



Accelerated and long-time creep testing of extruded polystyrene using isothermal and stepped isothermal method

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ARTICLE INFO

Keywords:

XPS
Creep behavior
Stepped isothermal method
Findley fit
High temperature
Thermal storage

ABSTRACT

Seasonal Sensible Thermal Energy Storages based on existing inside structures are cost effective and require insulation materials with high performance. The insulation must endure high temperatures and high pressure for a long period. In this study, the creep effects of the insulation material XPS (extruded polystyrene foam) at high temperatures and pressure are investigated to determine whether XPS is a suitable insulation material to be applied on the inside of STES.

Two studies were conducted: an isothermal long-term creep experiment and a SIM (Stepped Isothermal Method) experiment. The results of the first study were fitted using the Findley approach and extrapolated to 50 years. The SIM study is a method to accelerate creep effects using thermal effects and the Boltzmann superposition principle. Creep strain after 50 years is directly calculated by extrapolation. Both studies indicate that creep strain in XPS remains well below 6% with an applied load of 61.1 kPa at 60 °C after 50 years. Additionally, a material model that predicts creep strain at different or varying temperatures and stress was developed using the SIM data.

1. Introduction

In recent years, thermal insulation materials have gained increased attention in the efforts to improve the sustainability of the construction sector through increased energy efficiency and energy storage [1–3]. Particularly in the field of seasonal thermal energy storage (STES), in which heat losses can account for up to 50% of the energy stored, thermal insulation plays a key role in system design [4]. With the aim of reducing investment costs of STES, the reutilization of existing building structures has been suggested as a novel path to achieve the cost-effective integration for STES [5]. This path involves retrofitting the inner walls of underground room enclosures with appropriate vapor barrier and thermal insulation materials to allow such rooms to be transformed into hot-water reservoirs for thermal energy storage. One of the main challenges of incorporating insulation materials on the inside of the storage is limiting the long-term creep deformation that results from the simultaneous occurrence of temperatures up to 60 °C and hydrostatic gauge pressures up to 60 kPa. In this context, extruded polystyrene foam (XPS) had been identified as an attractive thermal

insulation material due to its availability, low cost, low thermal conductivity, and significant creep resistance at room temperature [5,6]. Moreover, XPS panels have been suggested as a suitable solution both thermally and with respect to pressure for road insulation in subzero degree regions in Norway [7]. The specifically in this study anticipated maximum creep strain of XPS at 60 °C and 60 kPa was 6%.

Due to its viscoelastic nature, XPS can be susceptible to significant time-dependent deformation when subjected to loading at an elevated temperature over a prolonged period. Merkel et al. [8] state that the difference between short-term deformation and long-term creep behavior is significant and must be considered when long-term loading is intended. Creep behavior is also of great significance in the field of civil engineering for the design of thermal insulation slabs to be placed underneath building foundations. The prediction of creep behavior is thus indispensable for ensuring the long-term integrity and performance of thermal insulation subjected to elevated temperature and pressure loads. However, long-term creep tests conducted under normal conditions are prohibitively expensive due to the prolonged measurements required to reliably extrapolate the measured data. Alternatively, accelerated creep tests can be used to obtain the creep master curve by fitting the measured data to an empirical

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<https://doi.org/10.1016/j.polymer.2022.124926>

Received 22 September 2021; Received in revised form 31 March 2022; Accepted 1 May 2022

Available online 5 May 2022

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Nomenclature

Symbols

a_0	Findley constant [1/h]
a	Fitting parameter used in Material Models [–]
$a_{T,i}$	Shift factor [–] of step i
b	Findley constant [h], Fitting parameter used in Material Models [–]
c	Fitting parameter used in Material Models [–]
d_i	Fitting parameter used in polynomial regression [–]
$d_i(t)$	Absolute creep measurement data of sample i [mm]
$d_{0,i}$	Initial value of dial gauge i [mm]
$d_{1,i}$	First value after 1 min of pressure of dial gauge i [mm]
$h_{0,i}$	Starting height of sample i [mm]
$h_{m0,i}$	Initial height of sample i [mm]
$h_{r,i}$	Reference height of sample i [mm]
i	Sample number, number of samples, number of steps, step
ke	Acceleration factor [–]
m	Fitting parameter used in Material Models [–]
q	Fitting parameter used in Material Models [–]
t	Time [h]
t_0	Findley constant [h]
$t_{c,i}$	Manipulated start time, manipulation i
t_v	Virtual start time [hours]
T	Temperature [°C]
T_0	Reference temperature with creep strain $\varepsilon_{T_0}(t)$ [°C]
T_1	Reference temperature with creep strain $\varepsilon_{T_1}(t)$ [°C]
$T_{mean,i}$	Mean temperature per step [°C]
$T_{ref,i}$	Reference temperature of step i
$T_{ref,n}$	Reference temperature of last step (n)

Greek symbols

$\varepsilon_0(t)$	Sum of all effects of thermal strain of the measurement setup and the sample [%]
$\varepsilon_f(t)$	Findley creep strain [%]

$\varepsilon_{fit,40}(t)$	Fitted creep strain of long-term experiment at 40 °C [%]
$\varepsilon_{fit,60}(t)$	Fitted creep strain of long-term experiment at 60 °C [%]
$\varepsilon_{i,k}(t)$	Creep strain of step i, manipulated step k [%]
$\varepsilon_{i,40}(t)$	Creep strain of long-term experiment at 40 °C, sample i [%]
$\varepsilon_{i,60}(t)$	Creep strain of long-term experiment at 60 °C, sample i [%]
$\varepsilon_{mas}(t)$	Creep strain of master curve [%]
$\varepsilon_{mat1}(t, T)$	Material Model 1 fitted creep strain [%]
$\varepsilon_{mat2}(t, T)$	Material Model 2 fitted creep strain [%]
$\varepsilon_{mc}(t)$	Creep strain corrected [%]
$\varepsilon_{mc,i}(t)$	Creep strain corrected per step i [%]
$\varepsilon_{mean,40}(t)$	Creep strain of long-term experiment at 40 °C, mean [%]
$\varepsilon_{mean,60}(t)$	Creep strain of long-term experiment at 60 °C, mean [%]
$\varepsilon_{s,i}(t)$	Creep strain of sample i, measurement [%]
$\varepsilon_{T_{ref,n}}(t, T)$	Fitted creep strain of last step (n) [%]
$\varepsilon_{T_{ref,i}}(t, T)$	Fitted creep strain of step i [%]
$\varepsilon_{T_0}(t)$	Creep strain at reference temperature T_0
$\varepsilon_{T_1}(t)$	Creep strain at reference temperature T_1
$\varepsilon_{y50,40}(t)$	Calculated creep strain after 50 years as a function of the amount of measurement data used for fitting at 40 °C [%]
$\varepsilon_{y50,60}(t)$	Calculated creep strain after 50 years as a function of the amount of measurement data used for fitting at 60 °C [%]

Acronyms

DIN EN	European German Standards Organization
EPS	Expanded Polystyrene
PMMA	Polymethylmethacrylate
SIM	Stepped Isothermal Method
SSM	Stepped Isostress Method
STES	Seasonal Thermal Energy Storage
TTSP	Time-Temperature Superposition
TTSSP	Time-Temperature-Stress Superposition
WPC	Wood–Polypropylene Composites
XPS	Extruded Polystyrene Foam

model. Several alternatives exist for predicting the long-term performance using accelerated creep tests based on the application of the superposition principle from the combination of time, temperature, and applied load. Some of the methods commonly used to characterize the creep behavior of polymers include time-temperature-superposition (TTSP), time-temperature-stress-superposition (TTSSP), the stepped isostress method (SSM), and the stepped isothermal method (SIM). Briody et al. describe a method based on TTSP used for forecasting long-term compressive creep behavior on flexible polyurethane foam at elevated temperatures [9]. They conducted measurements between 45 and 83 °C using a purpose-built test rig and successfully generated and validated an extended creep compression curve at a reference temperature of 45 °C. Meanwhile, Chevali et al. applied the TTSSP method to assess the long-term flexural creep behavior of neat and reinforced polymers by conducting measurements between 23 and 120 °C [10]. They found a characteristic curvature at the higher end of the master curve, indicating a steep acceleration of the creep rate with the simultaneous application of an increased temperature and stress load. Furthermore, Hadid et al. used SSM to predict the long-term creep behavior of polyamide 6 [11]. They demonstrated that the master curves obtained via SSM correlate well with those obtained using the classical TSSP method.

