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K.-U.Bletzinger, E. Oñate, R. Wüchner and C. Lázaro (Eds)

Time-Temperature-Shift (TTS) Principle and Stepped Isothermal Method (SIM) applied on ETFE-Foils

K. Moritz*, J. Hornig**, B. Koenig†

* Taiyo Europe GmbH, Muehlweg 2, 82054 Sauerlach, Germany
e-mail: k.moritz@taiyo-europe.com, web page: https://taiyo-europe.com/de/

** Ingenieurgruppe Bauen, Besselstraße 16, 68219 Mannheim, Germany
e-mail: jonas.hornig@ingenieurgruppe-bauen.de, web page: http://www.ingenieurgruppe-bauen.de

† Benjamin Koenig, Wuerttemberger Str. 21, 89150 Laichingen, Germany
e-mail: benjaminkoenig@live.de

with the kind support of the company se-cover GmbH, Bahnhofstrasse 28, 83119 Obing, Germany
e-mail: info@se-cover.com, web page: https://seele.com/de/

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Summary

The application of the time-temperature shift (TTS) principle on plastic materials, especially viscoelastic polymers, has been known since a long time. This principle describes the influences of the parameters time (embodied by the strain rate) and temperature on the polymer's mechanical behaviour (stress, strain, stiffness, strength values). In case of polymers that allow the application of the TTS both parameters obviously have an inversely proportional influence on the molecular chains and, therefore, on the mechanical properties of the material. The TTS allows the conversion of both parameters, but also the prediction of the mechanical behaviour in ranges that haven't been tested. In case of ETFE-foils the TTS is the reason for their applicability in the building practice that means under snow loads with low strain rates and low temperatures and under wind loads at high strain rates and high temperatures. In both load case scenarios the materials stiffness and strength values keep in acceptable ranges. The TTS principle has been applied on ETFE foils in 2007 the first time [1].

The evaluation of an extensive parameter study in form of uniaxial short-term tensile tests was then used to derive a master curve and to formulate an *Arrhenius*-function for the mathematical description of the TTS of ETFE foils [2]. This function allowed the first time to predict the mechanical behaviour of ETFE foils for combinations of strain rates and temperatures that haven't been investigated before.

With the stepped isotherm method (SIM), an additional approach is available that allows the derivation of the TTS-function of such polymers, too. The method applied is based on uniaxial short-term creep tests at temperatures that increase step by step. From the preceding time-dependent expansion curves a master curve has been derived. Shifting the master curve along the time axis enabled the prediction of the long-term behaviour of the investigated ETFE-foils. The time-dependent strain curves determined with this method have been validated by uniaxial long-term creep tests. [3]

1 INTRODUCTION AND HISTORICAL REVIEW

The fluoropolymer ethylene tetrafluoroethylene (ETFE) was developed in the early 1970s [6]. Under the product name HOSTAFLON by the manufacturer HOECHST, it was initially used as a release film in the industrial sector, for example as a protective layer for radio telescopes, due to many advantageous properties compared to other polymer films [6], before it was applied by architects and civil engineers as a suitable transparent building material for lightweight surface structures in the early 1980s (cf. [1], [7], [8], [9]).

In the architectural sector, however, ETFE films were initially only used for temporary applications in the building envelope. At the 3rd International Solar Forum in Hamburg in 1980, two such transparent constructions, a dome and a sphere, were temporarily erected [6].

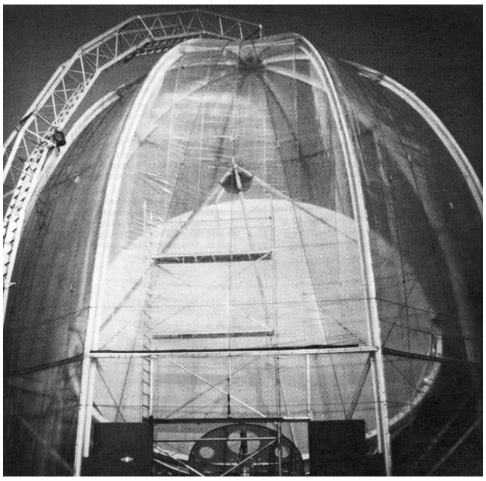

Figure 1: Dome - Temporary ETFE foil construction at the International Solar Forum 1980 in Hamburg [6].

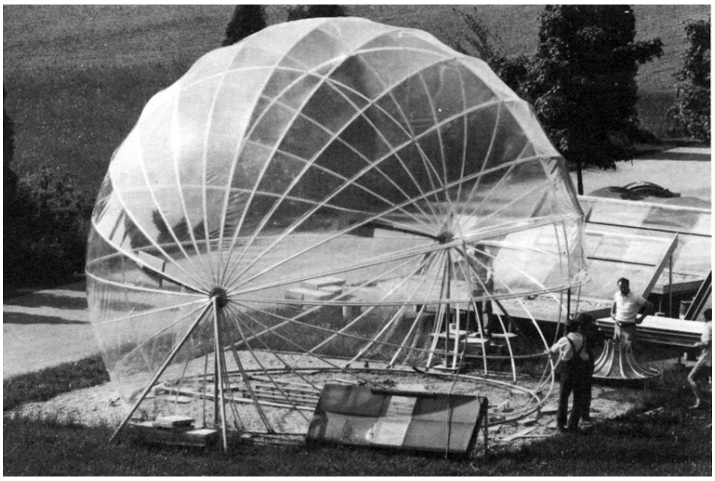

Figure 2: Sphere - Temporary ETFE foil construction at the International Solar Forum 1980 in Hamburg [6].

The first permanent ETFE construction of the building envelope is considered to be the transparent roof of the Mangrove Hall at Burger's Bush in Arnhem, in the Netherlands, from 1982 (cf. [6]). Many projects, which received worldwide recognition, followed, including the 30,000 m² ETFE domes of the Garden of Eden in Cornwall, England, from 2001 or the 64,000 m² ETFE cushion envelope of the Allianz Arena in Fröttmaning near Munich (2004) [7].

Apart from a few prototypes, there was no mechanically prestressed ETFE foil system as a permanent load-bearing element of the building envelope until 1999. This was because ETFE films were considered unsuitable for this type of construction. At the time, it was thought that the film would lose its pretension due to relaxation and creep processes - like many other types of film. But far from it: the canopy of the *Prienavera* swimming pool in Prien at Lake Chiemsee in Germany was built in 1999. It is supposedly the first permanent mechanically prestressed ETFE foil roof in the world (approx. 200 m², membrane statics by TENSYS, Ltd., Bath, UK) [8].

Two years later, in 2001, the roof of the *Infocenter Walchensee power plant* in Kochel was another project using this construction method (ETFE single-layer, mechanically prestressed, approx. 390 m², membrane statics by *E+D Linke & Moritz GbR*) [9].

Figure 3: Warm bath *Prienavera*, ETFE foil cushion as roof covering and transparent canopy (mechanically prestressed single-layer ETFE foil), photo K. Moritz 2001

Figure 4: *Infocenter Walchensee power plant* in Kochel (mechanically prestressed single-layer ETFE film), photo K. Moritz 2001

With the experience gained from these two projects and on the basis of several monoaxial and biaxial short-term tests at different temperatures, long-term tests at room temperature as well as investigations in a wind tunnel, the transparent ring of the grandstand roof of the AWD Arena (today' name HDI Arena) in Hanover was finally constructed in 2004 using this new construction method (ETFE film single-layer, mechanically prestressed, cable-supported, approx. 10,000 m², membrane statics *E+D Linke & Moritz GbR*) [9].

