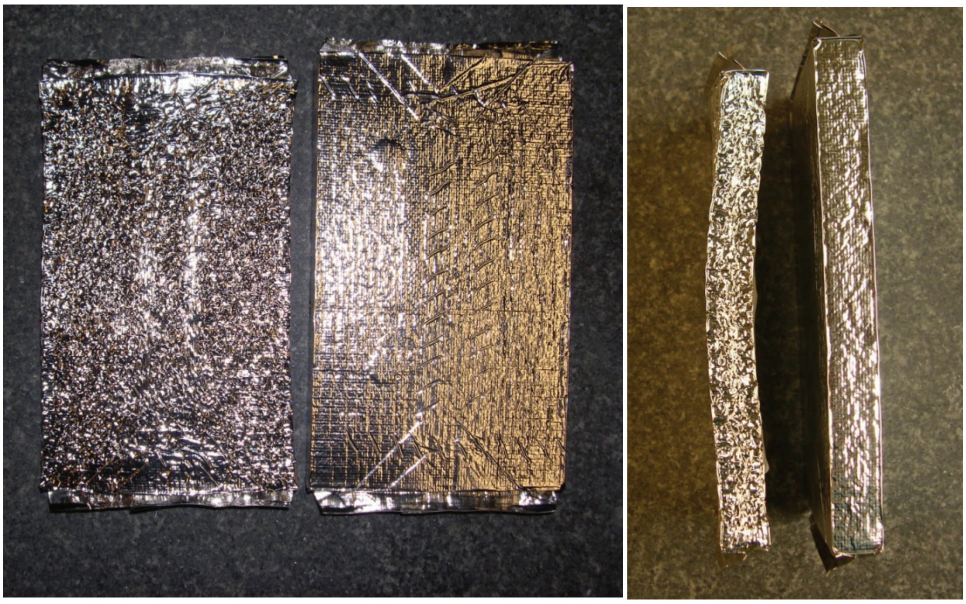

**Fig. 32 Visible compression of VIPs exposed to 14 bar overpressure. Pressurized VIP to the left, compared to a normal VIP on the right.**

Three different VIPs were subjected to pressure as an ageing condition. These panels are designated V2, V3 and V4, and were subjected to the following conditions:

V2: Pressurizing to 8 bar and then depressurized immediately
V3: Pressurizing to 8 bar in dry air and stored for 33 days
V4: Pressurizing to 8 bar in dry air and stored for 33 days

Both panel thickness and thermal conductivity was measured before and after the panels were subjected to elevated pressures. Results from these measurements are summarized in Table 3 and Fig. 33. The estimated error for the thermal conductivity measurements are 8%, based on comparison of measurements done on samples of known conductivity.

**Table 3 Results from exposure of VIPs to elevated pressures.**

| VIP Sample | V2 | V3 | V4 |
|---|---|---|---|
| Initial Thickness (mm) | 18.8 | 19.3 | 19.0 |
| Thickness after ageing (mm) | 16.9 | 16.6 | 16.1 |
| Percentage Difference | -9.9 % | -14.2 % | -15.3 % |
| | | | |
| Initial Thermal Conductivity (mW/(mK)) | 5.0 ± 0.4 | 5.3 ± 0.4 | 5.3 ± 0.4 |
| Thermal Conductivity after ageing (mW/(mK)) | 6.5 ± 0.5 | 8.9 ± 0.7 | 9.2 ± 0.7 |
| Percentage Difference | 30.0 % | 67.9 % | 73.6 % |
| | | | |
| Initial Thermal Resistance | 3.7 ± 0.3 | 3.6 ± 0.3 | 3.6 ± 0.3 |
| Thermal Resistance after ageing | 2.6 ± 0.2 | 1.8 ± 0.1 | 1.8 ± 0.1 |
| Percentage Difference | -29.5 % | -48.3 % | -50.8 % |

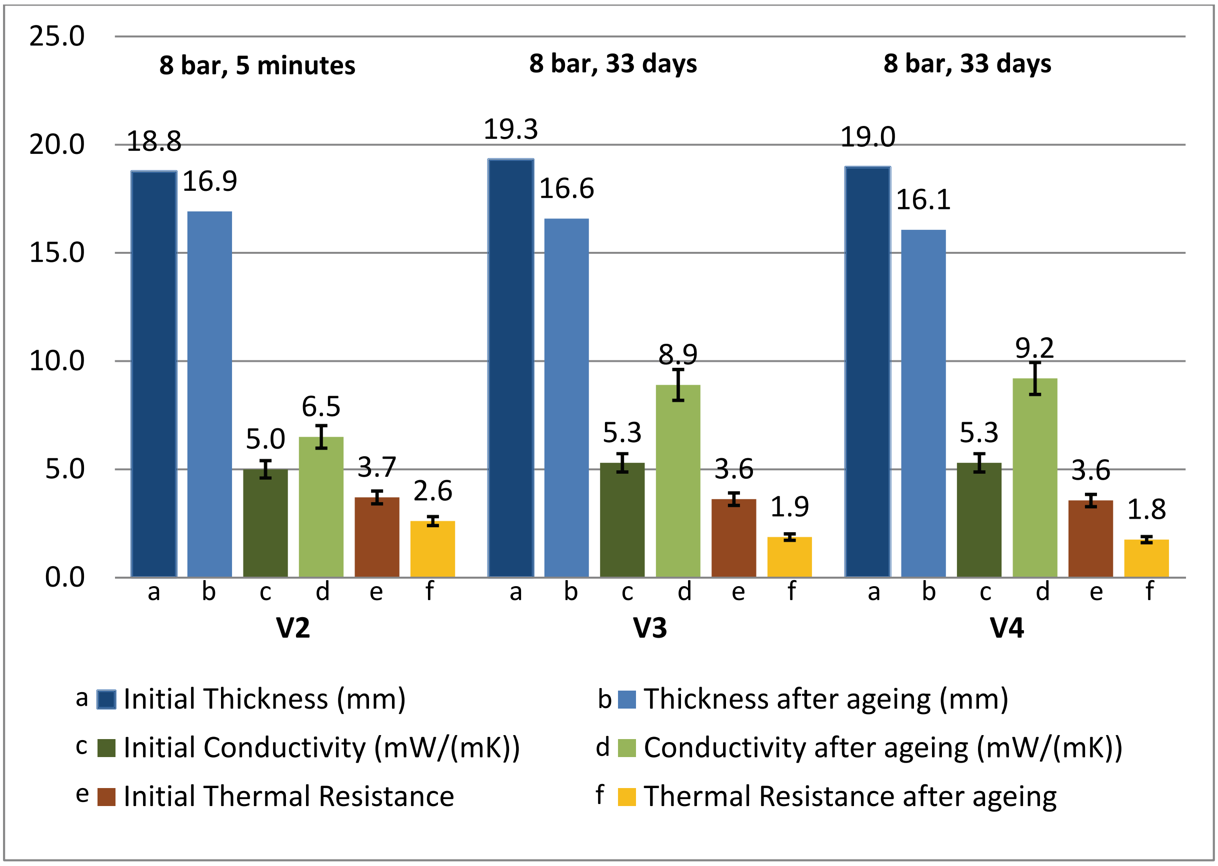


**Fig. 33 Thickness and thermal conductivity for VIPs exposed to elevated pressures (8 bar) , before and after exposure.**

As can be seen from Table 3 and Fig. 33 the thermal conductivity increase of VIPs V3 and V4 were significantly larger than the increase for the V2 VIP. The same effect can be seen on the thermal resistance of the VIPs. The permanent reductions of thickness for the three panels were similar in size, as all had a reduction of approximately 10-15%.

# 11 Discussion

## 11.1 Accelerated Ageing Tests

Accelerated ageing tests are laboratory methods used to evaluate the performance of a materials or a solutions over time. Various methods exist for the accelerated ageing of different materials. The obvious advantage of accelerated ageing tests is that they can be conducted within a limited timeframe. Hence, poor performance over time can be identified in the controlled environment of the laboratory, and costly damages and refurbishment of constructions in the future might be avoided.

Still, accelerated ageing has its limitations. The use of severe climate conditions such as extreme temperature variations, high relative humidity and exposure to UV-radiation, might lead to responses by the material that will not be caused in normal building application. This should be kept in mind when results from accelerated ageing tests are evaluated and used.

Despite this, accelerated ageing tests remain a useful and powerful tool in evaluating performance over time within a limited period, given that due care is offered to the limitations of the methods and their results.

## 11.2 Results from Ageing Tests

The total change of thermal conductivity for all VIPs subjected to ageing tests were relatively low, compared to the initial thermal performance. This is, however, in agreement with prediction curves for the ageing of VIPs under constant climatic conditions. For a VIP with a MF4 laminate and size 100 cm x 100 cm x 2 cm, the predicted thermal conductivity after 100 years is about 8.5 mW/(mK). This represents an increase of only 4.5 mW/(mK), or less than 0.05 mW/(mK) each year. Hence, a relatively low increase in thermal conductivity should be expected if the predictions are correct.

Of the several experiments conducted the CUAP experiments and the temperature and moisture experiments are the easiest to compare with the predictions, as the climate strain for these experiments are constant and uniform. In Fig. 34, the results from the CUAP experiment is compared to theoretical prediction curves. The prediction curves are based on the thermal conductivity model from Eq. (23). Properties for a MF4 VIP have been used. An Arrhenius factor of 5.8 has been applied, based on results for MF2 VIPs presented in Table 2. For the plotted measurement values for the CUAP VIP, the values have been normalized to the initial thickness of the VIP, to compensate for the increased thickness due to swelling.

As can be seen from Fig. 34, the measured values are comparable with what can be predicted using theoretical relationships. The predicted thermal conductivity is highly dependent upon the moisture content of the surrounding air. The moisture content in the heating cabinet where the VIP was stored is difficult to determine exactly, but is estimated at 2% RH. At as high temperatures as 80°C the sensitivity of the prediction models to increased RH becomes quite large. For increasing moisture contents in this temperature range, the prediction models might overestimate the thermal conductivity increase.

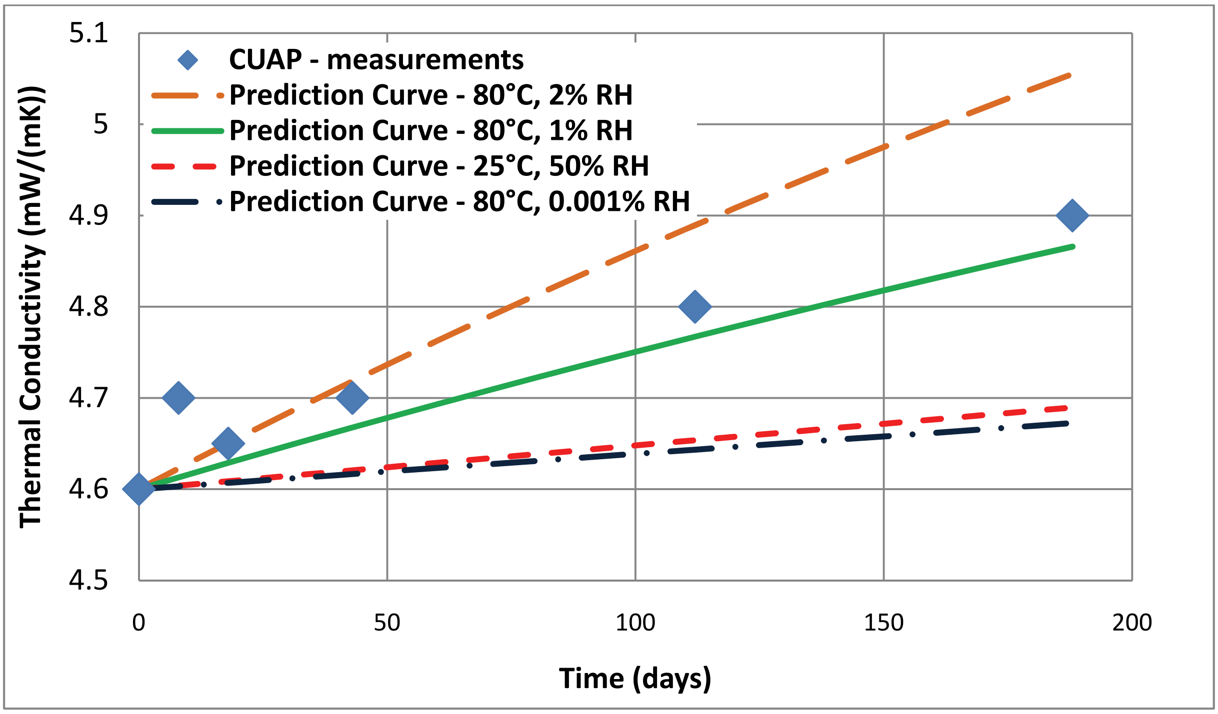


**Fig. 34 Comparison of results from the CUAP experiment with theoretical prediction curves. The thermal conductivity values measured for the CUAP VIP has been normalized for a standard thickness, to compensate for the swelling experienced during the experiment.**

In general the thermal conductivity increase was relatively low, as expected, and the VIPs showed high resistance to extreme climatic strains. The results indicate that the VIPs have indeed experienced an accelerated ageing, and final thermal conductivities also show that the experiments have not damaged the envelope barrier or compromised the resistance to gas and water vapour diffusion. If any of the latter effects had been discovered, the conducted ageing experiments would be deemed unfit.