As originally proposed by Thornton et al. [12] and restated more recently by numerous studies [13–16], SIM is a promising method for accelerated creep testing. In contrast to TTSP, which requires a test specimen for each temperature, the main advantage of SIM is the need of only one single test specimen to generate a sequence of creep responses under a constant load using a series of elevated temperature steps.

However, little information is available regarding the creep behavior of XPS at elevated temperatures, and the application of SIM with thick (100 mm) thermal insulation samples has not been reported yet in literature. In this work, the creep behavior of XPS is experimentally determined as a function of compressive load (up to 61.1 kPa) and temperature (up to 60 °C) and calculated for a service life of 50 years. The experimental investigation was conducted by applying both SIM and a standard long-term method to predict the long-term creep behavior. In addition, numerical models were developed to allow for predicting creep strains at different temperatures and pressures.

2. Methods and materials

2.1. Thermal insulation material

The material used is XPS (extruded polystyrene foam) in three configurations: XPS 300, 500, and 700. The denominations 300/500/700 are indicative of the compressive strength stress (in kPa) at 10% deformation, measured following DIN EN 826 [17,18]. Table 1 provides an overview of the main material properties provided by the supplier [19].

2.2. Measurement setup

Three measurement setups were built to experimentally determine the long-term creep behavior of XPS. Two setups were operated at constant temperature (40/60 °C), whereas the third setup was operated

Table 1

Thermophysical properties of the three types of XPS investigated in this work. Creep strength is given at room temperature. Values from data sheet [19].

	XPS 300	XPS 500	XPS 700	
Density	34	30	35	kg/m ³
Void fraction	97	97	96	%
Compressive strength at 10% deformation	300	500	700	kPa
Thermal conductivity	33–35	33–35	33–35	mW/m-K
Specific heat capacity	1.40	1.40	1.40	kJ/kg-K
Creep strength (50 years, strain <2%)	130	180	250	kPa

at a varying temperature according to the SIM procedure. A schematic illustration and a photograph of one of the setups are displayed in Fig. 1.

The setups were built based on DIN EN 1606:2012 [20] and adapted for the high-temperature application. The main modifications were the thermal insulation, enclosing the entire measurement chamber, the heating system incorporated inside the chamber, and the temperature resistant equipment required to conduct tests up to 75 °C. The referred DIN-standard specifies test equipment and methods for determining the long-term deformation of thermal insulation caused by a defined combination of pressure, temperature, and time. The pressure load is applied to the test specimen in two stages. First, pressure (by mass load) is applied at 23 ± 1 °C. After 48 ± 1 h, the actual testing temperature is applied and kept constant for the time of one-thirtieth of the total period to be extrapolated. For an extrapolated period of 50 years, a minimum of 1.67 years is required. The actual measurement duration of this work was 2.34 years, 20500 h. The first stage at 23 °C serves to define the initial reference state so that the actual creep measurement during the second stage would not be affected by elastic strain. The set-up was placed in a closed room in a basement in order to protect the setups from outside effects like temperature and humidity changes over a long period of time. Although the setups were thermally insulated and heated, the outside environment had to be stable enough for the heating of the setups to properly work and the humidity not to change significantly.

As illustrated in Fig. 1, each setup allows for measuring six samples simultaneously. As no temperature resistant precision length gauge was available, an analog dial gauge allowed for monitoring the compressive deformation of each sample. The dial gauges (MarCator 810a, accuracy ± 0.01 mm, T_{\max} 90 °C) were photographed and evaluated periodically;

this was particularly advantageous in combination with SIM, in which automatic cameras mounted directly in front of the dial gauges allowed for shooting a photo within a fixed short interval. An image recognition algorithm based on the Hough transform [21–23] was applied to accurately identify the gauge reading using the photographs. Following this approach, the accuracy was improved to ± 0.0025 mm.

The additional reference sample (unloaded) served to determine the deformation that results from the thermal expansion of the setup and the sample itself. This measurement was essential for correcting the actual creep measurement in the SIM experiments. In the long-term experiments, the reference sample was used only to monitor the temperature. The reference temperature was measured using type-K thermocouples inserted in the geometric center of the reference sample. The measurements were recorded using a Pico Technology datalogger (model KA028 TC08 USB) [24] with an accuracy of ± 0.025 °C. The temperature in the testing chambers was controlled using electric heaters, circulation fans, a PT100 temperature sensor, measuring air temperature, placed between the samples (see Fig. 1), and a commercial PID temperature controller (FOX-1004) [25]. The relative humidity was measured using a commercial humidity sensor (DKRF410, supplier Driesen + Kern) with a relative accuracy of 2% in the range of 20–80 °C [26].

2.3. Methods

The goal of the numerical and experimental methods applied in this work is to generate a master curve that allows for predicting the long-term creep behavior of the investigated materials. The numerical methods are based on the Boltzmann superposition principle [27] and Arrhenius equation [28]. Creep phenomena are described using the Findley equation [29,30]:

$$\varepsilon_f(t) = a_0 \cdot (t - t_0)^b \quad (1)$$

where ε_f is the time-dependent creep strain, t is time, and a_0 , t_0 and b are fitting parameters.

3. Experimental procedures

Two types of experiments were performed in this study:

1. *Long-term creep test*: Measurements conducted over a long period (months) at constant temperature and pressure.

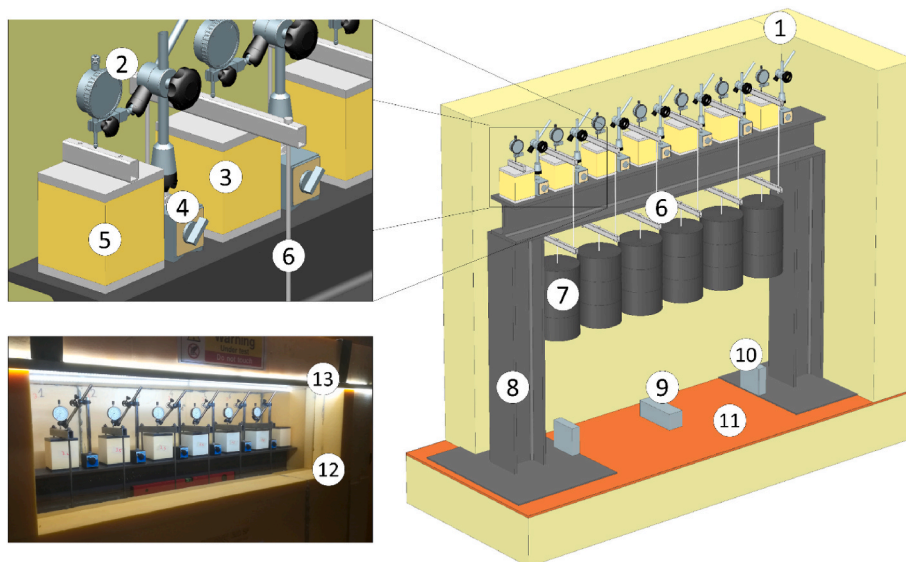


Fig. 1. Setup for measuring creep strain as a function of temperature and time; (1): thermal insulation, (2): high-temperature analog dial gauge, (3): 6 thermal insulation samples ($10 \times 10 \times 10$ cm), (4): fixation of the dial gauge, (5): reference sample (not subjected to stress), (6) steel rod system, (7): weights, (8): steel beams, (9): electric heating element, (10): circulation fans, (11): wooden board, (12): transparent window, (13): removable lamp. Setup dimensions including insulation: width: 200 cm, depth: 80 cm, height: 187 cm.

2. *Accelerated, short-creep test*: Accelerated creep tests with stepwise temperature increase. Calculation of a master curve using SIM [15].

The two methods were applied to allow cross validating the results and ensure the required accuracy in extrapolation.