With this impressive project at the latest, it was clear: building with mechanically prestressed ETFE foils is possible if the time-temperature shift of the ETFE co-polymer as well as constructional rules are considered. Today, the roof of the HDI Arena is about 17 years old. Losses of the films pretension due to creep or relaxation are not known. The project was the breakthrough for building with mechanically prestressed ETFE foils, thanks to the time-temperature shift.

Of course, the pretension of ETFE films is temperature-dependent: In summer, the film is soft and its pretension is low; in winter, the film is stiff and its pretension is high. This behaviour is ideal, because under wind loads in summer the film deforms strongly and its stresses are correspondingly lower. In winter, the deformations of the stiff film are low, so the risks of snow or water ponds are reduced. This is why the film was initially described as "intelligent", which sounds absurd for an object. This ideal material behaviour of the ETFE film to summer and winter conditions could only be explained by the time-temperature shift (TTS). It states that the two parameters (increasing) temperature and (increasing) time (here in the form of the decreasing strain rate) have a proportional influence on the mechanical properties of the ETFE film. The molecular chains of the fluoropolymer ETFE obviously do not care whether they are stressed quickly at a high temperature or stressed slowly at a low temperature. Temperature and strain rate could therefore be expressed in terms of one single parameter (modification factor) for ETFE-foils (and for other materials that follow the TTS principle, too) at least up to the viscoelastic strain range. However, no one has taken on this task yet.

Figure 5: AWD-Arena (today's name HDI Arena) in Hanover, 10,000 m² cable-supported ETFE foils, single-layer, mechanically prestressed, Photo K. Moritz, 2004

The stress-strain behaviour of ETFE films can be identical for different combinations of the two parameters temperature and strain rate. The application of the time-temperature shift (TTS) principle makes it possible to apply the same or at least very similar material resistances (creep limit, yield point, breaking point and stiffness) for the decisive load cases wind (fast at high temperatures) and snow (slow at low temperatures). ETFE films do not possess intelligence, but they are ideally suited for use as a load-bearing element in the building envelope (roof, façade) over a wide range of temperatures and strain rates.

The time-temperature displacement principle (TTS) has been known in plastics science for a long time - at the latest with the WLF equation, published by *Williams, Landel* and *Ferry* in 1955 [13] and with the *Arrhenius*-equation from 1889 [18]. The time-temperature shift is also called time-temperature equivalence or time-temperature superposition principle [18].

The time-temperature shift (TTS) principle was first applied to ETFE films by *Moritz* [1]. Based on a big number of uniaxial short-term tensile tests at different temperatures and strain rates, he estimated the magnitude of the TTS for this material [1]: An increase of 10 K in temperature has a similar influence on the stress-strain behaviour than a decrease in the strain-rate of one power of ten. This order of magnitude is consistent with data referring to other viscoelastic polymers found in the relevant literature [17].

The application of the TTS principle suddenly made the good-natured material behaviour of ETFE films at high and low temperatures, which is often observed in practice, plausible. It also forms the basis for the design and safety concept for ETFE foil systems also described in [1], which for the first time comprehensively took into account the influences of temperature and strain rate on stiffness, strengths and modification factors (at that time they were still called reduction factors). The derived design equations in the serviceability limit state (SLS) and in the ultimate limit state (ULS) are presented in the European Design Guide for Tensile Structures published in 2013 (Appendix A5 - Design Recommendations for ETFE-Foil Structures, Chapter A5.4.4) [5]. The design concept described in [1] and [5] is still reliably applied to ETFE foil systems by engineers and companies due to its plausibility and applicability to both limit states (SLS and ULS).

2 EXPERIMENTAL DETERMINATION OF THE TIME-TEMPERATURE-SHIFT OF ETFE-FILM WITH SHORT-TERM-TENSILE-TESTS

2.1 TARGETS

If one thinks of the material resistances at different temperatures and strain rates, for example with snow loads (slow, cold) and wind loads (fast, warm), the time-temperature displacement has a great practical significance for the structural design of ETFE foils.

Basing on the results from *Moritz* 2007 [1] (chapter 1) and *Schiemann* (Load-bearing behaviour of ETFE films under biaxial loading) 2009 [4], *Koenig* 2012 [2] was the first who derived master curves and the time-temperature shift function for ETFE-foils on the basis of systematic investigations in form of hundreds of uniaxial short-term tensile tests under different parameter combinations [2]. The tests were done at a big number of temperature levels and crosshead speeds and - for comparison - with different engineering strain rates and specimen-clamps used. They were carried out in the air-conditioned laboratory of the seele cover GmbH (today se-cover GmbH) on an advanced uniaxial testing machine with temperature cabinet [2], [7]. The test equipment, the test procedure and the results are described in detail in [2].

The comprehensive test program aimed to verify the existing design and safety concept for ETFE films [1] with regard to the estimated time-temperature shift. In addition, the tests were intended to open up the possibility of interpolating and, to a limited extent, extrapolating stress-strain functions and material characteristics. With verified values of the TTS, the safety of the static design can be predicted for extreme climatic regions, from arctic to desert zones, and thus the testing effort for complex long-term tests can be reduced [2], [19], [20].

2.2 STATE OF THE ART

With the help of strain-controlled tensile tests (with constant engineering strain rate) and the time-temperature shift it is possible to approximate the mechanical characteristic values of polymers as a function of time and temperature in a wide strain range [17].

Koenig [2] confirmed the magnitude of the conversion factor from temperature to strain rate for ETFE-foils, stated by *Moritz* (cf. [1], [19], [20]). In the nominal strain range up to 2% he found a similar stress-strain behaviour if the temperature is increased by approx. 10 Kelvin (at constant strain rate) and if the strain rate is decreased by one power of ten (at constant temperature) and vice versa (Figure 6).