The temperature and moisture experiment showed potential to give the highest acceleration effect, but was discontinued when failure of the VIP was discovered.  The few results found prior to this are inconclusive as to the acceleration effect of the procedure. From theoretical relationships it seems likely that this method will provide at least as high acceleration effect as the CUAP method, as moisture permeates more easily through the envelope than air gases, and since moisture can potentially contribute greatly to the thermal conductivity. However, results from other studies suggest that such high moisture contents and temperatures as used here will lead to failure of the VIPs within 2 years (Brunner et al. 2008). Some moisture diffusion is to be expected for VIPs in building applications, and one of the weaknesses of the CUAP method is that it does not specify the moisture content for the external climate of the VIP. The VIP exposed to the ageing procedure from the CUAP experienced a steady reduction of thermal performance. The change was slow, however, and the VIP showed high resistance to the extreme temperatures. The CUAP procedure is evaluated below.

Two of the VIPs exposed to ageing conditions, the temperature and moisture aged VIP and the protected VIP in the climate simulator, showed reductions in thermal properties that cannot be accounted for by ageing effects alone. The temperature and moisture aged VIP had a thermal conductivity of 17.9 mW/(mK) after 60 days in ageing conditions. The protected VIP in the climate simulator experienced a more modest increase, but 6.6 mW/(mK) is still far higher than expected, especially when the exposed VIP in the simulator showed no increase in conductivity in the same period. These failures show the obvious weakness of using only one sample for each ageing procedure. Ideally, more samples should have been used, to eliminate the effects of possible outliers or premature failures. The failures does also show one of the major weaknesses of VIPs; their relative fragility. When VIP failures is found even in the controlled environment of the laboratory, where special care is taken to avoid mechanical damage, it shows that it is a thermal insulation solution that demands extreme care in building applications. And even this is no guarantee to avoid failure.

The exposed panel in the vertical climate simulator showed remarkably stable thermal properties. Apart from the physical alterations to the VIP at the beginning of the exposure, no further changes occurred during the 180 days of climate exposure. The thermal conductivity rose merely 0.25 mW/(mK) during this period. This demonstrates the high resistance of VIPs to the various climatic loads experienced in the climate simulator.

## 11.3 Evaluation of the CUAP Method

For the temperature ageing test performed in this work, a method suggested in CUAP 12.01/30 has been employed. The basis for choosing this method is twofold. Firstly, in lack of other standardized methods, it was a natural starting point for the evaluation and design of accelerating experiments. Secondly, the performed experiments would help in evaluating the suitability and ageing effects of the CUAP method itself.

The basis for the proposed procedure is based on the common understanding that increased temperature has accelerating effects on building materials and components. For VIP application however, the method lacks several identifiable variables. Most pronounced of these is that no climate factors except temperature are specified. With knowledge on the vital impact moisture has on long term performance of VIPs, limits for relative humidity should be specified. The CUAP states that the ageing procedure is supposed to cover 25 years of ageing, but it is not specified for what conditions or applications this assumption is made. This ought to be included in a complete procedure. The final measured thermal conductivity for the VIP tested in this work, after 180 days, was 5.15 mW/(mK). Comparing this to the 100 years plots for thermal conductivity in Fig. 13 and Fig. 14 it seems that the CUAP falls a little short of covering an ageing period of 25 years.

Finally, results presented in this work suggest that a temperature of 80°C might be too harsh on the VIPs. Although temperatures up to 80°C might be encountered in parts of a normal building, e.g. in roofs and wall constructions, it is limited to short periods of exposure. Subjecting the VIPs to 80°C for an extended period of time might lead to effects on the VIP that is unlikely in real life application, and will thus give wrong information on the VIP performance. As a comparison, a typical upper maximum temperature used for ageing of polymers is between 60°C - 70°C.

## 11.4 Pressure Tests

The results from the pressure testing are summarized in Table 3 and Fig. 33. VIP sample V2, which was exposed to 8 bar overpressure for a short time only, experienced an instantaneous increase in thermal conductivity of 1.5 mW/(mK). This can possibly be explained by increased solid conductivity of the VIP as pores collapse under the applied high pressure. This is in agreement with results performed within the IEA/ECBS project on VIPs by the National Research Council of Canada (NRC). In that study 4 VIP samples were subjected to 5 bar overpressure for 30 days and then 3 bar overpressure for 15 days. All panels experienced shrinkage of approximately 6%, and a sharp decrease in thermal resistivity. This is concluded to be an effect of collapsed pore structure (IEA/ECBS, Annex 39).

In this study, VIP samples V3 and V4 experienced a much larger increase in thermal conductivity than the V2 panel, but only a slight difference in shrinkage. This relatively high increase cannot be interpreted in terms of the theoretical predictions for accelerated gas diffusion due to increased external pressure as stated in ch. 8.3 alone. It could either be explained by a further change of the pore structure of the core over time, or by some other unknown effect on the VIP, owing to the high pressure. Another explanation could be that the increased pressure leads to an increased diffusion of gases through microscopic failures in the VIP envelope which cannot be accounted for with the prediction models applied.

Whatever the reason, it is considered significant that the thermal properties of the VIPs continued to degrade over time at elevated pressures, despite the geometrical reductions remaining comparatively constant. The mechanisms leading to increased thermal conductivity over time is difficult to ascertain, but is assumed to be a combination of increased solid conductivity due to collapsed pores and some unknown effect on the core or envelope due to the high external pressure.

Based on these preliminary results on pressure ageing of VIPs, the suitability of the procedure ought to be evaluated. One obvious weakness of the method is that it is difficult to determine the cause of the observed changes in VIP properties. Ideally the internal gas pressure of the VIPs should be determined to register any diffusion of gases into the VIPs. In addition, ideally more samples should be used, and should be stored for longer periods of time to evaluate any difference this might have on the VIP properties. This could be the scope of future investigations. Despite these drawbacks, the procedure was intended to uncover any difference between just pressurizing VIPs for a short time and storing at an elevated pressure for an extended period of time. This was accomplished. The procedure is thus considered relevant for a situation where the possible effects were largely unknown.

Results in this work indicate that elevated pressures might serve as an accelerated ageing method of VIPs. Attention should, however, be given to the effects shrinkage has on the core material and VIP properties.

## 11.5 Physical alterations

During some of the experiments, physical changes occurred on the panels, which might or might not affect their performance over time. The most pronounced visible change was that the outer fire protection fleece layer loosened from the VIP envelope. The effect of this was purely visual, as no change in thermal conductivity occurred. The change in fire resistance of the panel has not been tested, though. The most likely reason the fleece lifted from the envelope was due to induced failures in the glue used to fasten it when subjected to high temperatures. One should note that temperatures up to about 60°C and somewhat above may occur at the surface of VIPs in some wall or roof applications.

Another interesting physical change occurred on the VIP exposed to the CUAP ageing method. This panel swelled after some time in the heating cabinet at 80°C. This did not have any direct effect on thermal performance, as the thermal resistance of the panel remained much the same. It is, however, difficult to judge what causes this swelling, and what effect it might have on the VIP performance over time. This has not been investigated further at this time.

When the exposed panel in the vertical climate simulator was taken out of the simulator for testing, it was found that the VIP had curved towards the exposed side. The curve was quite pronounced as can be seen from Fig. 29. This can possibly be explained either by a reaction to the rapidly changing temperatures, or to the exposure to UV-radiation. Some physical changes to the envelope laminate may cause tension in the VIP and thus force the panel to curve. This effect is especially interesting for interim storage of VIPs on construction sites. Special care should be taken to protect VIPs from severe temperature strains or UV-radiation during storage.

Most of the physical changes of the VIPs in this study might be an effect of the severe climatic strains they are exposed to. Too much emphasis on these changes should therefore be avoided. However, for the design and evaluation of future ageing experiments, it will be of interest to be aware of such changes occurring.

## 11.6 Possible Water Permeation Effects

The effects of water vapour on VIPs in actual building applications are difficult to estimate. Still it is one of the most important parameters for performance of VIPs over time. A discussion on the various effects moisture may have on the core material and on the thermal performance of VIPs is found in ch. 5 and 6.

It is certain that moisture permeates through the envelope over time. Desiccants are commonly not used for modern VIPs, as the silica based core material has a quite high capacity to absorb water. But, this absorbed water will also have an effect on the thermal conduction through the VIP. How much water can be absorbed by the core material before a pronounced effect is noticed is not presently known.

In addition, although VIP envelopes are more permeable to water vapour than air gases, one could argue that the effect of moisture on thermal conductivity is limited. Assuming a constant moisture isotherm of $X_w=0.08$ (mass% RH) the moisture content of the VIP reaches equilibrium at 6 mass% for 75 % RH. This leads to a rise in thermal conductivity of 3 mW/(mK). Although this is a considerable increase, a constant external environment of 75 % RH is unlikely. If, in a real life situation, RH varies between 20 % and 75 %, the moisture content would be between 1.6 mass%, corresponding to an increase in thermal conductivity of 0.8 mW/(mK), and 6 mass% with alternating moisture transport into and out of the VIP. Moisture content will consequently vary, and not reach a definite value. However, it should also be remembered that water vapour contributes to the increase in thermal conductivity, through increased gas pressure. Even if the absorbed moisture has a limited effect, the total effect of moisture may be substantial. Still, it could be argued that the most critical parameter for VIPs in actual constructions is the air pressure increase, as this is only limited as the internal pressure reaches atmospheric pressure.

The effect of moisture on VIP performance over time is not sufficiently known. Especially for VIPs exposed to severe moisture loads, either in special applications or in accelerated ageing, the effects of moisture should be studied more extensively.

# 12 Conclusions

A comprehensive study of ageing of vacuum insulation panels (VIP) has been performed. Through theoretical studies a variety of climate factors important for the ageing of VIPs have been identified e.g. temperature, humidity and pressure. Predictions have been made to evaluate the performance of VIPs with various barrier laminate solutions over time. These predictions have been used as a basis for comparison with VIPs subjected to accelerated ageing. Based on theoretical relationships for ageing of VIPs, miscellaneous strategies of performing accelerated ageing experiments on VIPs have been investigated.

In general, the thermal performance of the VIPs subjected to ageing procedures changed very little. This is, however, in agreement with theoretical predictions, and would be expected from a high performance thermal insulation solution such as VIP.

The temperature and moisture experiment seemed to achieve a quite high acceleration effect, but experiments had to be discontinued due to panel failure. Evidence from literature suggests that the climatic loads in this test might be too severe to serve the purpose of accelerated ageing, as high moisture content in combination with high temperature is found to cause VIP failure.

Some physical changes were observed on the VIPs. On the panels subjected to thermal ageing the outer fire protection fleece layer lifted from its substrate after less than a week, and after approximately 100 days the panel had swelled about 10%. The exposed panel in the climate simulator experienced a similar effect as the thermally aged panel, with the fire protection fleece lifting from the substrate. In addition this panel curved permanently during exposure. However, too much emphasis should not be given to these aspects, as they may be an effect of the extreme climatic conditions that would not be encountered in real building applications.