3.1. Long-term creep

Table 2 displays the main conditions of the two long-term tests with XPS. The same initial conditions and start-up procedures were applied in both tests A and B. In a first step, the chambers were completely closed, and the temperature was set to 60 °C for test A and 40 °C for test B. After 10 h, the targeted temperature level was reached in both chambers, and the temperature was kept constant. The relative humidity was monitored continuously and was confirmed to remain within the measurement range (chamber A: $16.8 \pm 2\%$, chamber B: $6.5 \pm 2\%$). A load of 61.1 kPa was applied to all samples in both chambers following an initial stabilization period characterized by the combined effects of thermal elongation and a small creep deformation caused by the weight of the steel rod system itself (786 g). As a reference, the creep deformation measured between 63 and 256 h from the start of the experiment (i.e., caused by the steel rod system itself) was only 0.07% for test A and 0.05% for test B. The target load was then applied using the cord system by adding the weight, and the conditions remained unaltered for the total duration of the experiment (2.34 years).

To determine the reference height to calculate the creep strain, the same principle as was used as described in section 3.2.1.

The dial gauges were photographed 1 min before applying the weight-load, 1 min after, and afterward following the time scheme described in DIN EN 1606:2012 [20]. After one year, the measurement was recorded every 2 months. The creep data were used to fit parameters using the Findley approach described by Eq. (1) [31].

3.2. Stepped isothermal method (SIM)

SIM was chosen as the method to perform the accelerated experiments. One of the main advantages of SIM is that only one experiment is required to generate the master curve. This is particularly convenient compared with TTSP, which requires multiple samples to ensure the adequate reproducibility of the measurements. In the SIM experiments, the pressure load was varied between 61.1 and 200 kPa. The weight applied to the samples was fixed at 62.3 kg for all experiments. The applied load was changed between experiments by varying the cross-sectional area of the samples. The height of the sample remained unchanged.

For better understanding, some results are presented in this section. The data presented in this section correspond to experiment 5 in Table 4, which was conducted with XPS 700, a load of 61.1 kPa, and four isothermal steps applied over the course of 56 days. The core idea of SIM is based on a temperature-driven acceleration of the creep strain of thermoplastic materials [13,15]. The pressure is kept constant, while the temperature is increased stepwise. According to the Boltzmann superposition principle [27], the data gained from every isothermal step can be regarded as if they arose from independent experiments. In combination, the data of each step represents a constituent part of a master curve, which finally describes the creep behavior over an extended (extrapolated) period. The Boltzmann superposition principle applies to only limited strains undergone by thin samples, given that its foundations are based on linear viscoelastic behavior [32]. However, previous

Table 2
Long-term creep tests.

test	material	Pressure (kPa)	Temperature (°C)	Duration (years)
A	XPS 700	61.1	40	2.34
B	XPS 700	61.1	60	2.34

Table 3
Duration and temperature per step for the SIM.

Step	Duration (h)	Temperature (°C)
1	337	27
2	336	45
3	336	65
4	335	75

Table 4
All SIM tests conducted in this study.

Test N#	Material	Pressure [kPa]	Temperatures [°C]	Duration [days]
1	XPS 300	61.1	30, 40, 50, 60, 70	40
2	XPS 500	61.1	25, 45, 65, 75	56
3	XPS 500	100	30, 40, 50, 60, 70	43
4	XPS 500	200	30, 40, 50, 60, 70	35
5	XPS 700	61.1	25, 45, 65, 75	56
6	XPS 700	100	30, 40, 50, 60, 70	43
7	XPS 700	200	30, 40, 50, 60, 70	35

evaluations of the validity of SIM for geosynthetics have demonstrated that an extension of this rule to larger strains is suitable for design purposes [9]. This rule is further stretched in this study by using samples whose ratio of thickness to cross-sectional area is about one. Another novelty of the procedure described in this work is that the duration and extent of the temperature steps can be adjusted individually for each step. This simplifies the experimental procedure and increases its flexibility. This is achieved by using fitting methods in all post-processing steps, as described in Sections 3.5.3 and 3.5.4.

3.2.1. Experimental run description

The initial height ($h_{0,i}$) of every sample i was measured manually using a digital caliper with an accuracy of ± 0.01 mm. After the samples were placed in the measurement setup, the initial gauge values ($d_{0,i}$) were logged. Afterward, the stress of 61.1 kPa was applied. Due to the applied stress, the dial gauges indicated elastic strain. The initial elastic strain was subtracted. The gauge value measured 1 min after applying the load ($d_{1,i}$) was used as the basis for calculating the reference height $h_{r,i}$:

$$h_{r,i} = h_{0,i} - (d_{0,i} - d_{1,i}) \quad (2)$$

The time-dependent creep strain $\varepsilon_{s,i}(t)$ of every sample was then calculated using the gauge values $d_i(t)$:

$$\varepsilon_{s,i}(t) = \frac{d_{1,i} - d_i(t)}{h_{r,i}} \quad (3)$$

The experiment, described in Table 3, was conducted over four sequential steps and lasted 1344 h in total. The time step and temperature increase per step were chosen based on the experience that large temperature steps are favorable [15]. The total temperature difference of 50 K was divided into four steps to achieve an acceptable ratio between the number of steps and the relevant temperature increase per step. The duration of the steps was decided on the fact that the longer the duration of one step, the more creep strain will occur, thereby causing the calculated timeframe for a master curve to be longer.

Between steps, the temperature increased by 20 K and stabilized without overshooting after 7 h, while for the 10 K increase in the last step, it stabilized after 5 h. This timeframe is 2% of the time of each step and should therefore have no relevant influence on the calculated mean temperature per step.

3.2.2. Thermal strain correction

The measured data were preprocessed to eliminate the effects of the thermal strain of the sample and measurement setup. The goal was to obtain a mean creep curve $\varepsilon_{mc}(t)$ of three identical samples without

thermal strains of the measurement setup and sample itself during temperature switch. The vertical shift (change in height) from the thermal strain between two steps is higher than the creep strain in the former step. Hence, $\varepsilon_{mc}(t)$ is calculated as follows:

$$\varepsilon_{mc}(t) = \frac{\sum \varepsilon_{s,i}(t)}{i} + \varepsilon_0(t) \quad (4)$$

where $\varepsilon_0(t)$ represents the sum of all effects of the thermal strain caused by temperature switch of the measurement setup and the sample, and $\varepsilon_{s,i}(t)$ is the compressive strain measured with sample i . In Fig. 2, the solid blue curve indicates the raw measurement of thermal strain $\varepsilon_0(t)$ using a reference sample (see Fig. 1). After the thermal expansion at the beginning of each step, there is a small creep strain of 0.1–0.2% (depending on the step) visible. This can be explained by the fact that the sample was loaded with 786 g because of the metal plate and distance holder (see Fig. 1). Moreover, because of XPS's thermoplastic nature, some creep deformation was expected without loading. However, with the method described before the effects aside of the long-term creep could be eliminated.

With means of a derivative dT/dt the exact points in time regarding abrupt temperature change can be determined. Indicated as black dashed lines around 340 h, 670 h, 1010 h and 1350 h in Fig. 3.

The individual isothermal steps are determined by separating the creep strain data at the location of the identified peaks. The mean temperatures of each curve $T_{mean,i}$ are used as the reference temperatures of each curve section. The reference temperature for the SIM is the reference temperature of the first curve ($T_{mean,1} = 27^\circ\text{C}$).

The creep data $\varepsilon_{mc,i}(t)$ of each step is treated as an individual experiment at the corresponding temperature $T_{mean,i}$. For each step, the Findley constants $a_{0,i}$, $t_{0,i}$, and b_i in Eq. (1) are determined using a nonlinear least-squares fit procedure. The results of this procedure are illustrated in Fig. 3.

3.2.3. Stepwise time shifts and horizontal stretch

At least two time-shifts are required as part of the SIM. The Findley fits lead to lower strain values at the beginning of each temperature step. Therefore, two different. First, all but the first curve (corresponding to the first temperature step) are shifted to the left to ensure that the starting point of each (Findley fit) curve does not exhibit a lower creep strain than any point in the previous curves. This is illustrated in Fig. 4 as shift 1 and is mathematically described as follows: $\varepsilon_{2,s}$ is the minimum value of the curve $\varepsilon_2(t)$, $t_{c,2}$ is the time at which the curve of $\varepsilon_{2,s}$ is considered to start, and $t_{c,1}$ is the time where $\varepsilon_{2,s} = \varepsilon_1(t)$. The curve is then horizontally shifted by $t_{c,2} - t_{c,1}$ and is equal to $\varepsilon_{2,1}(t)$:

$$\varepsilon_{2,1}(t) = \varepsilon_2(t) - (t_{c,2} - t_{c,1}) \quad (5)$$

The second shift accounts for the fact that all creep curves in the several steps are evaluated as individual experiments. The nominal start time (common virtual start time $t_{v0,1}$) of each curve must be determined and moved towards $t = 0$. In doing so, they can be stretched by the factors a_{T_i} to determine a master curve as if they were curves measured using TTSP [33] This is illustrated as horizontal stretch in Fig. 4.