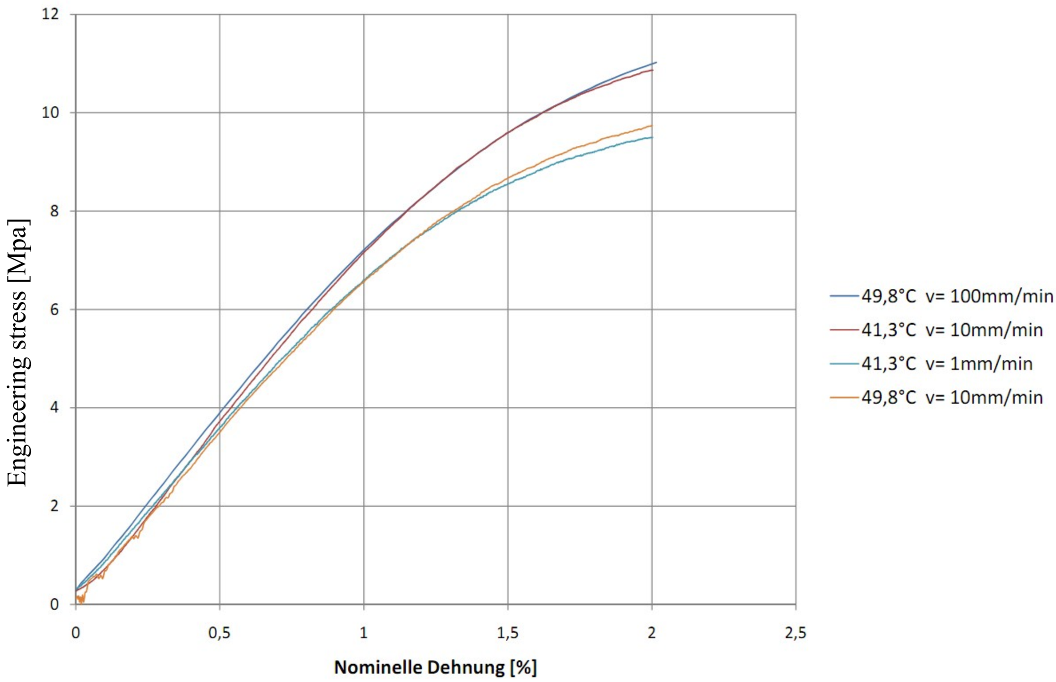

Figure 6: Relationship of changes in temperature and strain rate in case of ETFE-foils (NOWOFOLON ET6235Z) in the nominal strain range ε = 0-2 % [2]

The determination of the time-temperature shift of polymers as a function of temperature, time and stress level can be determined with the help of various tensile tests. According to *Michaeli* et al. [18], it is possible to perform creep tensile tests or strain-controlled tensile tests or tensile tests with constant crosshead speed [2].

The creep tensile test is a test regulated according to DIN EN ISO 899-1 [12]. The method examines the creep and deformation behaviour under long-term static tensile loading. According to the standard, the test duration is at least 1000 to 10000 hours (approx. 1.4 - 14 months). A considerable amount of effort is therefore required to create a series of measurements with the creep tensile test for a reference temperature [2]. For this reason, strain-controlled short-term tensile tests, i.e. with constant strain rate, are preferably used to determine the time-temperature shift with a lower effort. The constancy of the strain rate required for this refers to the specimen or measuring length that changes during the test - in contrast to the constant crosshead speed, which refers to the specimen or measuring length at the start of the test when the specimen is unloaded.

Essentially, creep tensile tests and short-term tensile tests differ in the duration of the test and the type of loading. Results from short-term tensile tests with different temperatures are well suited for determining the TTS factor in order to simulate arbitrary time durations of the properties. The test effort and duration can be kept low here. Measurements with short test durations and high temperatures allow conclusions to be drawn about the mechanical behaviour at lower temperatures and longer test durations.

2.2 TEST PROCEDURE AND TEST RESULTS

The mean value curves (see Annex 2 D of DIN EN 1990 [11]) exemplary shown below refer to four temperature levels. This represents only a small excerpt of the parameter-study systematically carried out and summarised in [2]. The study comprises a total of several hundred short-term tensile tests on various ETFE film products at 7 temperature levels (- 20°C, 0°C, 10°C, 23°C, 30°C, 40°C and 50°C) and 6 constant strain rates (0.01, 0.1, 1, 10, 100 and 1000 mm/min). The test set-up and the test procedure (testing machine (Figure 7), specimen type according to DIN EN 527-1/-3, grips, measuring devices etc.) are described in detail in [2].

Figure 7: Uniaxial testing machine with temperature cabinet, source: seele-cover GmbH (today: se-cover GmbH) [7], cf. [2]

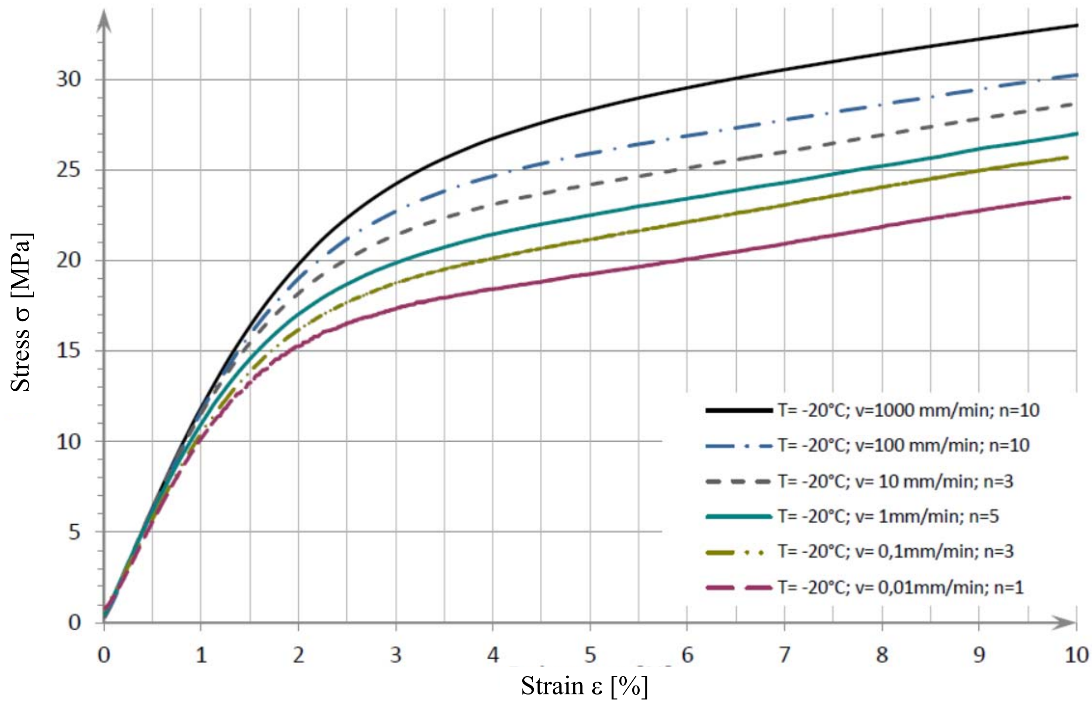

Figure 8: Stress-strain curves (mean values) for T = -20°C [2]