The panels stored at overpressure (8 bar) for 30 days showed a large increase in thermal conductivity. Some of this increase is an effect of the shrinkage of the panel and increased conductivity due to collapsed pore structure. Similar physical effects on a VIP that was pressurized to 8 bar, then depressurized after a few minutes and stored at atmospheric pressure did not, however, give a similar large increase in thermal conductivity. It could thus be assumed that some other factors contributed to the increase in thermal conductivity, either through physical changes to the core or due to diffusion of gases into the VIP.

Due to the high resistance of VIPs both to temperature, moisture and cyclic loads only a low acceleration effect could be observed for any of the experiments. The CUAP experiment and the moisture and temperature experiment gave the highest significant increase in thermal conductivity. Although VIPs show a high resistance to external climatic loads, it should be noted that it still remain a fragile thermal insulation solution.

## Acknowledgements

This work has been supported by the Research Council of Norway and several partners through the SINTEF/NTNU research project ”*Robust Envelope Construction Details for Buildings of the 21st Century*” (ROBUST) and ”*The Research Centre on Zero Emission Buildings*” (ZEB). The company va-Q-tec, by Roland Caps, is acknowledged for supplying the vacuum insulation panel test samples. Ole Aunrønning (NTNU), Per Christian Moe (SINTEF), Sivert Uvsløkk (SINTEF) and Egil Rognvik (SINTEF) provided valuable help during various experimental tasks.

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## Appendix A - Measurement of Small VIPs in Heat Flow Meter

Due to limited size of pressure tanks, the VIPs used for pressure testing were smaller in size than the panels applied for the other ageing tests. The panels used for the pressure tests were 20 cm x 12 cm x 2 cm, and not covered by the same protection fleece as the larger panels.

Due to the limited size of the panels, measurement of thermal conductivity with the heat flow meter (HFM) proved a challenge. Since the measuring area of the HFM is 30 cm x 30 cm, the VIP was placed in a frame of expanded polystyrene EPS. In addition, external heat flow meters were applied to be able to measure the actual heat flow of the VIP. This is shown in Fig. A1.

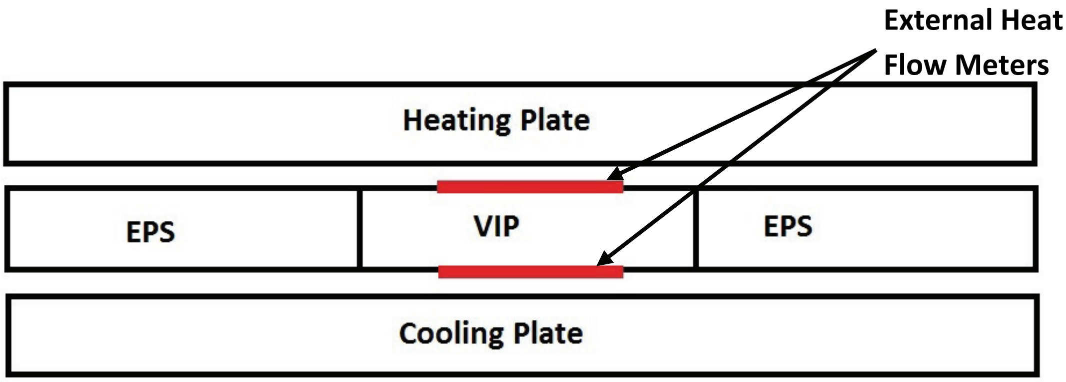


**Fig. A1 Schematic setup for measurement of thermal conductivity of small VIPs using Heat Flow Meter.**

Since the envelope foil might contribute substantially to the heat transport for such small panels, the edge effects on thermal transport were evaluated using the simulation program THERM. Here the thermal conductivity were set as 0.004 W/(m K) for the VIP core, and 0.54 W/(m K) for the envelope. Results of these simulations can be seen in Fig A2 and A3.

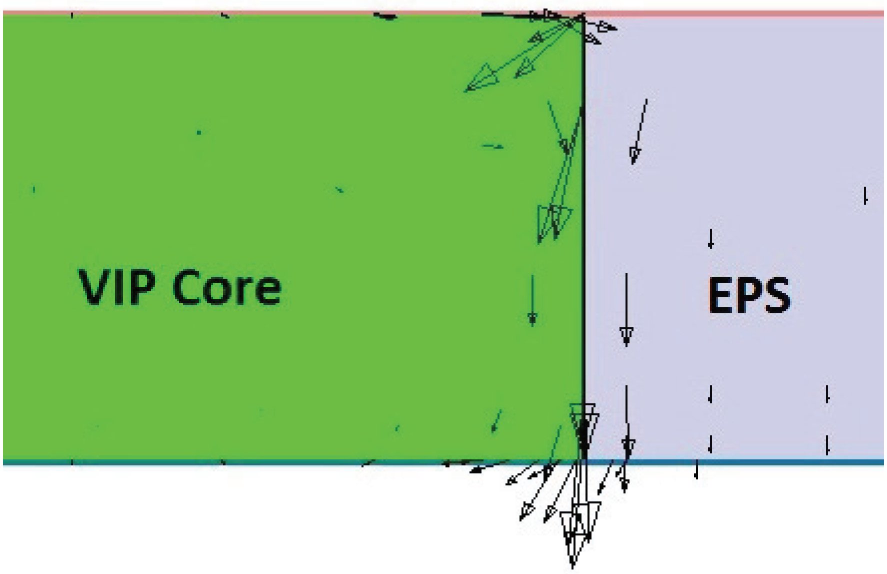


**Fig. A2 Flux Vectors in contact zone bewteen VIP and EPS frame. The heat flux due to the envelope is quite substantial at this point compared to the flux through both the VIP core and the EPS.**

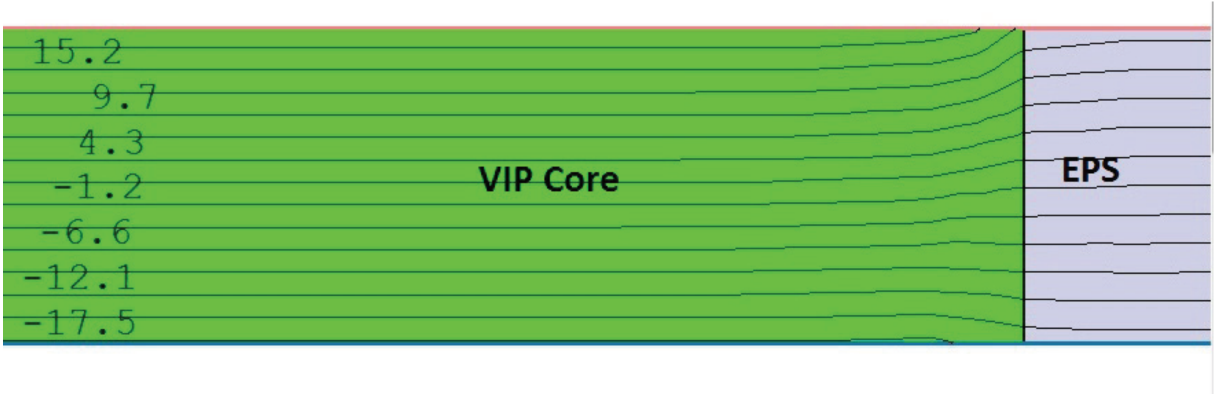


**Fig. A3** **Isotherms in the contact zone between VIP and EPS. The isotherms are relatively stable along the VIP lenght, and the effect of the envelope is limited to a relatively small zone at the edges**

As can be seen from Fig. A3, the envelope has little influence on the thermal transport through the panel. Although the heat flux at the edge of the panel is substantial compared to the center of the panel, it has little effect on the temperature or thermal transport of the rest of the panel.

To evaluate the accuracy of the measurements performed on small samples, as test measurement was performed. A material with known conductivity was first tested with a small area and the external heat flow meters. Then the same material was tested in full size with the normal setup of the heat flow meter. Results from this test gave a difference in conductivity and thermal resistance of 7-8% which is quite substantial. Some improvement to the method is desired. It should be noted, however, that since the measurements performed in relation to this work is meant to track a *change* in thermal conductivity, the uncertainty in the measurements is likely somewhat reduced. Elimination of systematic errors can be expected, at least to some extent.

# Appendix B – Temperature Measurements for VIP in Cyclic Climate Simulator

For the VIP in the wall construction in the vertical climate simulator the temperature on both sides of the panel and on the backside of the wall was measured while exposed to varying climates. Temperatures were measured while the wall was in the freezing chamber and in the UV-chamber. Results show that the temperature at the backside of the VIP is slowest in changing, as would be expected as this face is insulated from both sides. In addition, results show that the temperature at the backside of the wall is quickest in changing, forming a high temperature gradient over the mineral wool. The reason why the temperature at the front of the panel does not change as quickly is probably due to the fact that the ventilated air gap and the wind barrier provide some insulation. In any case the measurements show that there is a large thermal inertia even for this small wall segment. The effect of this inertia is that the panel is not exposed to the most extreme temperatures the test rig can provide. According to measurements, temperatures at the backside of the panel vary from approximately 3°C to +21°C and for the front of the panel the values are -16$^0$C to +34°C. Temperature differences across the panel are measured to be 22.3°C at maximum. This is measured for exposure in the freezing chamber. As the panel moves from the freezing chamber to laboratory climate the temperature difference across the panel levels out.

Fig. B1 and Fig. B2 show the temperature vs. time measurements for exposure in the UV-chamber and the freezing-chamber respectively.

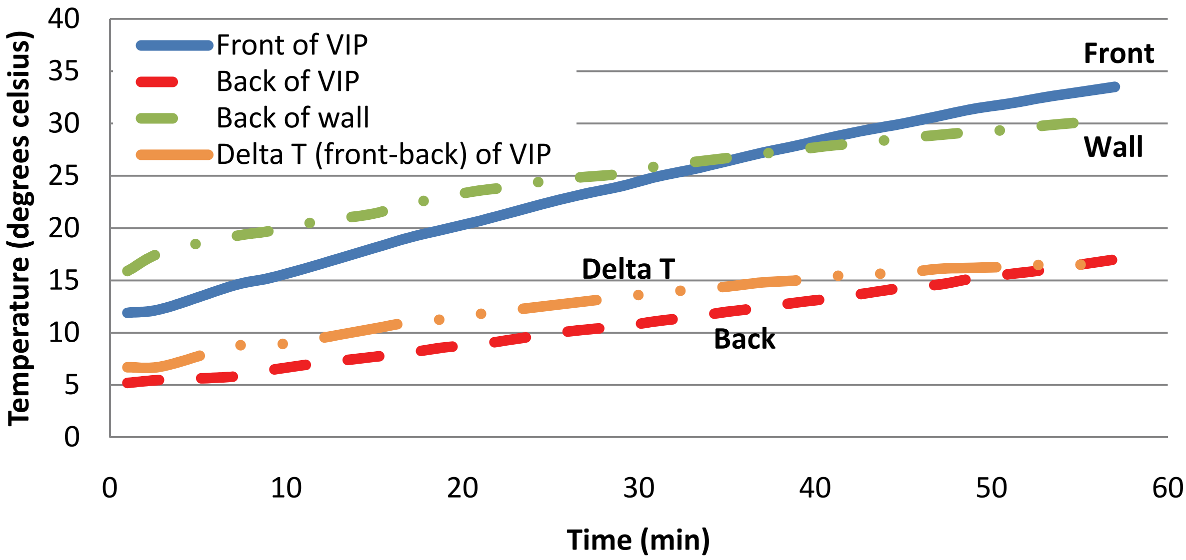


**Fig. B1 Temperature conditions at positions inside the wall construction while exposed in the UV-chamber of the test rig.**