To determine the virtual start time $t_{v0,1}$, several methods can be applied. A graphical approach is proposed in [33], whereas in [15] the virtual start time is computed iteratively for every step. Beginning with an arbitrary value for the virtual start time, the value is changed iteratively until the gradient at the beginning of a temperature step matches the gradient at the end of the previous step. In [14], the virtual start time is determined by a numerical method.

Since the method presented in this work uses a purely analytical approach by fitting the initial creep curves, the second horizontal shift and horizontal stretch can be determined with the assumption that $\varepsilon_1(t)$ and $\varepsilon_{2,1}(\alpha \cdot t + t_{v0,1}) = \varepsilon_{2,3}$ have the same value and the same gradient (1. derivation) at the point $(\varepsilon_{2,s}, t_{c,2})$.

Solving the equation system (6) for α and $t_{v0,1}$ yields the second time

shift $t_{v0,1}$ and the factor α for the horizontal stretch.

$$\left| \begin{array}{l} \varepsilon_{2,1}(\alpha \cdot t_{c,2} + t_{v0,1}) = \varepsilon_1(t_{c,2}) \\ \frac{d}{dt} \varepsilon_{2,1}(\alpha \cdot t_{c,2} + t_{v0,1}) = \frac{d}{dt} \varepsilon_1(t_{c,2}) \end{array} \right| \quad (6)$$

Since the procedure must be performed for every curve (step) separately, and in order of sequence, only the second shift factor $t_{v0,1}$ is applied, which leads to the second modified curve via

$$\varepsilon_{2,2}(t) = \varepsilon_{2,1}(t - t_{v0,1}) \quad (7)$$

The time shifts of all curves are illustrated in Fig. 5. All but the first step are then stretched horizontally using shift factors a_{T_i} so that the starting gradient equals the ending gradient of the curve before it. This is done stepwise for all but the first curve. $t_{c,3}$ is the time at which the curve $\varepsilon_{2,2}(t)$ is considered to start; respectively, the time value of $\varepsilon_{2,2}(t)$ is the minimum of the curve.

The shift factor $a_{T,1}$ is calculated as

$$a_{T1} = \frac{t_{c,1}}{t_{c,3}} \quad (8)$$

The shifted curve $\varepsilon_{2,3}(t)$ is calculated as

$$\varepsilon_{2,3}(t) = \varepsilon_{2,2}(t) \cdot a_{T,1} \quad (9)$$

This method is then applied to all curves $\varepsilon_{i,2}$, as illustrated in Fig. 6.

The sequence of the stretched curves $\sum \varepsilon_{i,3}(t)$ results in a master curve equivalent to one single long-time measurement. The reference curve, in this case the curve of step one, sets the reference temperature for the master curve. Finally, the sum of the stretched curves is fitted using the Findley approach with a nonlinear least-squares solver (see Fig. 6) to generate a master curve $\varepsilon_{mas}(t)$ with the parameters $a_{0,i}$, $t_{0,i}$, and b_i in Eq. (1)

In order to determine the accuracy of the applied process, an Arrhenius plot is recommended in [15]. The small average of all deviations of <3.5% from linear regression of the shift factors $a_{T,i}$ as displayed in the Arrhenius plot in Fig. 7 indicates a precise measurement and fitting method.

3.2.4. SIM for all reference temperatures

In the method described in Section 3.2.3, the reference temperature is the mean temperature of the first section $T_{mean,1}$. Using every step but the last as the starting point and its reference temperature $T_{mean,2 - (n-1)}$ generates an array of curves with individual reference temperatures $T_{mean,i}$ using data from one experiment. An example of the determination of a master curve with the reference temperature of 65°C using the method described in Section 3.2.3 is illustrated in Fig. 8 (corresponding to Fig. 5) and Fig. 9 (corresponding to Fig. 6).

3.2.5. Determination of a time- and temperature-dependent model for creep strain, fitting method 1: "modified Maxwell model stepwise"

Using the arrays of curves determined in Section 3.2.4, a general time- and temperature-dependent material model for creep strain is fitted using two approaches.

Instead of the Findley equation, a modified Maxwell model [34] is applied. Using equation (10), parameters a_0 , m , q , and c are fitted to the curve with the highest reference temperature. $T_{ref,n}$ ¹

$$\varepsilon_{T_{ref,n}}(t, T) = a_0 \cdot t^{(m \cdot T)} \cdot e^{\left(\frac{-q}{T}\right)} + c \quad (10)$$

In a second step, all remaining curves $\varepsilon_{T_{ref,i}}$ at lower temperatures are fitted, keeping parameters a_0 , m , and c fixed and only q_i varied using equation.

¹ Using the curve with the lowest reference temperature results in worse fitting quality overall.

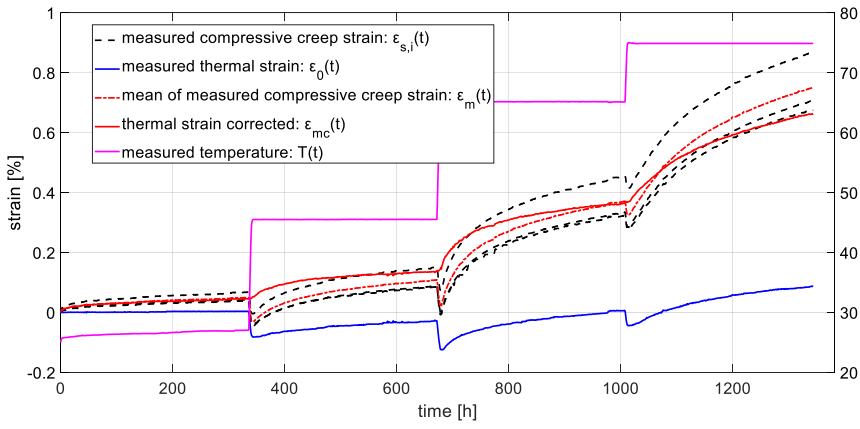


Fig. 2. Measured data obtained with three samples of XPS 700 using a constant compressive load of 61.1 kPa and four different temperature steps. Dashed black curves: strain measured for the three samples $\epsilon_{s,i}(t)$; dashed red curve: arithmetic mean of the three measured strain curves $\epsilon_m(t)$; solid blue curve: thermal strain measured with the reference (unloaded) sample $\epsilon_0(t)$ (see Fig. 1); solid red curve: mean strain corrected for thermal deformation $\epsilon_{mc}(t)$; solid purple curve: measured temperature in the set-up. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

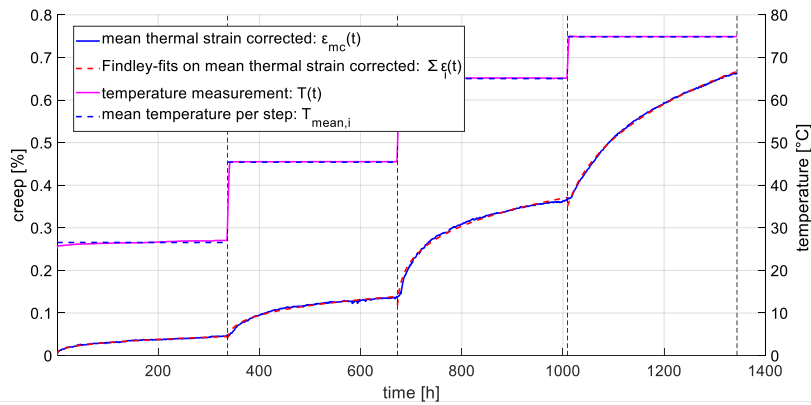


Fig. 3. Corrected, fitted mean measurement data and temperature profile. $\epsilon_{mc}(t)$ is the corrected mean thermal strain calculated using equation (4); $\Sigma\epsilon_i(t)$ are the Findley fits fitted from the mean thermal strain corrected $\epsilon_{mc}(t)$ curve; $T(t)$ is the measured temperature; $T_{mean,i}$ is the mean temperature per step derived from $T(t)$; $dT(t)/dt$ is the derivative of $T(t)$, where the peaks are used to identify the individual steps i indicated as vertical dashed lines.