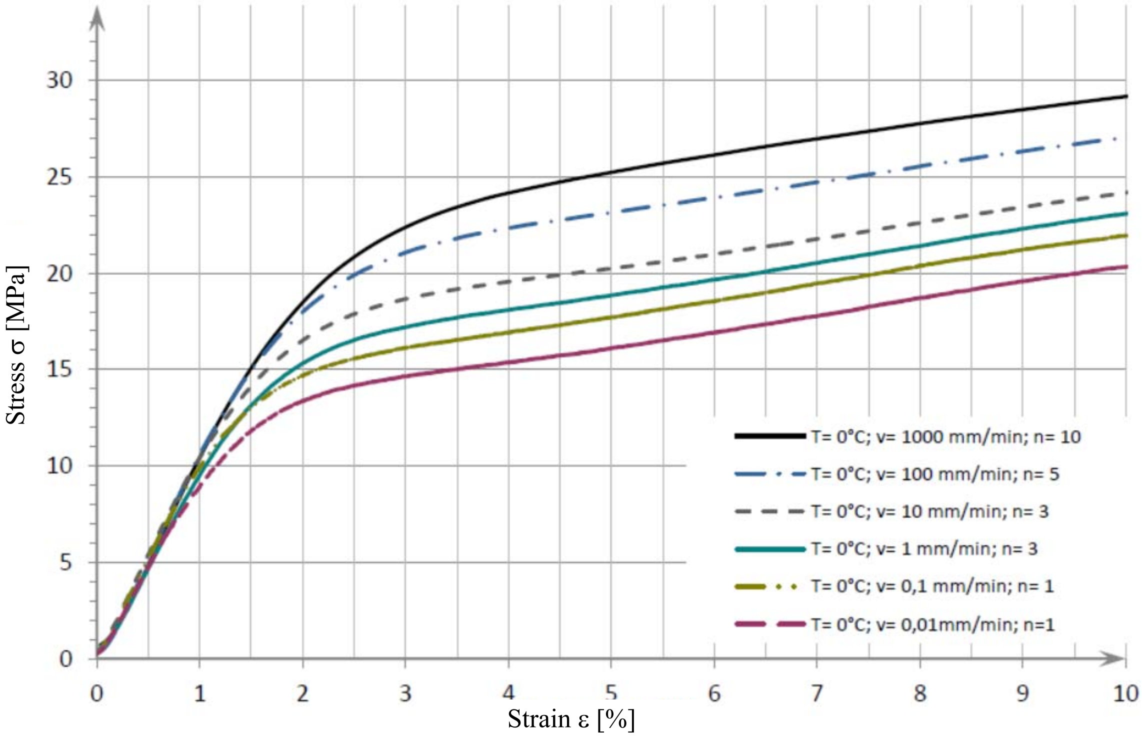

Figure 9: Stress-strain curves (mean values) for T = 0°C [2]

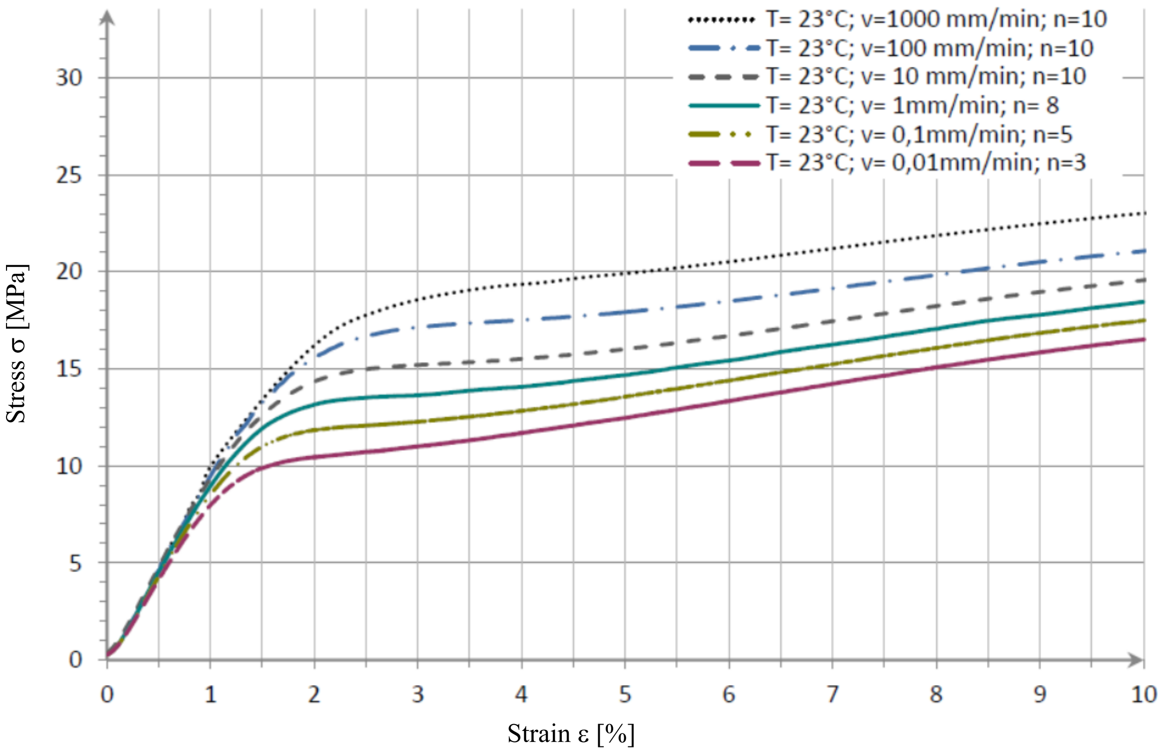

Figure 10: Stress-strain curves (mean values) for T = 23°C [2]

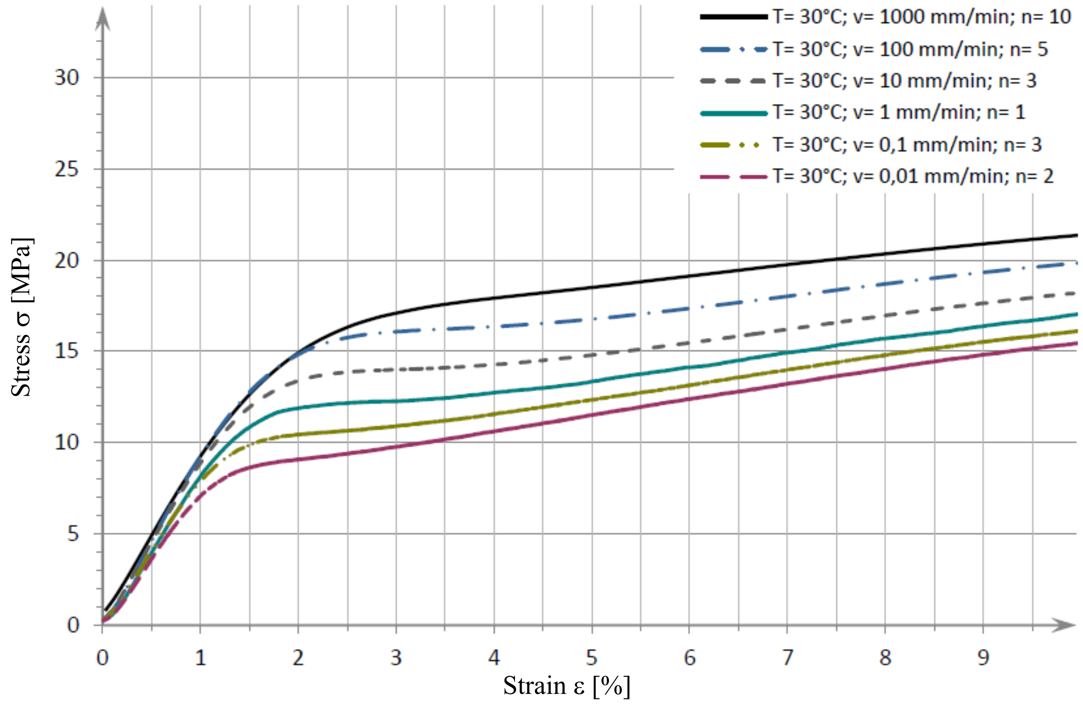

Figure 11: Stress-strain curves (mean values) for T = 30°C [2]