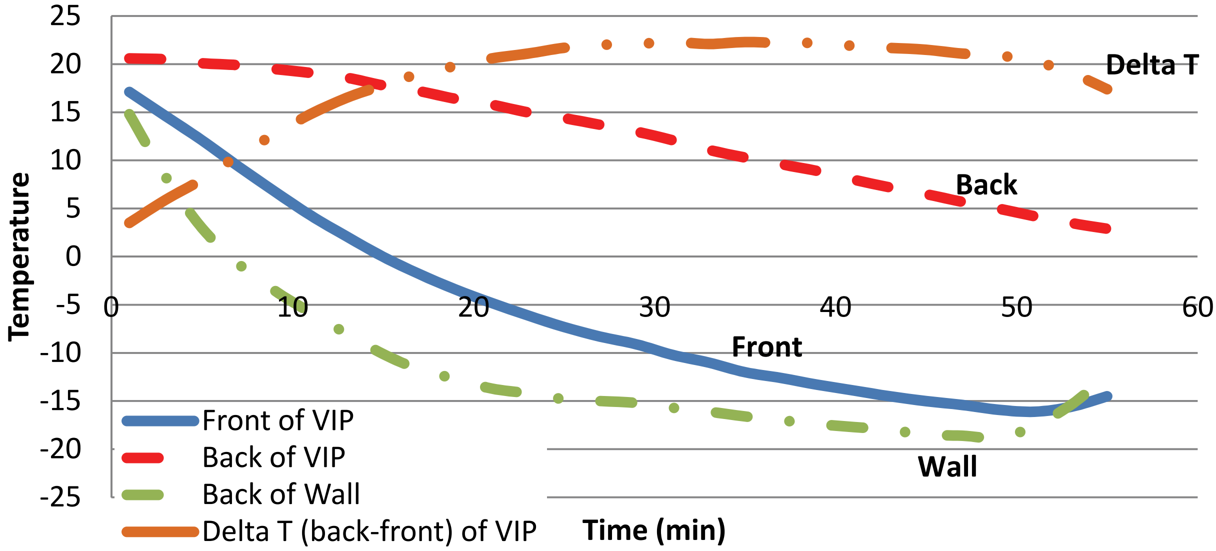


**Fig. B2 Temperature conditions at positions inside the wall construction while exposed in the freezing chamber of the test rig.**

# Appendix C – Analytical Solution for Internal Gas Pressure

The increase in pressure over time can be described as (see Eq. C1):

$$\frac{dp}{dt} = \frac{Q_{gas,tot}\Delta p_{app}}{V_{eff}}\left(\frac{T_m p_0}{T_0}\right) \tag{C1}$$

Where $\Delta p_{app}$ is the applied pressure difference ( $\Delta p_{app} = p_{app} - p$ ).

Setting $\frac{T_m p_0 Q_{gas,tot}}{T_0 V_{eff}}$ = A = constant with respect to time gives:

$$\frac{dp}{dt} = A(p_{app} - p)$$

$$\int_{p_{init}}^{p} \frac{dp}{-p + p_{app}} = A\int_0^t dt$$

$$-\ln(-p + p_{app}) + \ln(-p_{init} + p_{app}) = At$$

$$\ln\left(\frac{-p_{init} + p_{app}}{-p + p_{app}}\right) = At$$

$$\frac{-p + p_{app}}{-p_{init} + p_{app}} = e^{-At}$$

$$-p + p_{app} = (p_{app} - p_{init})e^{-At}$$

$$p = p_{app} - (p_{app} - p_{init})e^{-At}$$

Finally, obtaining an expression for internal pressure in terms of external pressure Eq. C2:

$$p(t) = p_{app} - (p_{app} - p_{init}) \cdot e^{\frac{-T_m p_0 Q_{gas,tot}}{T_0 V_{eff}} t} \tag{C2}$$

# Appendix D –Extended Abstract Submitted for the Building Enclosure Science & Technology (BEST 2 - 2010), Portland, Oregon, U.S.A., 12-14 April, 2010.

## Ageing Effects on Thermal Properties and Service Life of Vacuum Insulation Panels

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Full version of this article is accepted for publication in *Journal of Building Physics*.

*Keywords: Vacuum insulation panel, VIP, Building insulation, Service life prediction, Ageing properties, Accelerated ageing.*

### Introduction

Vacuum insulation panels (VIPs) represent in their pristine condition a high-tech state-of-the-art thermal insulation solution with thermal conductivities 5 to 10 times lower than traditional thermal insulation. VIPs consist of a solid porous core sealed with an airtight and watertight foil maintaining vacuum in the core.

This work presents an overview of the graphical plots relevant for predicting service life of VIPs with respect to increase in thermal conductivity. The governing equations are given in the full version of the article. Furthermore, the background for and how accelerated ageing of VIPs may be carried out is discussed. Finally, some preliminary accelerated ageing experiments are presented.

### VIP Build-up

VIPs are composed of a porous core, usually pressed fumed silica ($SiO_2$), with an air and water vapour tight foil material enveloping the core in order to maintain vacuum inside. In Fig.1 various VIP foil configurations are depicted.

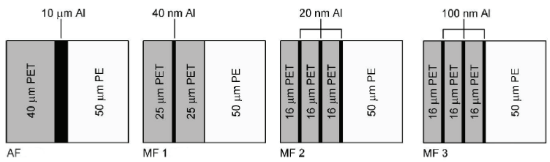


Fig.1. Cross-section of some typical VIP foil configurations. Note that different foil types with the same name are used in literature (Willems et al. 2005).

### Water Content and Air Pressure inside VIPs

Based on work by Scwab et al. (2005), Tenpierik et al. (2007), Baetens et al. (2010) and others, various models and equations are presented for calculation of water content and air pressure increase inside VIPs during time.

The calculated results are shown in Fig.2 and Fig.3 for a time period up to 100 years (Wegger et al. 2010).

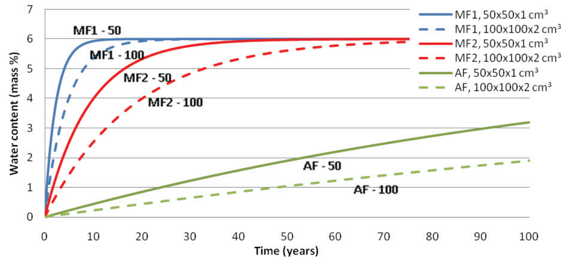


Fig.2. Water content inside VIPs vs. time for various foil types and panel sizes.

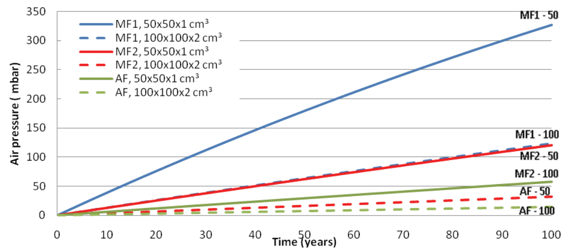


Fig.3. Air pressure inside VIPs vs. time for various foil types and panel sizes.

### Thermal Conductivity of VIPs

Applying the water content (Fig.2) and air pressure (Fig.3) increase inside VIPs, the centre-of-panel thermal conductivity of VIPs during time periods up to 100 years is calculated and shown in Fig.4, where thermal bridges thus are not included (Wegger et al. 2010).

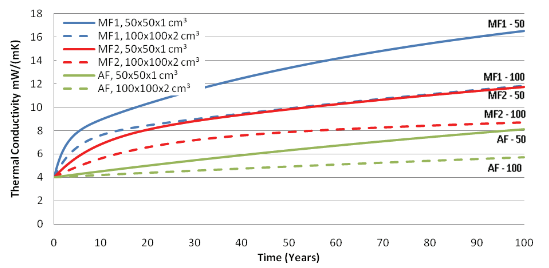


Fig.4. Centre-of-panel thermal conductivity for VIPs vs. time for various foil types and panel sizes.

### Accelerated Ageing of VIPs - Calculations

Accelerated ageing of VIPs during testing and development is desirable in order to determine their service lives within a limited time frame.

Utilizing the various known ageing mechanisms, accelerated ageing experiments may be performed by varying the external environment of the panels, including air temperature, humidity and pressure.

Figure 5 depicts the total thermal conductivity for

VIPs vs. time for different temperatures, and shows a larger increase in thermal conductivity during time with a larger temperature (Wegger et al. 2010).

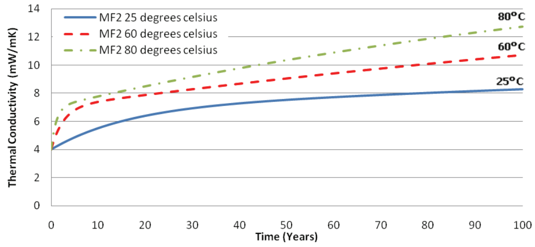


Fig.5. Centre-of-panel thermal conductivity for VIPs vs. time for different temperatures (MF2 foil).

The air pressure and water content increase for VIPs vs. time for different external pressures are demonstrated in Fig.6 and Fig.7, respectively.

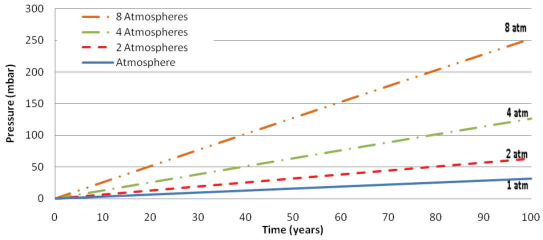


Fig.6. Air pressure inside VIPs vs. time for different external pressures (MF2 foil).

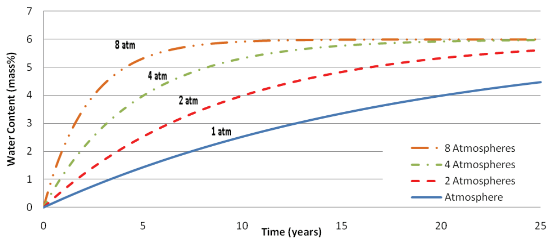


Fig.7. Water content inside VIPs vs. time for different external pressures (MF2 foil).

Based on Fig.6 and Fig.7 the total thermal conductivity for VIPs vs. time for different external pressures is given in Fig.8 (Wegger et al. 2010).

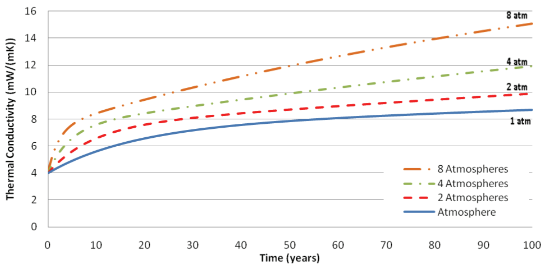


Fig.8. Centre-of-panel thermal conductivity for VIPs vs. time for different external pressures (MF2 foil).

Panel dimensions of 100 cm x 100 cm x 2 cm and MF2 foils are assumed for the VIPs in Figs.5-8. Further details are given in Wegger et al. (2010).

## Accelerated Ageing of VIPs - Experiments

The VIPs employed in the experiments presented here are of the type va-Q-vipB from the producer va-Q-tec (2009). Va-Q-vipB consists of a core of amorphous silicon dioxide and an inorganic opacifier, where the enveloping foil is covered with a black fire protection fleece.

The following two accelerated ageing procedures were carried out:

- **Temperature Ageing – CUAP 12.01/30**
  - Cycling for 8 cycles, where each 24 h cycle consists of 80°C for 8 h and -15°C for 16 h.
  - 80°C for 180 days (180 x 24 h).
  - Thermal conductivity measurements at certain ageing intervals.
- **Climate Ageing – NT Build 495**
  - UV radiation (UVA = 33 W/m$^2$, UVB = 2.4 W/m$^2$) and IR radiation giving a black panel temperature of 63°C.
  - Water spray (15 dm$^3$/(m$^2$h)).
  - Freezing at -20°C.
  - Thawing at laboratory climate.
  - Each 4 h cycle consists of 1 h of each of the above 4 exposures, i.e. 6 cycles per day (24 h). Total ageing duration to be determined.
  - Thermal conductivity measurements at certain ageing intervals.

The thermal conductivity of the VIPs was measured by applying a heat flow meter apparatus.

Two VIPs were employed for the accelerated climate ageing according to NT Build 495. One panel was directly exposed to the various climate strains, whereas the other panel was built into a timber frame wall and thus not directly exposed to the climate strains.

Figure 9 shows the delamination of the VIP fire protection fleece during temperature ageing at 80°C. A similar delamination, in addition to a panel curving, was observed during the ageing according to NT Build 495 for the directly exposed VIP.

The measured total thermal conductivity for three VIPs vs. ageing time for different ageing procedures and exposure conditions are depicted in Fig.10. With a measurement uncertainty of ± 0.1 mW/(mK) it is seen that for the temperature aged VIP (CUAP 12.01/30) and the built-in climate aged VIP (NT Build 495), the variation in thermal conductivity is within the uncertainty, thus no significant difference is observed for either of these two panels. The directly exposed climate aged VIP (NT Build 495) experienced an increase in thermal conductivity of 0.2 mW/(mK) during about 1 month.