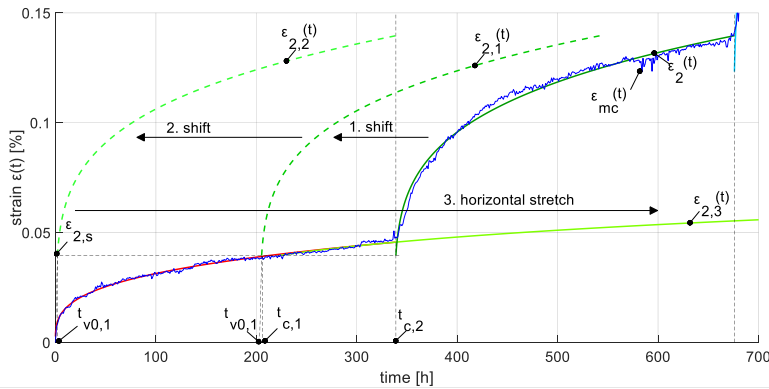


Fig. 4. Schematic example of the SIM for the curve of the second temperature step $\epsilon_2(t)$. The method is sequentially applied to all steps. The three manipulations are the horizontal shift by $t_{c,2}-t_{c,3}$ (1), the horizontal shift by $t_{v0,1}$ (2), and the horizontal stretch by the factor a_{T1} (3) of one creep curve. $\epsilon_{mc}(t)$ is the corrected mean thermal strain calculated using equation (4); $\epsilon_1(t)$ and $\epsilon_2(t)$ are the Findley fits fitted from the mean thermal strain corrected $\epsilon_{mc}(t)$ curve; $\epsilon_{2,1}(t)$ is the curve $\epsilon_2(t)$ after the first shift; $\epsilon_{2,2}(t)$ is the curve $\epsilon_{2,1}(t)$ with the subtraction of the virtual start time $t_{v,0}$; $\epsilon_{2,3}(t)$ is the final result of the manipulation: the curve $\epsilon_{2,1}(t)$ stretched by the Factor a_{T1} .

$$\epsilon_{T_{ref,i}}(t, T) = a_0 \cdot t^{(m \cdot T)} \cdot e^{\left(\frac{-q_i}{T}\right)} + c \quad (11)$$

Using the reference temperature $T_{ref,i}$ of every step, $q(T)$ is fitted with a polynomial regression (see equation (12)) determining the fitting parameters d_0 , d_1 , and d_2 .

$$q(T) = d_0 + d_1 \cdot T + d_2 \cdot T^2 \quad (12)$$

The material model describing temperature-dependent creep by a constant strain using method 1 equals

$$\epsilon_{mat1}(t, T) = a_0 \cdot t^{m \cdot T} \cdot e^{\frac{-(d_0 + d_1 \cdot T + d_2 \cdot T^2)}{T}} + c \quad (13)$$

3.2.6. Determination of a time- and temperature-dependent model for creep strain, fitting method 2: expanding the findley approach

To fit a time- and temperature-dependent material model with fewer constants than the modified Maxwell model, in fitting approach 2, the Findley equation is modified and expanded with factors accounting for the temperature (see equation (14)).

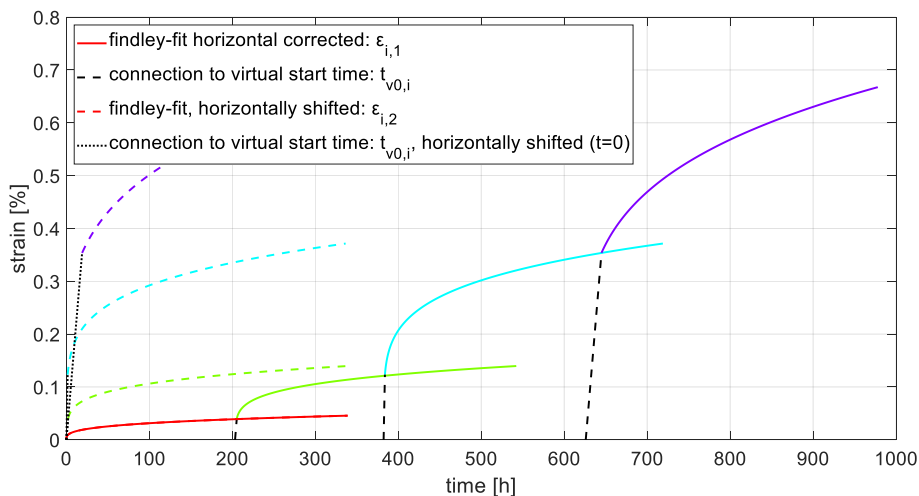


Fig. 5. Determination of $t_{v0,i}$ and horizontal shift of every step by $t_{v0,i}$. Solid curves: horizontally shifted curves $\epsilon_{i,1}(t)$; dashed colored curves: horizontally shifted curves $\epsilon_{i,2}(t)$; dashed black curves: connection of the horizontally shifted curves $\epsilon_{i,1}(t)$ to the respective virtual start time $t_{v0,i}$; dotted black curves: connection of the horizontally shifted curves $\epsilon_{i,2}(t)$ to the virtual start time $t = 0$.

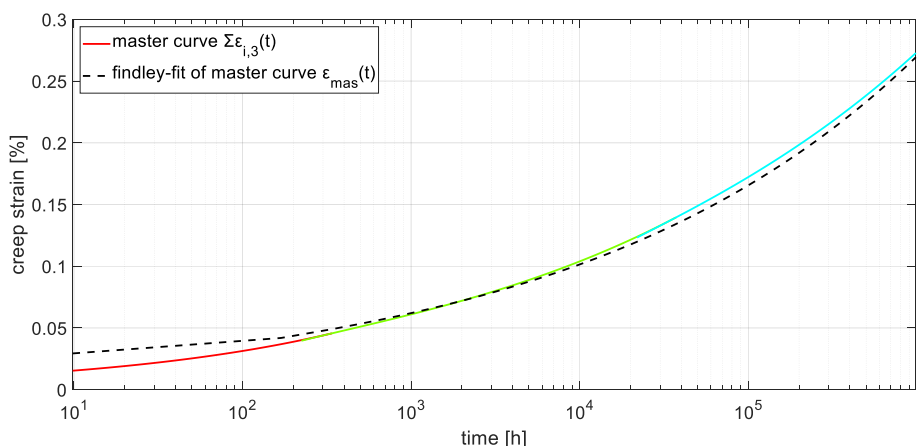


Fig. 6. Example of a master curve (here XPS 700) with a reference temperature of 27 °C. Calculated based on the method described in Section 3.2.4, using the data displayed in Fig. 2. Colored curves: master curve $\sum \epsilon_{i,3}(t)$ (the sum of all shifted and stretched sections of the initial curves $\epsilon_i(t)$); dashed black curve: master curve $\epsilon_{mas}(t)$ fitted from the sum of all stretched curves $\epsilon_{i,3}(t)$ using a nonlinear least-squares solver.