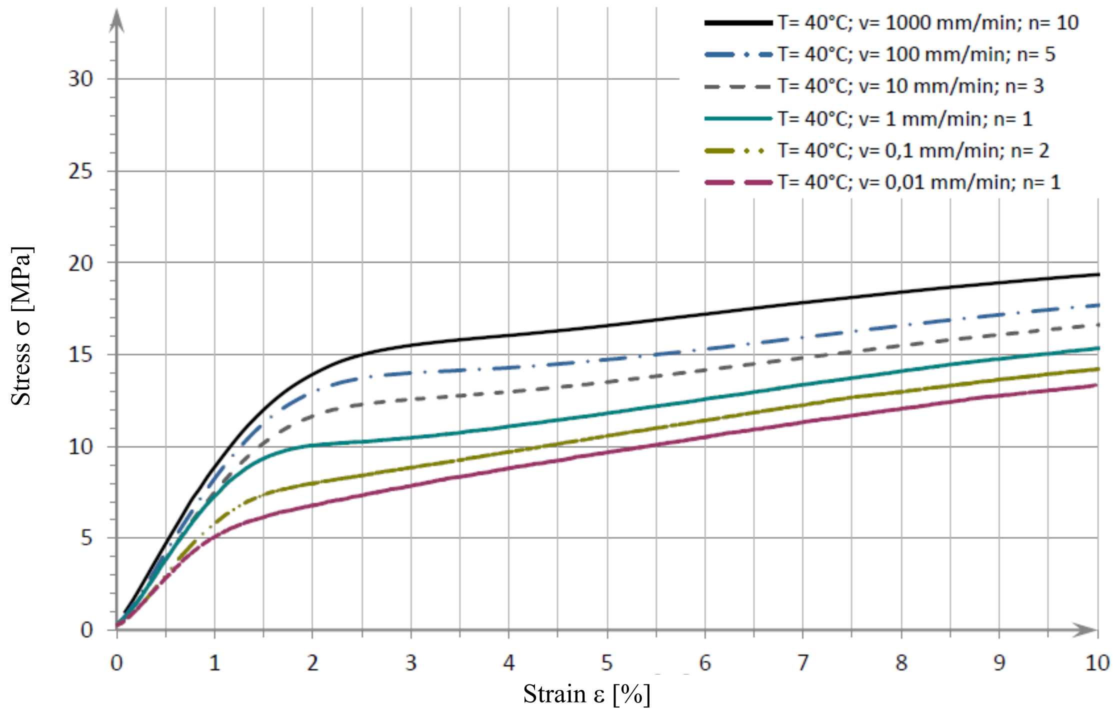

Figure 12: Stress-strain curves (mean values) for T = 40°C [2]

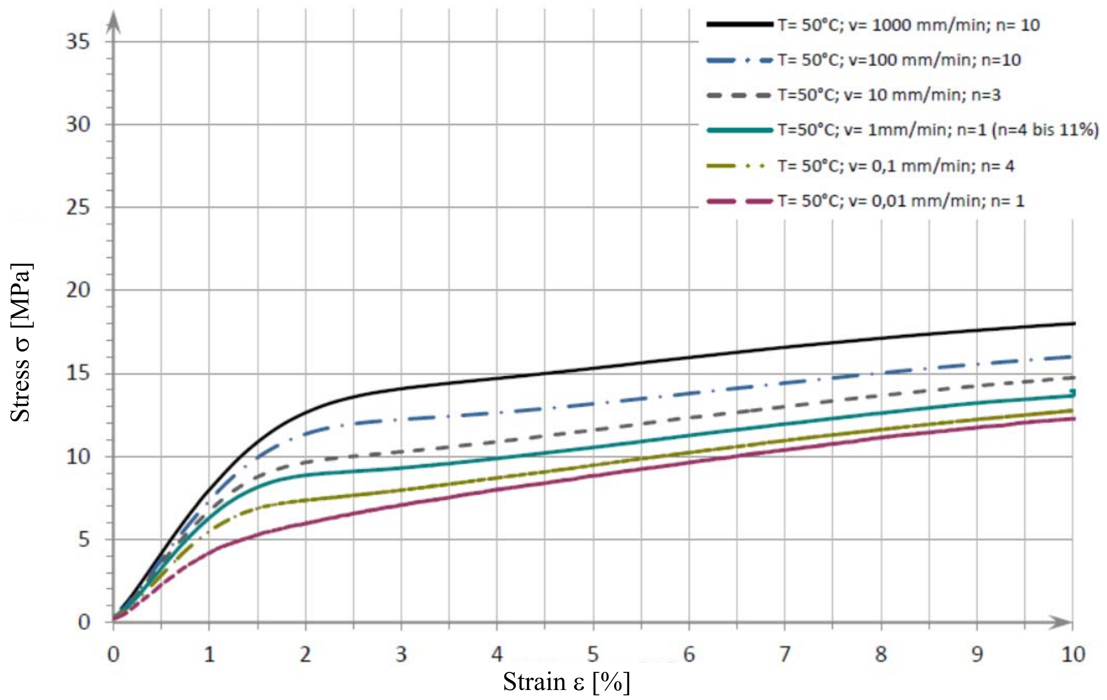

Figure 13: Stress-strain curves (mean values) for T = 50°C [2]

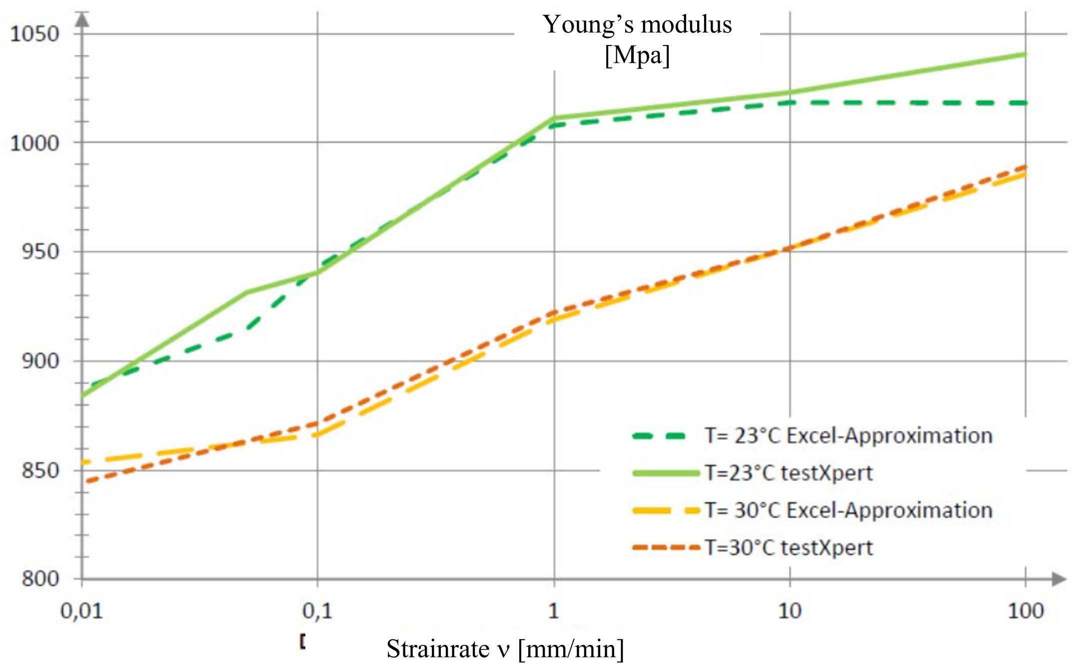

Figure 14: *Young's* modulus (uniaxial) for two temperature levels, plotted against the measured strain rate levels between 0.01 and 100 mm/min, determined by the testing software testXpert as well as calculated from single values with the software EXCEL [2].