The presented accelerated ageing tests are still ongoing, and firmer conclusions may be drawn after a more prolonged ageing period. It should also be noted that in an actual building application the VIPs are not supposed to be directly exposed to outdoor weather conditions. For further details it is referred to Wegger et al. (2010).

Fig.9. Visible delamination of the VIP fire protection fleece after temperature ageing at 80°C for about 1 month.

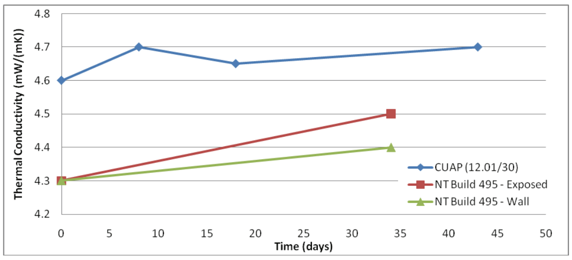


Fig.10. Centre-of-panel thermal conductivity for three VIPs vs. ageing time for different ageing procedures and exposure conditions measured by a heat flow meter apparatus.

## Conclusions

A summary of ageing relationships for VIPs have been presented. Changes in air pressure, water content and thermal conductivity have been plotted for a time span of hundred years for various foil types and panel sizes. Based on these theoretical relationships accelerated ageing of VIPs with respect to air temperature, humidity and pressure has been investigated and some preliminary experiments have been carried out.

## Acknowledgements

This work has been supported by the Research Council of Norway and several partners through the SINTEF and NTNU research project ”*Robust Envelope Construction Details for Buildings of the 21st Century*” (ROBUST). The company va-Q-tec, by Roland Caps, is acknowledged for supplying the vacuum insulation panel test samples.

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# Appendix E – Abstract Submitted for the Renewable Energy Research Conference, 7.-8. June 2010, Trondheim, Norway

## Accelerated Ageing of Vacuum Insulation Panels

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**Introduction**
Vacuum insulation panels (VIP) is a high performance thermal insulation material solution with thermal conductivity values as low as 4.0 mW/(mK). With time the thermal performance of the VIPs will degrade as moisture and gas permeates through the barrier envelope of the panels. To better evaluate these ageing effects, accelerated ageing experiments are needed. Several different experiments are initiated in order to investigate how various external loads affect the ageing of VIPs and which ageing effects that can be achieved. VIPs consist of a porous core of pyrogenic silica ($SiO_2$) and a gas and vapour tight foil. To achieve a sufficient gas and vapour tight barrier and not compromise thermal properties, the foil consists of various layers of metal sheets and polymers. The external factors that are found to contribute most to ageing of VIPs are temperature, moisture and pressure.

**Experimental**
Several experiments have been initiated to evaluate the accelerated ageing effects by the application of severe temperature, moisture and pressure conditions. The following experiments have been initiated:

1. Thermal ageing at 80°C for 180 days according to CUAP 12.01/30
2. Exposure to cyclic climate in a vertical climate simulator according to NT Build 495. One sample is fully exposed in the simulator and one is placed in a wooden frame structure.
3. Exposure to high vapour pressure by storage at 70°C and 90-100 % RH for 90 days.
4. Pressure testing of VIPs to evaluate the resistance to external pressure. Storage for extended period at high pressure.

**Results**
The results so far indicate that thermally aged VIPs show an increase in thermal conductivity similar to what can be predicted using theoretical models. In addition, the thermally aged VIP and the exposed VIP in the climate simulator show physical alterations, e.g. swelling, curving and delamination of the outer fire protection layer are observed.

# Appendix F – Proceedings Article for the Renewable Energy Research Conference, 7.-8. June 2010, Trondheim, Norway

## Accelerated Ageing of Vacuum Insulation Panels (VIPs)

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**ABSTRACT**

Vacuum insulation panels (VIP) is a high performance thermal insulation material solution with thermal conductivity values reaching as low as 4.0 mW/(mK). With time the thermal performance of the VIPs will degrade as moisture and gas permeate through the barrier envelope of the panels. To better evaluate these ageing effects, accelerated ageing experiments are needed. VIPs consist of a porous core of pyrogenic silica ($SiO_2$) and a gas and vapour tight envelope. The external factors that are found to contribute most to ageing of VIPs are temperature, moisture and pressure.

Several experiments have been initiated to evaluate the acceleration effects by the application of severe temperature, moisture and pressure conditions, including:

5. Thermal ageing at 80°C for 180 days according to CUAP 12.01/30
6. Exposure to cyclic climate in a vertical climate simulator according to NT Build 495. One VIP sample is fully exposed in the simulator and one is placed in a wooden frame structure.
7. Exposure to high vapour pressure by storage at 70°C and 90-100 % RH for 90 days.

The increases in thermal conductivity during ageing were relatively small compared to the initial thermal conductivity of the VIPs, which is in agreement with the theoretical predictions. The temperature and moisture experiment seemed to achieve a rather large acceleration effect.

In addition, the thermally aged VIP and the exposed VIP in the climate simulator show physical alterations. E.g. swelling, curving and delamination of the outer fire protection layer are observed.

**Keywords:** Vacuum Insulation Panel, VIP, accelerated ageing, thermal insulation

# 1. INTRODUCTION

For several decades, thermal insulation has been the preferred way to improve buildings energy efficiency, and the thermal insulation requirements have increased steadily. In Norway the requirement of a wall construction in 2010 is an U-value of 0.18 $W/(m^2K)$, which is equivalent to 250 mm mineral wool insulation. Future requirements in order to obtain zero emission standards may require wall thicknesses up to 500 mm filled with mineral wool. Obviously, such wall thicknesses and amounts of insulation are a challenge both for architects and engineers in building aesthetically, economically and in accordance with sound building physical principles.

Vacuum insulation panels (VIP) might offer a solution to this problem. VIPs consist of a solid, porous core which is sealed with an air- and watertight laminate maintaining a vacuum in the core. VIPs have thermal conductivities that are 5-10 times lower than for traditional thermal insulation. It will thus be possible to reduce the thickness of the building walls, and at the same time retain or even increase the thermal resistance.

As an innovative material, special concern is given to the performance of VIPs over time. The thermal performance will decrease over time, as air gases and water vapour penetrate through the barrier envelope. To evaluate the performance and ageing effects of VIPs, accelerated ageing experiments are vital for such investigations to be carried out within a limited timeframe. As no standardized methods exist at this time, a variety of ageing experiments are tested to evaluate their suitability as accelerated ageing methods for VIPs.

The ageing of VIPs depend both on the core material of the panels, and on the materials used for envelope barrier. The most common core material for VIPs are pressed fumed silica or pyrogenic silica ($SiO_2$), which is a fine powdered, highly porous, silica based material with low solid state conductivity. This low solid state conductivity combined with low gas conduction in the vacuum pores lead to the high thermal performance of VIPs.

As envelope barriers the most common materials are so called multilayer (MF) foils, which consist of several layers of aluminum-metalized polyethylene terephthalate (PET) or polypropylene (PP) sheets sealed on the inside with a polyethylene (PE) layer. This provides sufficient gas and vapour tightness and a minimal thermal bridge at the panel edges. A variety of foil configurations are shown in Figure 1.

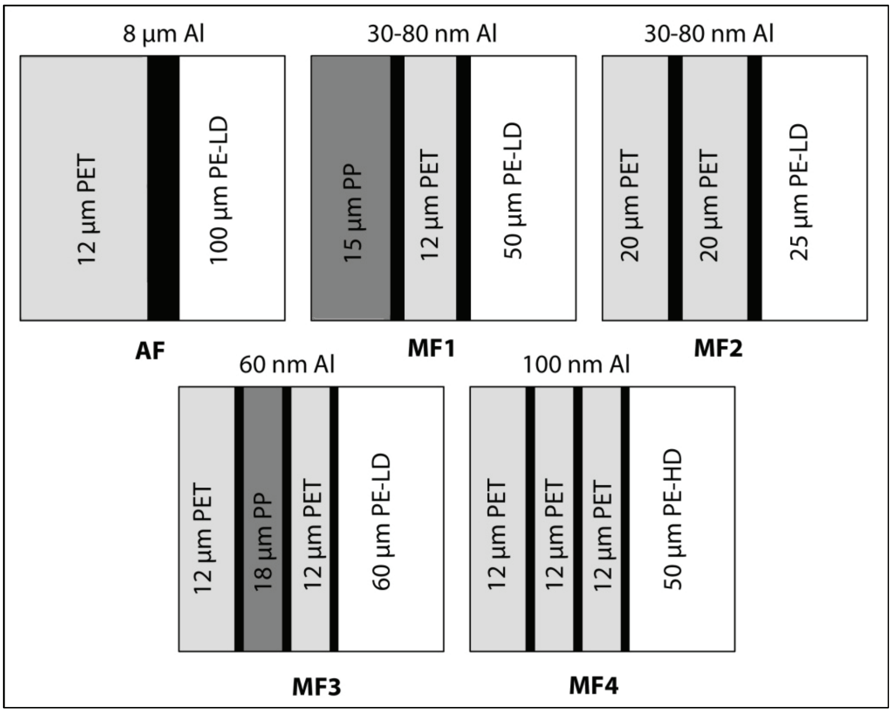


**Figure 1 Cross-sections of various envelope solutions for application in VIPs. The drawing are not to scale.**

## 2. BACKGROUND

The ageing of VIPs is dependent upon the gas and vapour diffusion through the envelope barrier into the core. This will only be summarized briefly here. A comprehensive review of most VIP aspects can be found in Baetens *et al.* (2010).

The total center-of-panel thermal conductivity of VIPs ($\lambda_{tot}$) is considered as a sum of the contributions from solid conductivity ($\lambda_{cd}$), gaseous conductivity ($\lambda_g$), radiative thermal conductivity ($\lambda_r$), and conduction due to air and moisture convection in the pores ($\lambda_{cv}$). In addition a coupling term ($\lambda_{coup}$) is added to account for the interaction between the gas molecules and the pore walls (Brodt 1995).

$$\lambda_{tot} = \lambda_r + \lambda_{cd} + \lambda_g + \lambda_{cv} + \lambda_{coup} \tag{1}$$

As gas and water vapour penetrate the barrier foil, the gas pressure and water content of the core increases, leading to an increased gaseous and solid thermal conductivity. The increase in thermal conductivity over time may be calculated as (Tenpierik 2010):

$$\begin{aligned}\Delta\lambda_c &= \frac{\partial\lambda_c}{\partial p_g}\Delta p_g + \frac{\partial\lambda_c}{\partial p_{wv}}\Delta p_{wv} + \frac{\partial\lambda_c}{\partial u}\Delta u \\ &\approx \frac{\partial\lambda_c}{\partial p_g} p_{g;e}(1-e^{-(t-t_{get})/\tau_g}) + \frac{\partial\lambda_c}{\partial p_{wv}} p_{wv;e}(1-e^{-(t-t_{des})/\tau_w}) + \frac{\partial\lambda_c}{\partial u}\frac{du}{d\varphi}\varphi_e(1-e^{-(t-t_{des})/\tau_w})\end{aligned} \tag{2}$$

where

$p_g$ = Pore gas pressure (Pa)

$p_{g,e}$= Atmospheric gas pressure (Pa)

$p_{wv,e}$ = Partial water vapour pressure outside the VIP (Pa)

$\varphi_e$ = Relative humidity of the air outside the VIP (-)

$u$ = Water content of the core material (-)

$t$ = Time (days)

$t_{get}$ and $t_{des}$ = Time shifts due to getters and desiccants (s)

$\tau_g$ and $\tau_w$ are time constants according to:

$$\tau_g = \frac{\varepsilon V}{GTR(T,\varphi)} \cdot \frac{T_0}{p_o T} \tag{3}$$

$$\tau_w = \frac{\rho_{dry} V}{WVTR(T,\varphi)} \cdot \frac{1}{p_{sat}(T)} \frac{du}{d\varphi} \tag{4}$$

GTR and WVTR are the empirically found *gas transmission rate* and *water vapour transmission rate* for VIPs, respectively. Graphical plots for thermal conductivity for constant climatic conditions up to 100 years can be found in Baetens *et al.* (2010) and Wegger *et al.* (2010).