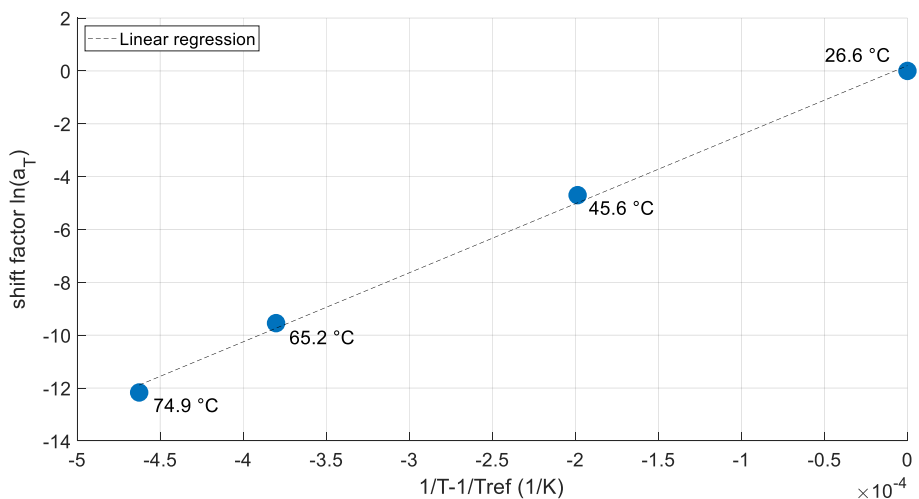


Fig. 7. Arrhenius plot of the shift factors $a_{T,i}$ (blue circles) determined using equation (8) and their linear regression (dashed black curve). The less error in the linear regression, the more accurate the master curve. The error in this example is less than 3.5%. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

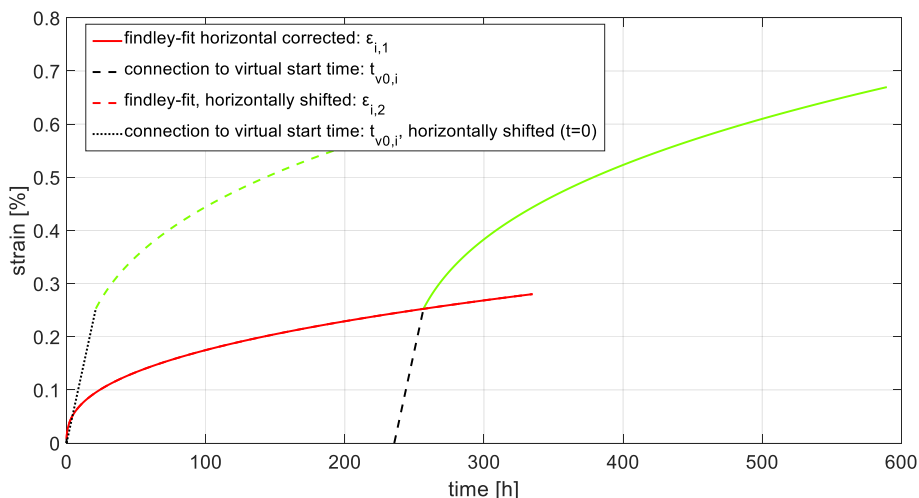


Fig. 8. Example of the determination of creep curve with a reference temperature $T_{ref,3}$ of 65 °C: Determination of $t_{v0,i}$ and horizontal shift of every step by $t_{v0,i}$. Solid green curve: horizontally shifted curve $\epsilon_{2,1}(t)$; dashed green curve: horizontally shifted curve $\epsilon_{2,2}(t)$; dashed black curve: connection of the horizontally shifted curve $\epsilon_{2,1}(t)$ to the respective virtual start time $t_{v0,2}$; dotted black curve: connection of the horizontally shifted curve $\epsilon_{2,2}(t)$ to the virtual start time. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.) $t = 0$.

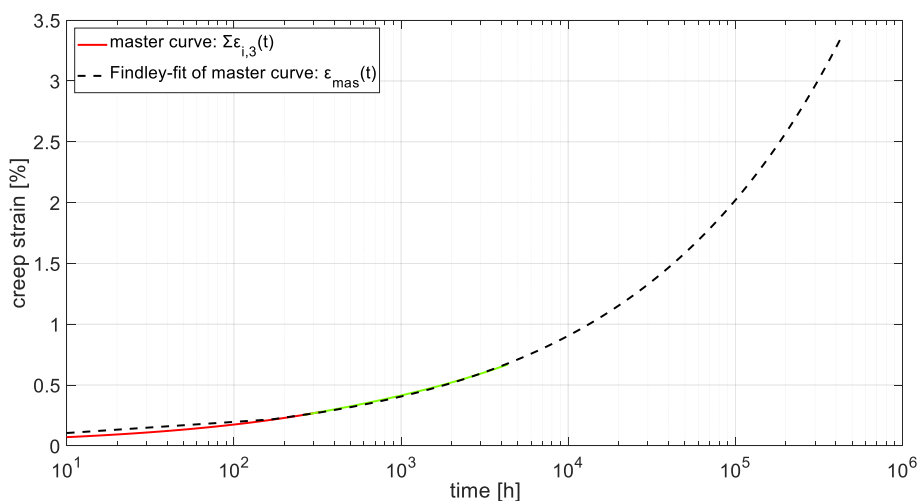


Fig. 9. Example of a master curve with a reference temperature of 65 °C. Calculated based on the method described in Section 3.2.4, using the data displayed in Fig. 2. Colored curves: stitched master curve $\sum \epsilon_{i,3}(t)$ (the sum of all shifted and stretched curve sections of the initial curves $\epsilon_i(t)$); dashed black curve: master curve $\epsilon_{mas}(t)$ fitted from the sum of all stretched curves $\epsilon_{i,3}(t)$ using a nonlinear least-squares solver.

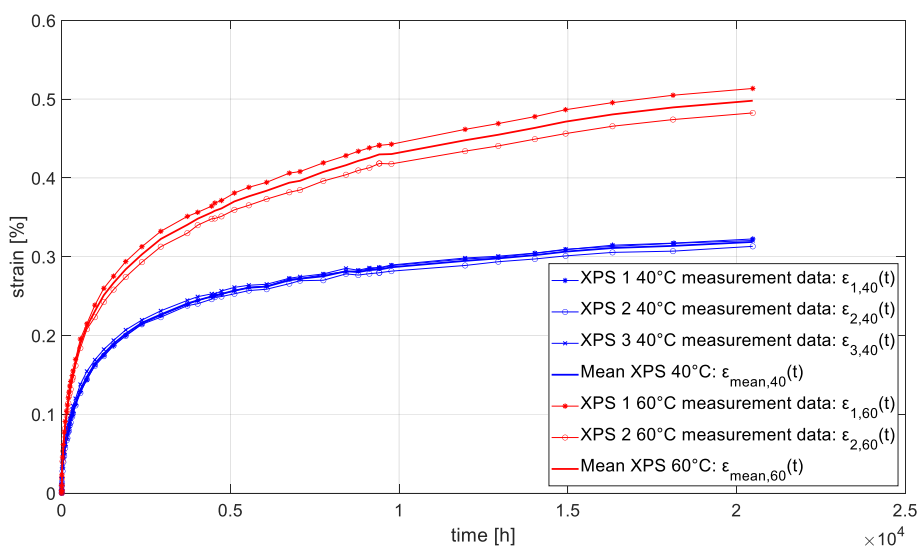


Fig. 10. Measurement and fitted XPS 700 creep curves eq. (1), long-term measurement. $\epsilon_{i,40}(t)$ and $\epsilon_{i,60}(t)$ are raw measurement data from the experiment at 40 °C and 60 °C, respectively; $\epsilon_{mean,40}(t)$ and $\epsilon_{mean,60}(t)$ are the mean curves determined from the measurement data using equation (15).

$$\epsilon_{mat,2}(t, T) = m \cdot t \left(\frac{T - a}{b} \right)^{\frac{1}{n}} \quad (14)$$

Other than in method 1, in this method, the fitting is performed in one step using Non-Linear Least-Squares Minimization [35].

4. Results and discussion

4.1. Long-term creep measurements and fitting

The results for XPS 700 of the long-term measurements with a constant temperature of 40 °C and 60 °C are plotted in Fig. 10. Within the setup with a constant temperature of 60 °C, the measurement of one sample (XPS3) became inaccurate due to a malfunction in the measurement gauge and is neglected for further analysis. The mean curves ("Mean XPS 40 °C" and "Mean XPS 60 °C") are calculated as follows:

$$\epsilon_{mean,T} = \frac{\sum \epsilon_{i,T}(t)}{i} \quad (15)$$

The mean data presented in Fig. 10 was fitted using the Findley approach according to equation (1) using a nonlinear least-squares fit procedure and is extrapolated by a factor of 30 in time (solid black and dashed curves). The creep strain of XPS 700 after 50 years is at 0.95% at a constant load of 61.1 kPa and 60 °C and at 0.49% at 40 °C. The curves for 40 °C and 60 °C are plotted in Fig. 11.