A comparison of figures 8 to 13 shows the influence of temperature and strain rate on the stress-strain behaviour of the tested ETFE film (NOWOFLON ET6235Z). The evaluation of the test results also shows that the tensile modulus of elasticity (*Young's* modulus) in the approximately linear elastic range also depends on the two parameters (Figure 14).

It was taken into account that the first yield point also changes as a function of temperature and strain rate (Figure 15). The yield stress observed in uniaxial tests for the product tested here is in the order of magnitude of the stress at 10% strain. This stress value, which is easy to measure in the test, is therefore an indicator of the influence of temperature and strain rate on the area around the first yield point too. The recognisable changes in the slope of the stress-strain curves mean that the linear elastic range has already been exceeded in the short-term tensile test with regard to the stresses and the strains. A description of the possibilities to determine and describe the yield point can be found in [1].

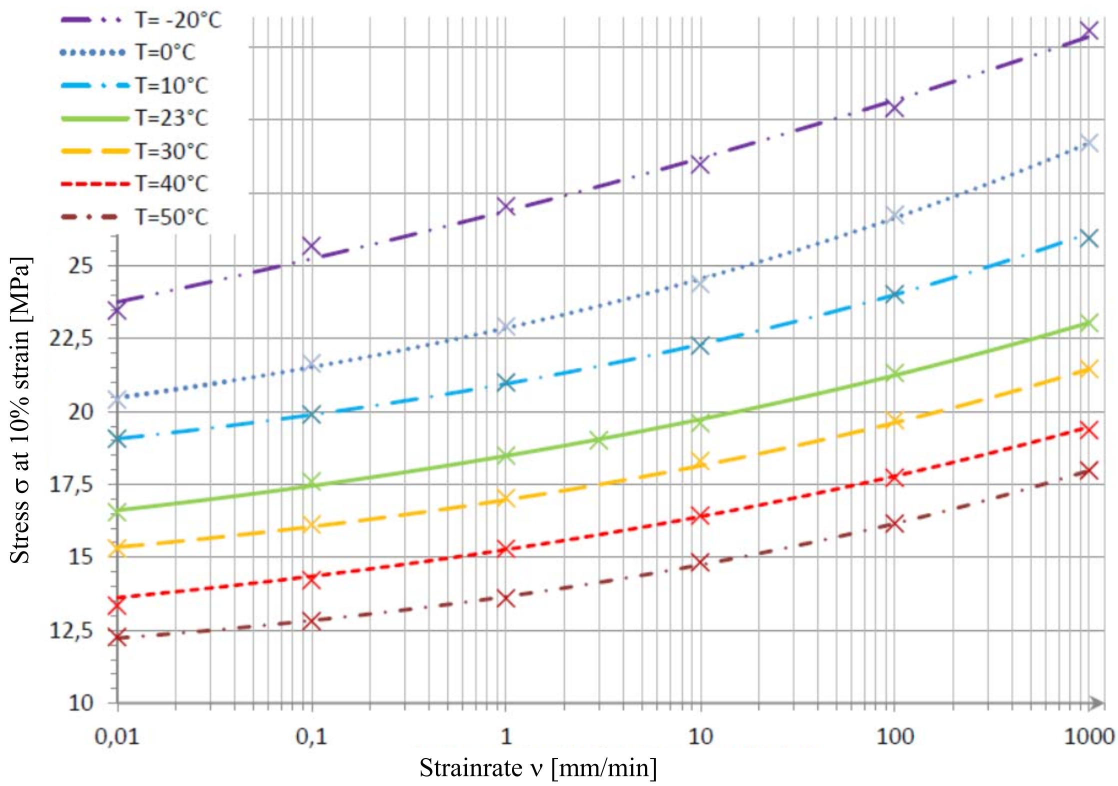

Figure 15: Stress at 10% strain by means of approximation function for the investigated temperature levels logarithmically plotted against the constant strain rate (crosshead speed) between 0.01 and 1000 mm/min [2].

2.3 APPLICATION OF THE ARRHENIUS-EQUATION

According to [13], the approach according to *William, Landel* and *Ferry* is valid for a temperature range of ±50 Kelvin around the glass transition temperature. Since the glass transition range of ETFE is at TG ≅ +130°C and a secondary softening point at TN ≅ -100°C [1], the approach according to *William, Landel* and *Ferry* can only cover temperature ranges that are hardly or only partially relevant for the design of ETFE-foil structures in building. Therefore, in [2] the *Arrhenius*-equation was applied to the stress-strain curves determined in the tests in order to predict curves for low strain rates, for example, which were not tested in the test. The TTS factor was verified by tests at least in the temperature and strain rate field recorded in the tests. The *Arrhenius*-factor can be represented as follows according to [2]:

$$\ln(\mathrm{k}) = \ln(A) - \frac{E_A}{R \cdot T} \tag{1}$$

with:

k	=	*Arrhenius*-factor
A	=	frequency factor
E_A	=	energy content of the reaction system
R	=	general gas constant
T	=	temperature (in Kelvin)

The *Arrhenius*-equation is based on the artificial time lapse of a change brought about by an increase in temperature, for example the ageing of polymers. The application of the equation presupposes various conditions, such as the limitation to temperature-steps of a maximum of 15 Kelvin. The conditions are listed, for example, in [13] and also in [2].

For the application of the *Arrhenius*-equation to TTS, the modified form shown below has become established in polymers technology [20]:

$$\log(a_T) = \log\left(\frac{\dot{\varepsilon}_{ref}}{\dot{\varepsilon}}\right) = k \cdot \left(\frac{1}{T} - \frac{1}{T_{ref}}\right) \tag{2}$$

with:

k	=	*Arrhenius*-factor (also called activation factor)
T	=	temperature (in Kelvin)
T_{ref}	=	reference-temperature (in Kelvin)
$\dot{\varepsilon}$	=	strain rate
$\dot{\varepsilon}_{ref}$	=	strain rate at reference-temperature
a_T	=	Time-Temperature-Shift-Factor

The *Arrhenius*-factor k is usually not constant but a function. In the case of the ETFE films examined, the function is a straight line in logarithmic representation. If the function is derived from experiments, the time-temperature shift factor a_T can be calculated for one parameter as a function of another parameter. The parameters can be, for example, the strain rate, the stress at 10% strain or the modulus of elasticity, each plotted against another parameter like the temperature, for example.

The application of equation (2) to the mean value curves of the stress-strain relationship at a temperature of T = 23°C for different strain rates (Figure 10) yields further curves for the strain rates v = 0.001 and v = 0.0001 m/min (Figure 16), which were subsequently verified with corresponding tests. Source [2] provides the example calculation for this.