To be able to accelerate the ageing of VIPs, it is necessary to increase the permeation of water vapour and air gases through the envelope barrier. In Wegger *et al.* (2010) it is found that temperature, moisture and pressure are the external factors contributing most to ageing of VIPs. These external factors are all elements in the thermal conductivity formula in eq. 1.

## 3. EXPERIMENTAL

Three different ageing methods have been applied in this study, based on existing methods and knowledge on ageing. The ageing procedures are described below.

The VIPs used in the experiments presented in this paper are of the type va-Q-vipB from the producer va-Q-tec (2009). Va-Q-vipB consists of a core of amorphous silicon dioxide and an inorganic opacifier. The panel is sealed with a high barrier film which is again covered on the exterior with a black fire protection fleece. The high barrier film consists of three layers of metalized polyethylene terephthalate (PET) with polyethylene (PE) as a sealing layer on the inside. Total thickness is approximately 100 µm. The panel dimensions are 100 cm x 60 cm x 2 cm. One VIP sample is used for each procedure.

To evaluate the change in thermal conductivity of VIPs, a heat flow meter apparatus (HFM) has been used. All measurements are performed in accordance with current versions of ISO 8301 and NS-EN 12667.

## 3.1 Temperature Ageing According to CUAP 12.01/30

One method for testing ageing effects on VIPs is suggested in CUAP 12.01/30. The test is based on severe temperature conditions over an extended period of time. The accelerated temperature ageing is supposed to cover a natural ageing time span of 25 years.

### 3.1.1. Scope

The main scope of the experiment is to verify whether an ageing of 25 years can be achieved by application of this procedure. The procedure has been altered somewhat, to accommodate more measurements than originally specified.

### 3.1.2 Procedure

- Conditioning at (23 ± 2)°C and (50 ± 5)% RH for at least 72 hours.
- Determination of initial thermal conductivity
- Cycling in alternating climate (8 cycles), where one cycle consists of:
  - 8 hours at (80 ± 3) °C
  - 16 hours at (-15 ± 3) °C
- Determination of thermal conductivity
- Temperature ageing for 90 days at (80 ± 3) °C
- Determination of thermal conductivity
- Temperature ageing continued for another 90 days at (80 ± 3) °C
- Final determination of thermal conductivity

Additional measurements of thermal conductivity were conducted when considered required. Alternating climate was achieved by manually transferring the VIP between a heating cabinet and a freezer at the end of each period.

## 3.2 Cyclic Climate Ageing According to NT Build 495

The Nordtest Method NT Build 495 is a test method exposing materials in the vertical position to accelerated climate strains.

### 3.2.1. Scope

The scope of this experiment is to evaluate the resistance of VIPs to varying climate strains. This involves the integrity of the panels in addition to the thermal properties. By using two samples, one exposed and one protected by a timber-frame, the durability and robustness of exposed VIPs can be evaluated and compared to that of protected VIPs. The testing of the exposed VIP would especially be interesting for storage and handling of VIPs during the construction phase.

### 3.2.2 Procedure

The test rig consists of the following successive climate strains:

- UV-radiation (UVA = 33 W/m$^2$, UVB = 2.4 W/m$^2$) and IR-radiation giving a black panel temperature of (63 ± 5) °C
- Wetting with a spray of water
- Freezing at -20 ± 5°C
- Thawing at laboratory climate

The time interval in each of the climate strain positions is one hour. The setup of the test rig is shown in Figure 2.

The test consists of two different specimens. One is a VIP that is directly exposed to the climatic strains. The other specimen is a VIP built into a ventilated timber frame wall. Wall construction details are shown in Figure 3.

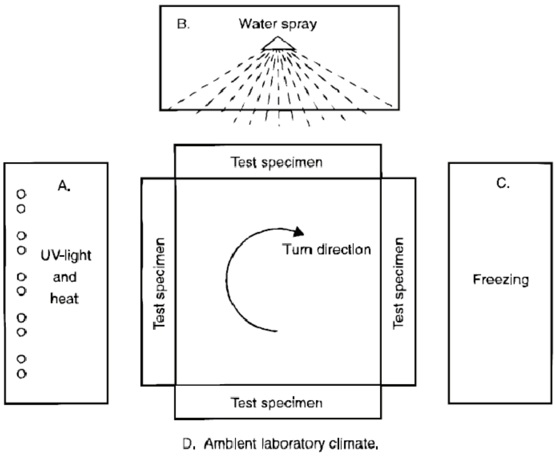


**Figure 2 Test rig for accelerated climate exposure according to NT Build 495 (2000)**

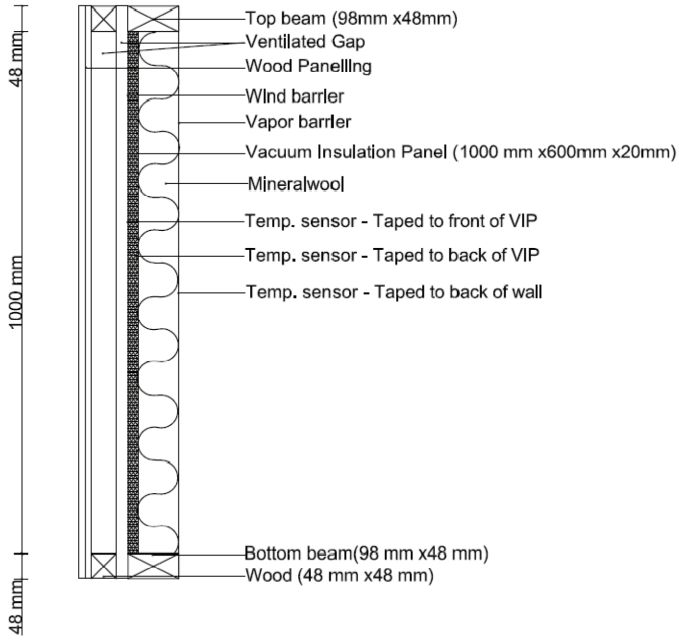


**Figure 3 Construction detail for wall exposed to accelerated climate strains.**

To be able to evaluate any change in the internal conditions after exposure, the initial VIP weight and thermal conductivity were determined.

## 3.3 Moisture and Temperature Ageing

To evaluate the effect of severe hygrothermal conditions on VIPs, a test is designed to expose a VIP to high temperature in combination with high moisture pressure.

### 3.3.1 Scope

The scope of the experiment is to evaluate which ageing effect that can be achieved by exposing a VIP to high relative humidity and high temperature simultaneously. Since saturation vapour pressure show an exponential increase with temperature, a very high external moisture pressure is possible when the temperature is increased.

### 3.3.2 Experimental Setup

In this preliminary experiment it is desired to maximize the moisture pressure within the specified temperature limits for the VIP. To facilitate this, the VIP is sealed inside a plastic envelope together with a water container. The whole envelope is then placed in a heating cabinet at 70°C, giving a RH of between 90 and 100%.

The following procedure has been employed in the testing:

- Conditioning at (23 ± 2) °C and (50 ± 5)% RH for at least 72 hours.
- Determination of initial thermal conductivity
- Storage in heating cabinet (with water container) for 30 days at 70°C
- Determination of thermal conductivity
- Storage in heating cabinet (with water container) for 30 days at 70°C
- Determination of thermal conductivity
- Storage in heating cabinet (with water container) for 30 days at 70°C
- Final determination of thermal conductivity

# 4. RESULTS AND DISCUSSIONS

## 4.1 Temperature Ageing According to CUAP 12.01/30

The initial thermal conductivity was measured to be 4.6 ± 0.1 mW/(mK). The panel was then subjected to freeze/thaw cycles. At the end of these cycles the outer fleece had begun to fray, and the laminate beneath were visible.

The VIP was then stored at 80°C in a heating cabinet. After less than a week, delamination of the outer fleece layer of the VIP was visible. Large areas of the fleece had loosened from the substrate, creating blisters of various shapes and sizes. These blisters became more pronounced over time, which is depicted in Figure 4.

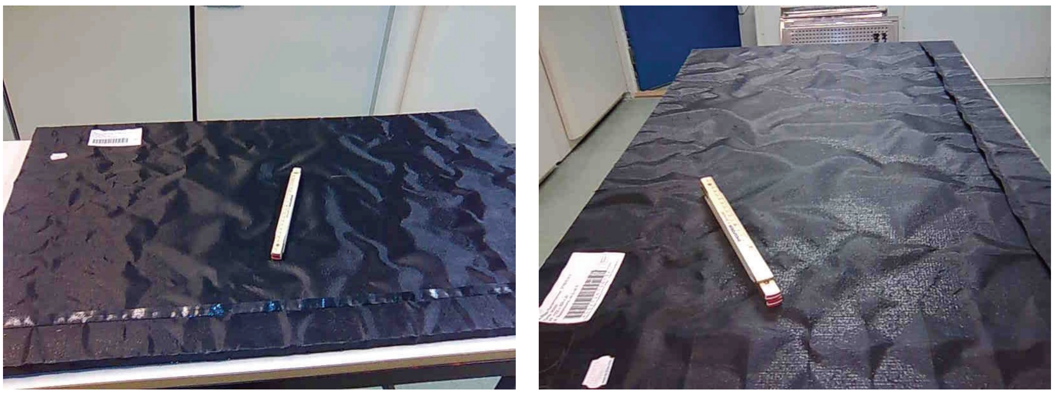

**Figure 4 Visible delamination of the fleece cover after exposure at 80°C for approximately 1 month.**

When thermal conductivity measurements were performed after approximately 100 days in ageing conditions, it became evident that the panel had swelled approximately 2 mm. This corresponds to 5-10% increase in thickness for the panel. Thermal conductivity measurements for the temperature ageing are summarized in Figure 6 and 7.

### 4.2 Cyclic Climate Ageing According to NT Build 495

After less than a day in the vertical climate simulator, the outer fleece layer on the exposed panel began blistering from the substrate, similar to the panel in the heating cabinet. The delamination did not, however, sustain, and only relatively small areas blistered. Another effect on the exposed panel was that it curved slightly towards the exposed side, as can be seen in Figure 5. For the protected VIP, no such effects was observed.

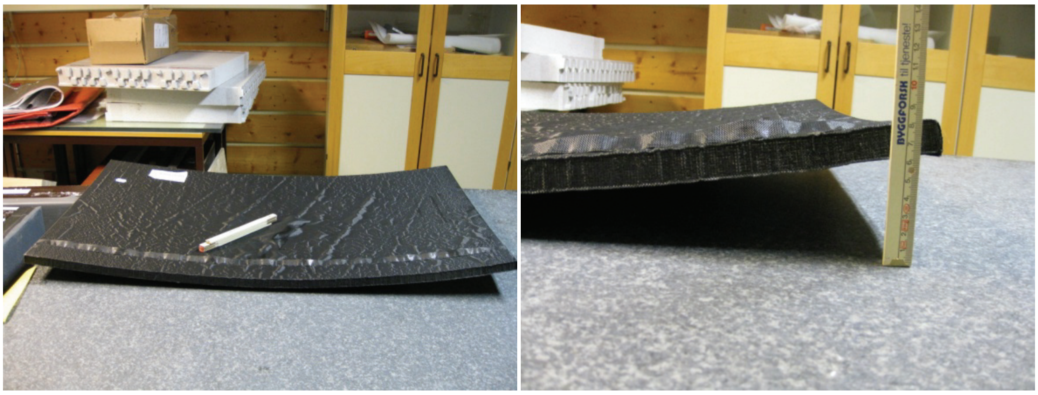


**Figure 5** **Uncovered panel after exposure to cyclic climate strains in vertical climate simulator for approximately one month. Some delamination of the fleece cover is visible. The panel had curved during exposure.**

Thermal conductivity measurements for the cyclic climate ageing are summarized in Figure 6 and 7.

### 4.3 Moisture and Temperature Ageing

When the VIP was tested after 60 days in ageing conditions, its thermal conductivity had increased drastically to 17.9 mW/(mK). This might be best explained by failure of the VIP due to some external source, such as mechanical damage. The experiment was then discontinued. Thermal conductivity measurements for the moisture and temperature ageing are summarized in Figure 6 and 7.