To minimize the time needed for long-time experimental campaigns in the future, a study was conducted to analyze the minimum measurement time needed for accurate results. The longer the measuring time was, the more did the results converge to the final one. In Fig. 12, the calculated creep strain after 50 years as a function of the amount of measurement data used for fitting is plotted. The plot in the figure indicates that, depending on the acceptable error, a much shorter measurement time (about 50%) would be sufficient to achieve the same accuracy in the extrapolated results.

4.2. SIM measurements and fitting

The following tests were performed and evaluated using the SIM to determine the creep characteristics depending on temperature and pressure:

The calculated creep strain after 50 years of all tests listed in Table 4 using the SIM Method as described in chapter 3.2 is illustrated in Fig. 13. The data displays the temperature dependence of the creep behavior, as well as the density differences of the materials XPS 300, 500, 700. Note, that the y-axis is in logarithmic scale.

It is noticeable, that XPS 500 and XPS 700 perform comparable at pressures of 61.1 kPa and 100 kPa. Also, XPS 300 has a significant higher creep strain than the other materials. This indicates that density and porosity changes of the material do not account for direct extrapolation of the creep behavior. The high creep strains of XPS 500 and XPS 700 at 200 kPa indicate the application limits of the material at higher operation temperatures than the material is originally designed to.

The plot on the left side of Fig. 14 is the result of the procedure described in Section 3.2.5. Using SIM-raw data from three steps (the same data as the example data in Section 3.2 or in Table 4 test N# 5), three master curves with corresponding reference temperatures of 27 °C, 45 °C, and 65 °C are determined and plotted (blue, red, yellow). For the array of curves, the fitting method described in Section 3.2.6 is applied (dashed curves). The plot on the right side of Fig. 14, the q-dependence of the three master curves needed for the material model 1 $\epsilon_{mat,1}(t, T)$, is plotted.

Using fitting method 2 (see Section 3.2.7), the material model $\epsilon_{mat,2}(t, T)$ is determined (see Fig. 15), and the results are plotted analog to Fig. 14. As can be seen in a direct comparison of Figs. 13 and 14, both methods can be applied with similar accuracy.

4.3. Comparison of long-term and SIM

To compare the data evaluated from the long-term tests and the SIM tests, in Fig. 16, all data for XPS 700 are plotted. With the determined material models 1 and 2, curves for 40 °C and 60 °C are calculated and plotted.

The comparison between long-term creep data and SIM data indicates a difference in the results, depending on the method. For 40 °C, the deviation of the calculated creep strain after 50 years is 14% (material model 1) and 10% (material model 2). For 60 °C, the deviation of the calculated creep strain after 50 years is 72% (material model 1) and 81% (material model 2). This inaccuracy indicates that the two experiments are not fully comparable, and at least one method is not accurate enough. Nonetheless, the thermo-mechanical properties of the material XPS 700 are highly dependent on the specific batch and can vary depending on production conditions.

5. Conclusions

The long-term creep test indicates that XPS 700 has a creep resistance at 60 °C and 61.1 kPa of less than 1% (long-term) and 2% (SIM) after 50 years. This makes the material suitable for the insulation of STES up to 60 °C. The change of the calculated values in the extrapolated result (Fig. 12) is negligible (less than 0.01%) after a measurement time

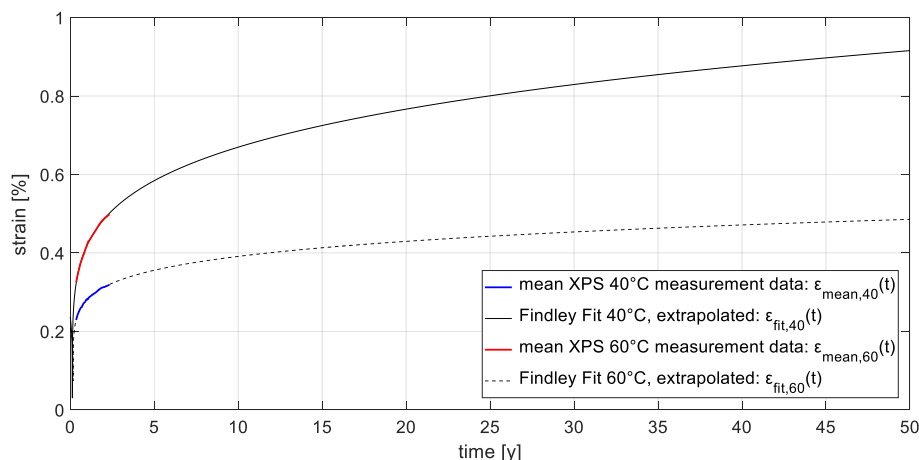


Fig. 11. XPS 700 long-term fitted and 30-times-extrapolated creep curves. $\epsilon_{mean,40}(t)$ (solid blue) and $\epsilon_{mean,60}(t)$ (solid red) are the mean curves determined from the measurement data using equation (15); $\epsilon_{fit,40}(t)$ (dashed black) and $\epsilon_{fit,60}(t)$ (dotted black) are calculated curves determined from the Findley fit of the mean curves for 40 °C and 60 °C, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

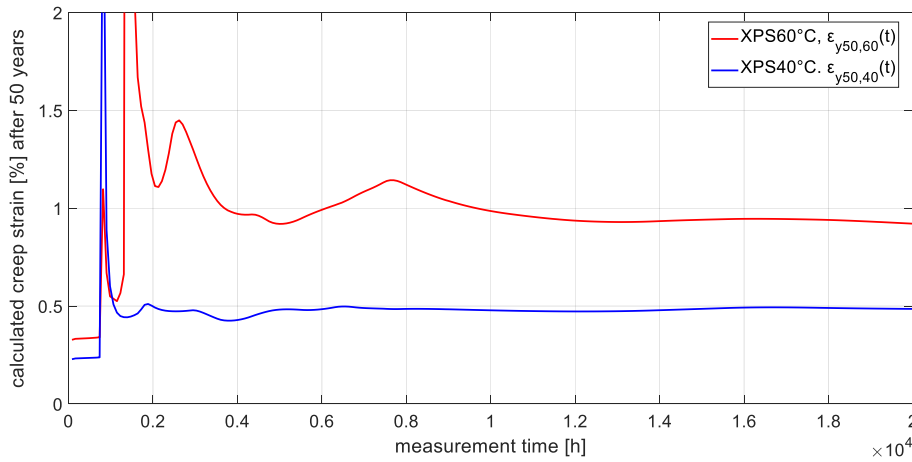


Fig. 12. Creep strain after 50 years, fitted using different measurement data sets. $\epsilon_{y50,40}(t)$ (solid blue) and $\epsilon_{y50,60}(t)$ (solid red) are the calculated creep strains after 50 years as a function of the amount of measurement data used for fitting. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

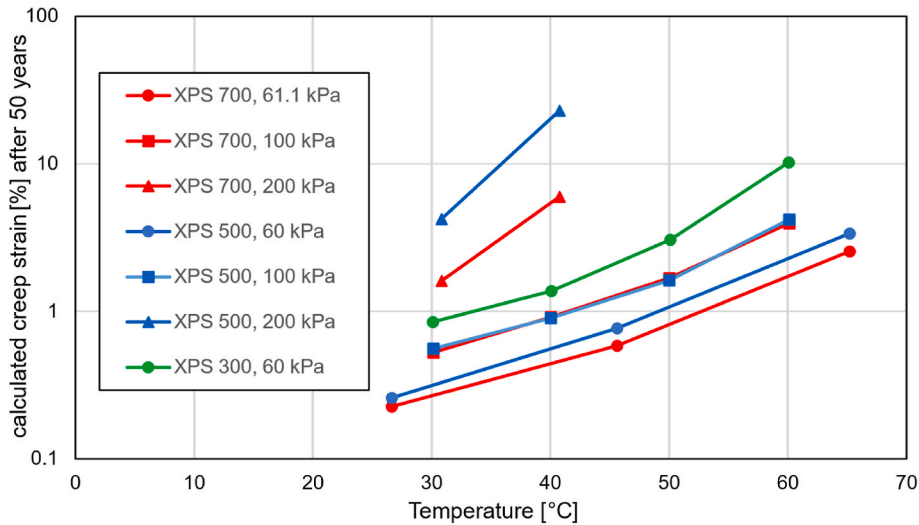


Fig. 13. Calculated creep strain [%] of all SIM tests conducted (listed in Table 4) using the SIM Method as described in chapter 3.2.