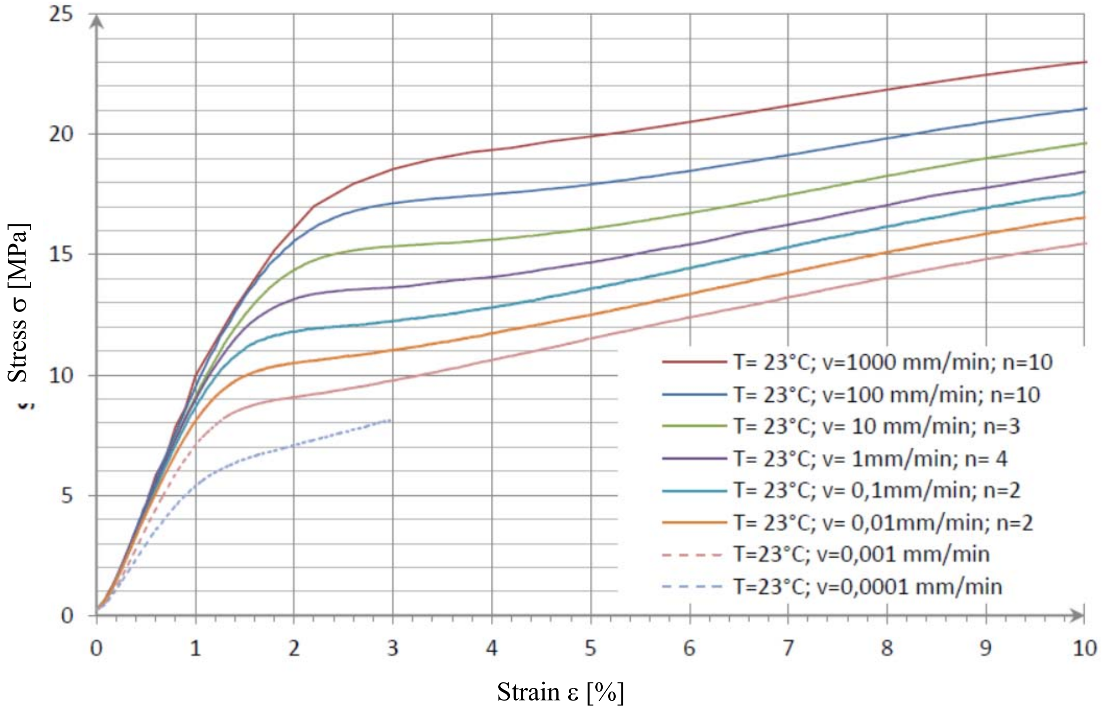

Figure 16: Extension to the stress-strain curves for strain rates v = 0.001 and 0.0001 mm/min (mean value curves) determined by means of *Arrhenius*-function and derived time-temperature shift factors [2].

3 STEPPED ISOTHERM METHOD (SIM) APPLIED ON ETFE-FOILS

Studies on the long-term behaviour of ETFE films have already been carried out sporadically under various aspects. Due to the great effort involved in long-term tests, suitable time-lapse test procedures are of great importance. From this point of view, the SIM has come into focus, which represents a further development of the experimental performance of the tests for the ZTV.

With SIM, individual short-term strain-time-tests with increasing temperature level are combined into one test by *Hornig [3]*. The specimen is subjected to a constant load in a short-term creep test and the temperature is increased after a certain time. The temperature cycles are thus run through coherently like consecutive short-term tensile tests according to the TTS.

A stepped strain-time diagram is thus obtained according to Figure 17. When applying Boltzmann's superposition principle, virtual start times can be determined for the individual increases, as shown in Figure 18. The *Boltzmann* superposition principle states that successive, time-dependent impacts (or changes in impacts) result in the same total result as the individual effects of the impacts (or differences in effects of the impacts) put together. There is, therefore, a linear relationship between impact and effect. It is obvious that this principle can only be applied if a linear viscoelastic behaviour is given.

By shifting the initial time horizontally according to Figure 19, a separate strain-time curve is obtained for each temperature. If the shift is carried out logarithmically on the basis of the *Arrhenius*-equation, the master curve shown in Figure 20 is obtained.

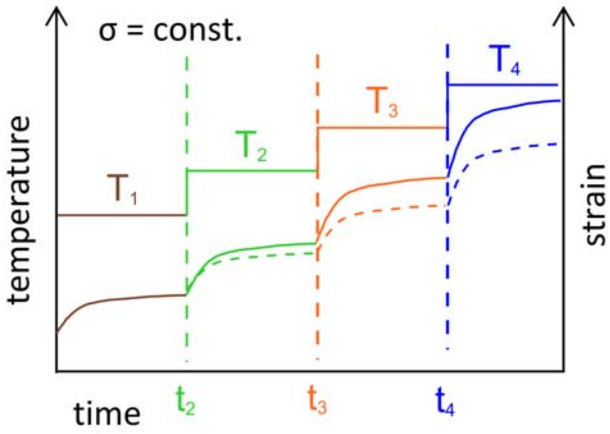

Figure 17: SIM – schematic procedure - step 1: stepped strain-time diagram

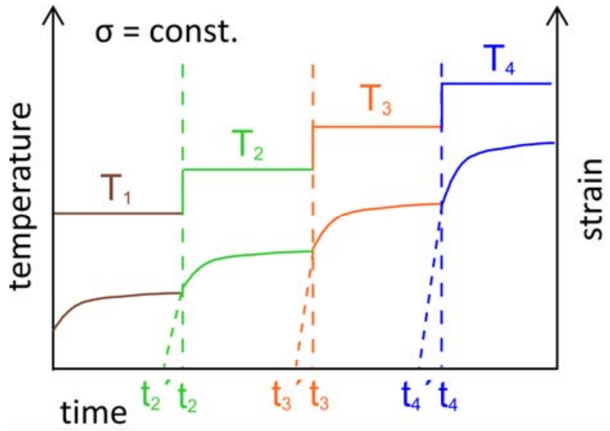

Figure 18: SIM – schematic procedure - step 2: Application of the Boltzmann's superposition principle

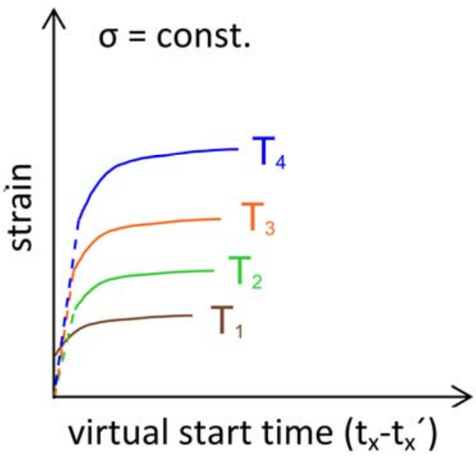

Figure 19: SIM – schematic procedure – step 3: horizontal shift of the initial (start) time

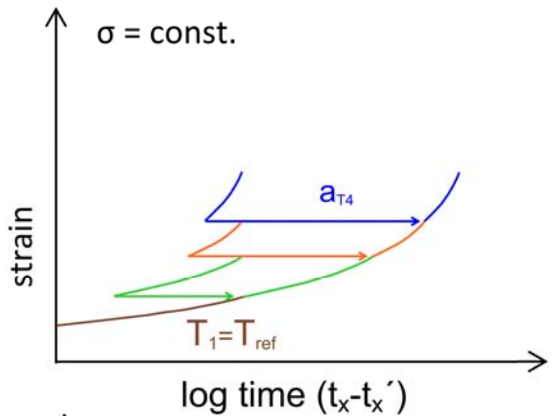

Figure 20: SIM – schematic procedure – step 4: logarithmic shift of the initial (start) time based on the Arrhenius-equation

The SIM method based on time-temperature shift (TTS) is a well-known method for assessing long-term behaviour of geo-synthetics (e.g. made of polypropylene [14] or aramid fibres [15]). The method is regulated in ASTM D6992 - 16 [10].