### 4.4 Thermal Conductivity for VIPs exposed to Ageing Experiments

Results from the thermal conductivity measurements are summarized below in Figure 6 and 7. Figure 6 show the changes of thermal conductivity for the various VIPs over time. The test periods vary somewhat. Figure 7 show the thermal resistance for all VIPs. Since some panels experienced physical changes like swelling, the thermal resistance might give a more appropriate measure of the acceleration effect of the various methods.

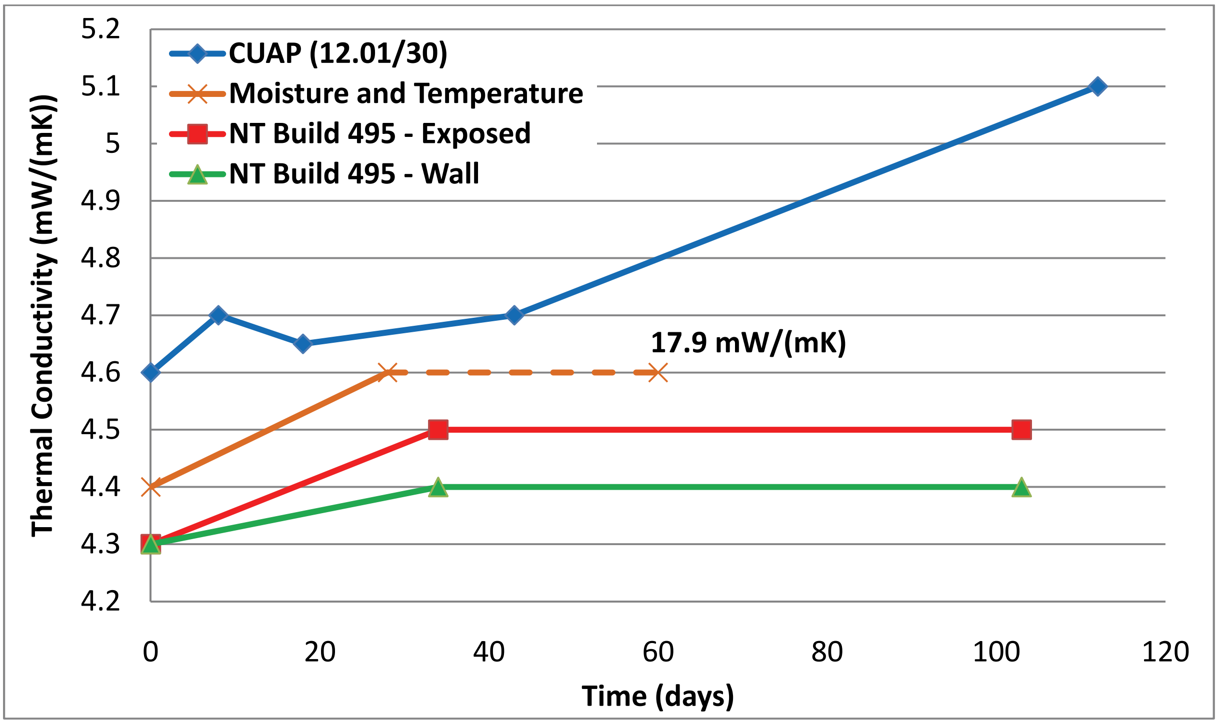


**Figure 6 Thermal conductivity versus time for VIPs exposed to various acceleration procedures**

Note that the initial non-aged thermal conductivity of the VIPs vary by 0.3 mW/(mK) which is approximately 7% of the total conductivity. Due to the relatively low rise in thermal conductivity for the VIPs exposed to ageing procedures, the variation in initial thermal conductivity might have as large or larger impact on thermal performance as the ageing effects. This variation also makes it necessary to confirm results with more extensive testing on several VIP samples.

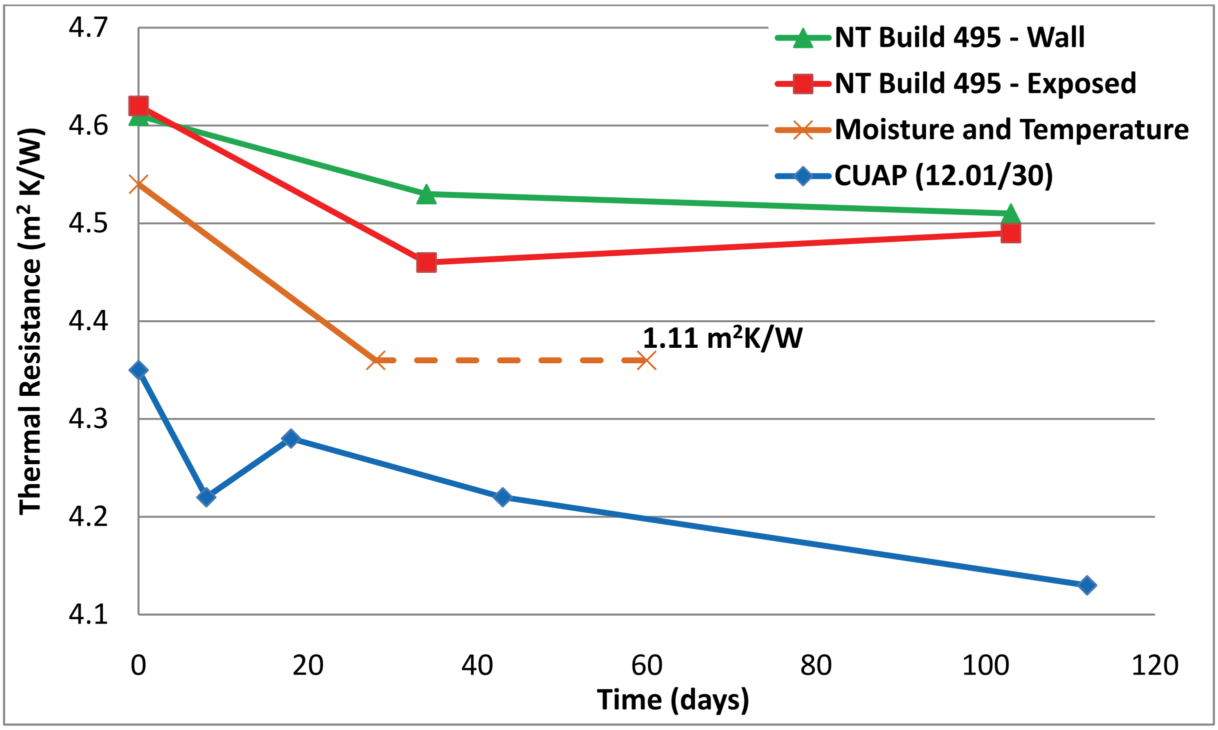


**Figure 7 Thermal resistance versus time for VIPs exposed to various acceleration procedures**

For all ageing procedures the changes in thermal conductivity and resistance are small, compared to the overall performance of VIPs. This shows that the VIPs have a relatively high robustness to severe climatic loads. This is in agreement with prediction curves for the ageing of VIPs under constant climatic conditions. For a VIP with MF2 foil and size 100 cm x 100 cm x 2 cm, the predicted thermal conductivity after 100 years is ≈8.5 mW/(mK). This represents a total increase of 4.5 mW/(m K), or less than 0.05 mW/(mK) each year. In other words, a relatively low increase in thermal conductivity should be expected if the predictions are correct.

One of the most interesting result of these experiments are the physical alterations experienced by the panel at 80°C in heating cabinet (CUAP 12.01/30), and the exposed panel in the vertical climate simulator. The swelling of the panel in the heating cabinet, approximately 10% of panel thickness, is most likely a result of physical changes in the panel core. Seemingly this has no significant effect on the thermal performance. It should also be noted that 80°C is the maximum upper limit of the temperature range specified by the producer for these panels, and will not normally be encountered during the application of VIPs in buildings. It might be experienced for shorter time interval though e.g. on roofs. The temperature is also higher than the temperatures usually applied for ageing of polymers and other plastic materials that might be similar to the VIP envelope. The curvature on the exposed panel in the climate simulator is relevant for interim storage or exposed condictions of VIPs at construction sites. Exposure of VIPs to extreme temperatures and UV-radiation on building sites should therefore be avoided.

Note that most of the physical changes of the VIPs in this study might be an effect of the severe climatic strains they are exposed to. Too much emphasis on these changes should therefore be avoided. However, for the design of future ageing experiments, it will be of interest to be aware of such changes occurring.

The temperature and moisture experiment showed potential to give the highest acceleration effect, but was discontinued when the VIP was punctured. The few results found prior to this are inconclusive as to the acceleration effect of the procedure. From theoretical relationships it seems likely that this method will provide at least as high acceleration effect as the CUAP method, as moisture permeates more easily through the envelope than air gases, and since moisture can potentially contribute greatly to the thermal conductivity. However, results from other studies suggest that such high moisture contents and temperatures will lead to failure of the VIP within 2 years (Brunner et al. 2008).

Of the acceleration methods conducted, only the protected VIP in the climate simulator is considered to receive realistic ageing conditions. Also the CUAP experiment is considered giving a realistic load, although the duration of testing might be discussed. Despite this, all procedures are considered important for the design of future ageing experiments.

## 5. CONCLUSIONS

Miscellaneous ways to perform accelerated climate ageing of vacuum insulation panels (VIP) have been investigated. The changes in thermal conductivity were relatively small compared to the initial thermal conductivity of the VIPs. This is in agreement with the theoretical predictions. The temperature and moisture experiment seemed to achieve a quite high acceleration effect. Evidence from literature suggests that the climatic loads in this test might be too severe to serve the purpose of accelerated ageing.

Some physical changes were observed on the VIPs. On the panels subjected to thermal ageing the outer fleece layer lifted from its substrate after less than a week, and after approximately 100 days the panel had swelled 10%. The exposed panel in the climate simulator experienced a similar effect as the thermally aged panel, with the protection fleece lifting from the substrate. In addition this panel curved permanently during exposure. Too much emphasis should not be given to this, as they might be an effect of the extreme climatic conditions.

## ACKNOWLEDGEMENTS

This work has been supported by the Research Council of Norway and several partners through the SINTEF/NTNU research project ”*Robust Envelope Construction Details for Buildings of the 21st Century*” (ROBUST) and ”*The Research Centre on Zero Emission Buildings*” (ZEB). The company va-Q-tec, by Roland Caps, is acknowledged for supplying the vacuum insulation panel test samples. Ole Aunrønning (NTNU), Per Christian Moe (SINTEF) and Egil Rognvik (SINTEF) provided valuable help during various experimental tasks.

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## Appendix G – Abstract Submitted for the 12th International Conference on Durability of Building Materials and Components, 12.-15. April 2011, Porto, Portugal

# Accelerated Laboratory Ageing of Vacuum Insulation Panels

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**ABSTRACT**

Vacuum Insulation Panels (VIPs) is a high performance thermal insulation material solution. With thermal conductivities as low as 4.0 mW/(mK) it offers a solution of how to meet future requirements for thermal efficiency of buildings, but retain slim building envelopes.

VIPs consist of a porous core, usually fumed powder silica ($SiO_2$), which is sealed in a gas an vapour-tight envelope while there is vaccum in the core. During the VIPs lifetime, air gases and water vapour permeates through the barrier envelope, reducing the thermal performance. This process is highly dependent upon the external climatic factors of the panel.To better evaluate the performance of VIPs over time, and to study how the external climate affect VIP ageing, accelerated ageing test are neccessary. A theoretical and experimental study into accelerated ageing have been performed.

The external factors found theoretically to contribute most to the ageing of VIPs are temperature, moisture and pressure. Utilizing these theoretical relationships, acceleration ageing experiments are designed to test the theoretical predictions and evaluate the ageing effects on VIPs. Within this study, thermal conductivity is employed for evaluation of VIP performance.