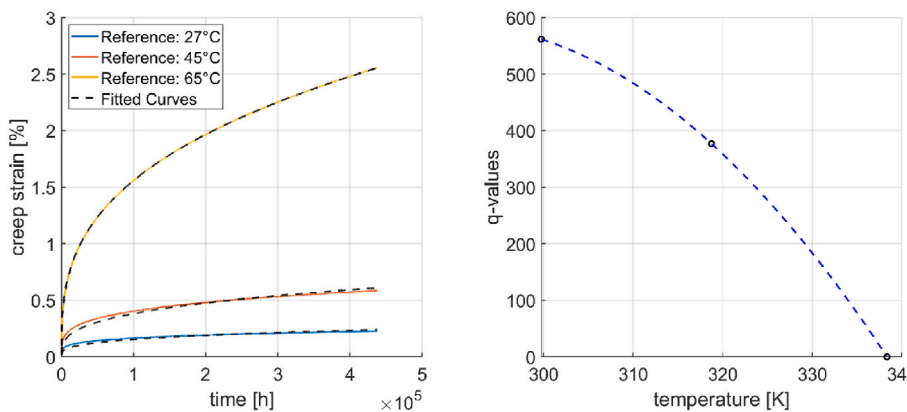


Fig. 14. Example of determination of $\epsilon_{mat-1}(t, T)$ using the procedure described in Section 3.5.5. Left plot: Using SIM-raw data from three steps (the same data as the example data in Section 3.2 or in Table 4 test N# 5), three master curves with corresponding reference temperatures of 27 °C, 45 °C, and 65 °C are determined and plotted (blue, red, yellow). With this data, the material model $\epsilon_{mat-1}(t, T)$ is calculated, and the results are indicated by the corresponding dashed black curves. Right plot: q-values of Eq. (12) are plotted as a function of temperature. The dashed blue curve corresponds to the quadratic fit of the three temperature-dependent values (black circles). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

of 400 days (1.1 years). This finding can be considered in future long-term measurements with similar materials. The results of the SIM measurements indicate that SIM is suitable for samples with a very low height to cross-section ratio of less than one. Additionally, the results indicate that steps of uneven amplitude (ΔT) and duration (Δt) do not

affect the accuracy of the proposed methodology.

Long term experiments were conducted, using the well validated industry standard DIN EN 1606:2012 [20]. The only difference was the elevated temperature of 7% (40 °C) and 14% (60 °C) using the kelvin scale compared to the room temperature of 20 °C that is used in the

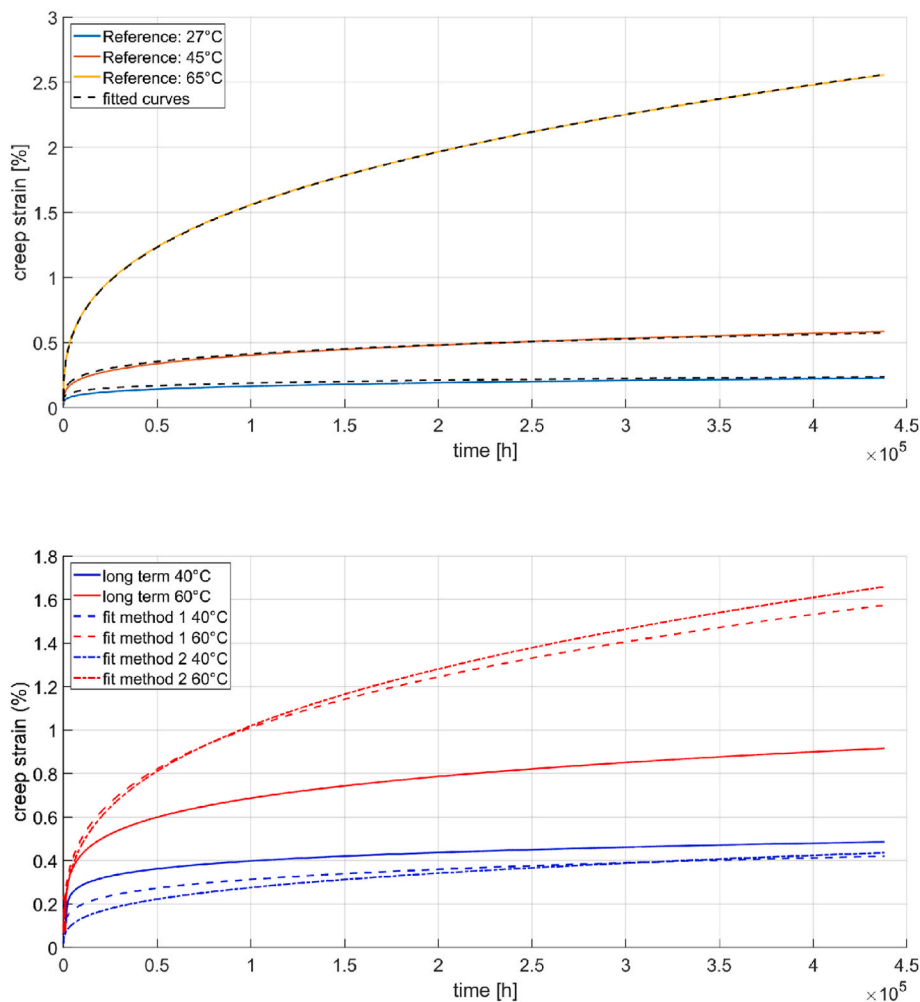


Fig. 15. Example of determination of $\epsilon_{mat,2}(t, T)$ using method 2 described in Section 3.2.7. Using SIM-raw data from three steps (the same data as the example data in Section 3.2 or in Table 4 test N# 5), three master curves with corresponding reference temperatures of 27 °C, 45 °C, and 65 °C are determined and plotted (blue, red, yellow). With this data, the material model $\epsilon_{mat,2}(t, T)$ is calculated, and the results are indicated by the corresponding dashed black curves. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Fig. 16. Comparison of long-term creep fitted data versus SIM fitted data. Solid curves: Fitted and extrapolated long-term creep data of XPS 700 at 40 °C (blue) and 60 °C (red) at 61.1 kPa. Dashed curves: Calculated curves using the material model of $\epsilon_{mat_1}(t, T)$ for XPS 700 at 40 °C (blue) and 60 °C (red), (equation (13)). Dot-dashed curves: Calculated curves using the material model of $\epsilon_{mat_2}(t, T)$ for XPS 700 at 40 °C (blue) and 60 °C (red), (equation (14)). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

industry standard. However, the effects of thermal oxidation of polymers were not taken into consideration in the study and can have an influence on the short term and long-term results. This aspect of the aging process will be analyzed in further studies.

The results indicate that the creep values in both tests are far lower than the targeted 6%. From this point of view, all the tests indicate that the proposed use of the material XPS 700 for STES is applicable. In addition, the small amount of data generated from the experiments (three samples per curve, 2 steps at 65 °C to fit the material models) impacts the accuracy of the results, since the method used relies on precise measurement. In future measurement campaigns, more samples per experiment from the same production batch will be beneficial to obtain more accurate results with less error and insecurity. Nevertheless, the data indicate that SIM works not only on thin materials but also on insulation material with a low height to surface area factor.

The material models $\epsilon_{mat,1}(t, T)$ and $\epsilon_{mat,2}(t, T)$ can be used in simulation to determine the creep behavior of insulation materials by considering the thermal gradient from 60 °C to the wall temperature of 10 °C in the insulation.

Founding

This research was founded by Innosuisse (Innosuisse, 27954.1).

CRediT authorship contribution statement

David Schiffmann: Writing, Conceptualization, Methodology,

Investigation, Visualization, Data curation. **Willy Villasmil:** Writing, Conceptualization, Methodology. **Sebastian Ammann:** Conceptualization, Methodology, Investigation. **Hans Simmler:** Writing – review & editing, Materials Specialist, swisspor Representative. **Jörg Worlitschek:** Writing – review & editing. **Ludger Fischer:** Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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