*Keywords:* *Accelerated ageing, Laboratory, Vacuum insulation panel, VIP.*

# Appendix H – Presentation given at the Renewable Energy Research Conference, 7.-8. June 2010, Trondheim, Norway

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## Accelerated Ageing of Vacuum Insulation Panels (VIPs)

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*Renewable Energy Research Conference,*
*Trondheim, Norway, 7-8 June, 2010.*



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## Introduction

- Vacuum Insulation Panels (VIPs)
- High-tech state-of-the-art thermal insulation solution
- Ageing of VIPs highly dependent on water content and gas pressure
- Accelerated ageing necessary to evaluate VIP performance over time
- Various accelerated ageing experiments performed

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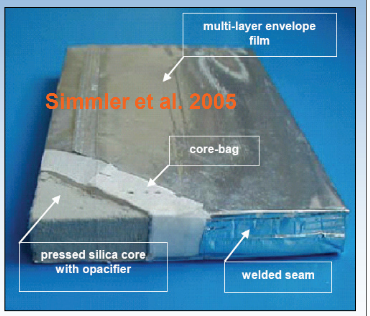
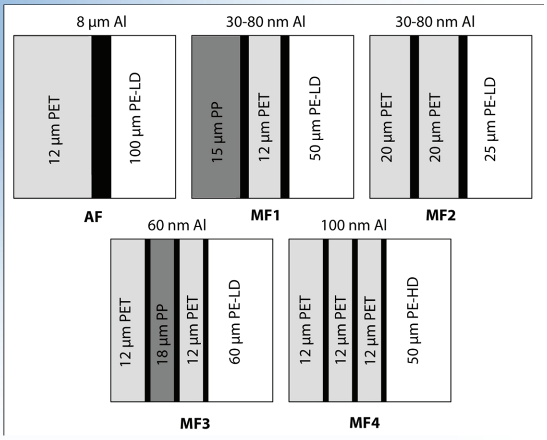
# VIP Buildup

- Porous core – Usually pressed fumed silica ($SiO_2$)
- Air and water vapour tight foil maintaining vacuum in the VIP

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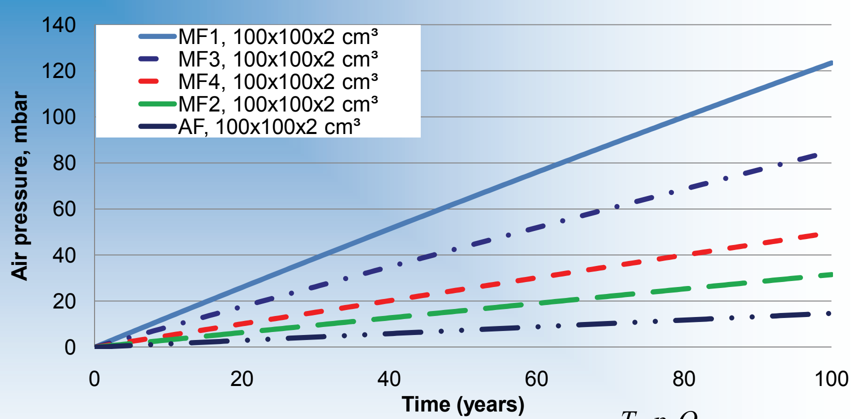
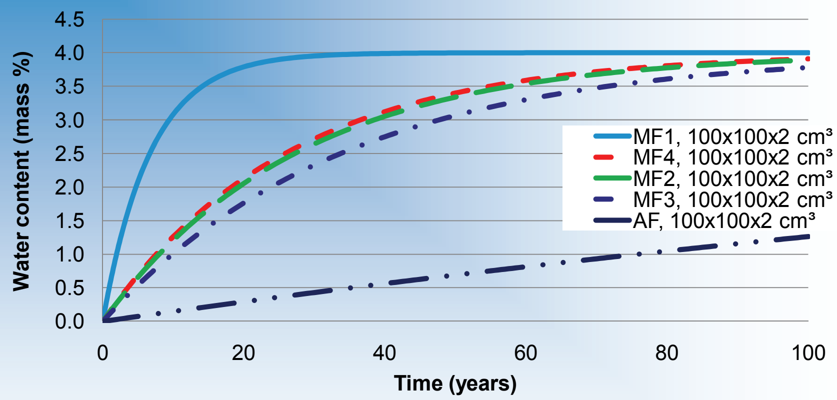
# Prediction Curves – Air Pressure




$$p(t) = p_{app} - (p_{app} - p_{init})e^{\frac{-T_m p_0 Q_{gas,tot}}{T_0 V}t}$$

NTNU 4 SINTEF

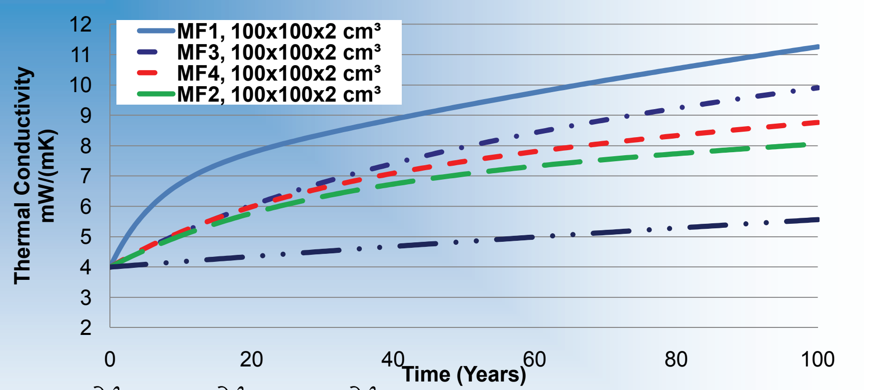
## Prediction Curves – Water Content




$$X_w(t) = k\varphi_{out}\left(1 - e^{\frac{-Q_{wv,tot}\,p_{wv,sat}(T)}{m_{VIP,dry}k}t}\right)$$

5

## Prediction Curves – Thermal Conductivity




$$\Delta\lambda_c = \frac{\partial\lambda_c}{\partial p_g}\Delta p_g + \frac{\partial\lambda_c}{\partial p_{wv}}\Delta p_{wv} + \frac{\partial\lambda_c}{\partial u}\Delta u$$

$$\approx \frac{\partial\lambda_c}{\partial p_g} p_{g;e}(1-e^{-(t-t_{get})/\tau_g}) + \frac{\partial\lambda_c}{\partial p_{wv}} p_{wv;e}(1-e^{-(t-t_{des})/\tau_w}) + \frac{\partial\lambda_c}{\partial u}\frac{du}{d\varphi}\varphi_e(1-e^{-(t-t_{des})/\tau_w})$$

6

## Ageing Experiments

- **Based on known relationships of ageing effects due to gas and water vapour diffusion.**
- **Three experiments conducted:**
  - Temperature ageing according to CUAP 12.01/30
  - Cyclic climate ageing according to NT Build 495
  - Combined moisture and temperature ageing



## CUAP 12.01/30 - Procedure

- **Conditioning at (23 ± 2)°C and (50 ± 5)% RH for at least 72 hours.**
- **Determination of initial thermal conductivity**
- **Cycling in alternating climate (8 cycles), where one cycle consists of:**
  - **8 hours at (80 ± 3)°C**
  - **16 hours at (-15 ± 3)°C**
- **Determination of thermal conductivity**
- **Temperature ageing for 90 days at (80 ± 3)°C**
- **Determination of thermal conductivity**
- **Temperature ageing continued for another 90 days at (80 ± 3)°C**
- **Final determination of thermal conductivity**

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# NT Build 495 - Procedure

- Successive climate strains
  - UV-radiation (UVA = 33 W/m$^2$, UVB = 2.4 W/m$^2$) and IR-radiation giving a black panel temperature of (63 ± 5)°C
  - Wetting with a spray of water
  - Freezing at -20 ± 5°C
  - Thawing at laboratory climate
- The time interval in each of the strain positions is 1 hour.



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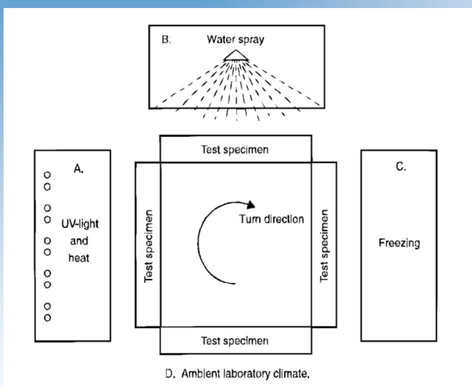
# NT Build 495 - Setup

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## Moisture and Temperature Ageing - Procedure

- **VIP is sealed inside a plastic envelope together with a water container**
  - Conditioning at (23 ± 2)°C and (50 ± 5)% RH for at least 72 hours.
  - Determination of initial thermal conductivity
  - Storage in heating cabinet (with water container) for 30 days at 70°C
  - Determination of thermal conductivity
  - Storage in heating cabinet (with water container) for 30 days at 70°C
  - Determination of thermal conductivity
  - Storage in heating cabinet (with water container) for 30 days at 70°C
  - Final determination of thermal conductivity

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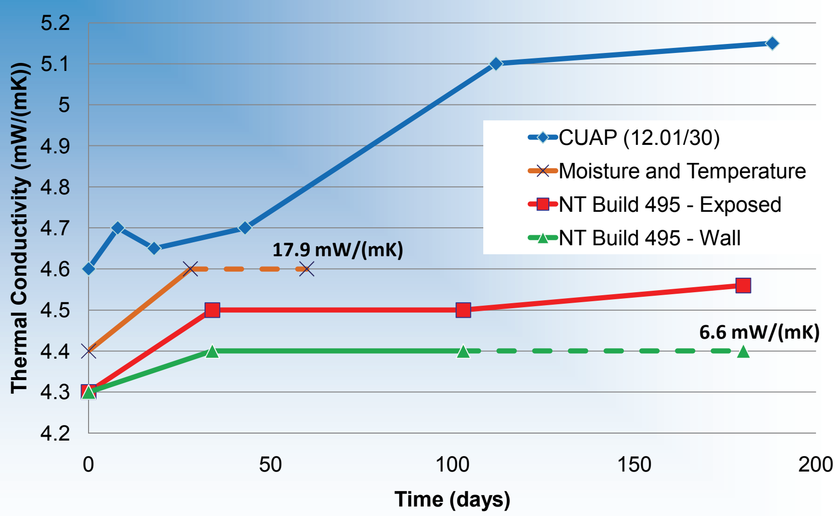
## Results – Thermal Properties




NTNU 12 SINTEF

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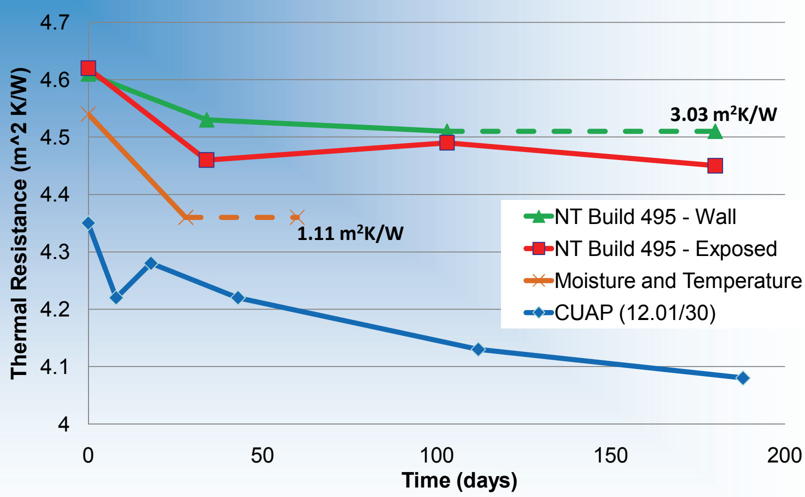
## Results – Thermal Properties



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## Results – Physical alterations

- Some physical changes were observed on the panels
  - Outer fleece layer loosened from substrate
  - Swelling of CUAP VIP
  - Curving of exposed panel in climate simulator
- To much emphasis on these effects should be avoided

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## Results – Physical alterations

CUAP-panel, 1 month

Exposed NT Build-panel, 1 month

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## Conclusions

- Accelerated ageing with respect to temperature, humidity and various other climatic strains have been investigated and presented.
- Generally the changes in thermal properties were small
- Some physical effects were observed on the panels, possibly as an effect of the ageing procedures.

NTNU 16 SINTEF

## Acknowledgements

This work has been supported by the Research Council of Norway and several partners through the SINTEF and NTNU research project ”Robust Envelope Construction Details for Buildings of the 21st Century” (ROBUST). The company va-Q-tec, by Roland Caps, is acknowledged for supplying the vacuum insulation panel test samples.

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