The SIM was applied here for the first time to ETFE films to investigate long-term behaviour and verified with long-term creep tests. As shown in Table 1, two test series were carried out with defined temperature steps and holding times of the individual steps. For each run, five samples were loaded with three load levels, which approximately represent the elastic range of the ETFE film.

LS2: σ_{LS2} = 5,95 MPa

LS3: σ_{LS3} = 11,91 MPa

LS4: σ_{LS4} = 14,94 MPa

SIM 1 - Temperature Steps with ΔT =16 K (after 5 minutes initial heating)			**SIM 2 – 5 Temperature Steps with ΔT =11 (after 3 minutes initial heating)**		
step [-]	**temperature T [°C]**	**holding time [h:min]**	**step [-]**	**temperature T [°C]**	**holding time [h:min]**
1	23	2:30	**1**	23	2:00
2	39	2:30	**2**	34	2:00
3	55	2:30	**3**	45	2:00
4	71	2:30	**4**	56	2:00
-	-	-	**5**	67	2:00
Σ		**10:00**	Σ		**10:00**

Table 1: Sequence of the two SIM test series - temperatures and holding times [3]

To determine the master curves from the obtained individual curves by means of the *Arrhenius*-equation, the activation energy ΔH is required. In the course of the evaluation, the activation energy was considered as a constant and averaged over the adjustments of the master curves with the long-term tests.

Figures 21-23 show the master curves for the three load levels LS2, LS3 and LS4. The graphs marked "MK LS ..." were each approximated with *Findley's* power function, while the graphs marked "middle LS ..." were averaged from the curves of the long-term (creep) tests.

Load level 2 provides the best compliance with the long-term tests and also the best approximation. A predominantly linear viscoelasticity can be assumed for this load level, as the master curve matches the creep curve qualitatively and quantitatively well. Based on this finding, it can be assumed that the SIM can be applied at least up to a stress of about 6 MPa.

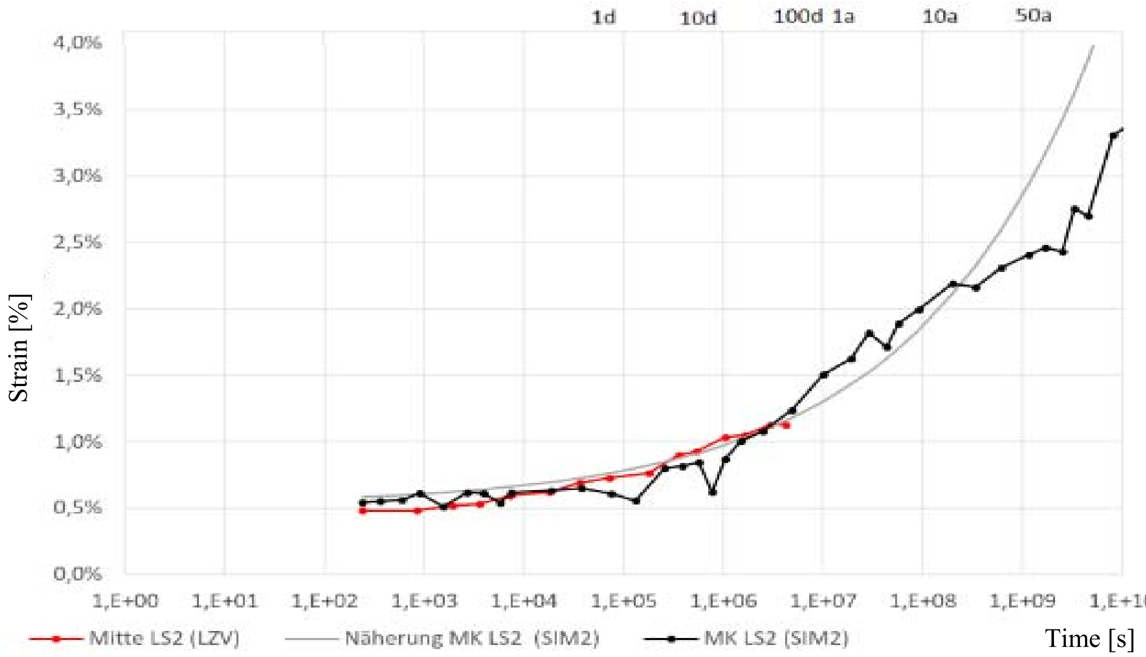

Figure 21 Evaluated curves of load level 2 [3]

With increasing load, the curves show a more homogeneous picture, but deviate significantly from the creep curves in the range of higher temperatures (Figures 22 and 23).

Thus, the master curves all show a progressive course, while the creep curves tend to follow more a degressive course. Although this effect is small for the creep curves and the statement is limited by the test time, this tendency can be observed at the both higher load-levels.

At load level 4, the master curve is similar to that of load level 3 of the same SIM test. The steeper increase in the master curve at higher temperatures or with longer test times is due to the fact that the *Arrhenius*-factor deviates more strongly from the selected constant at higher temperatures and can, therefore, no longer be used. In addition, non-linear viscoelastic or viscoplastic effects have an increasing influence on the strains at high loads or increased temperatures.

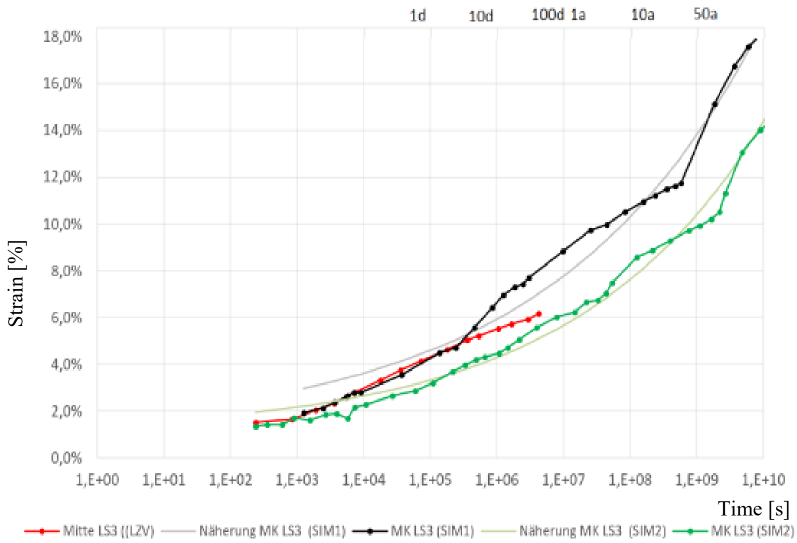

Figure 22: Evaluated curves of load level 3 [3]

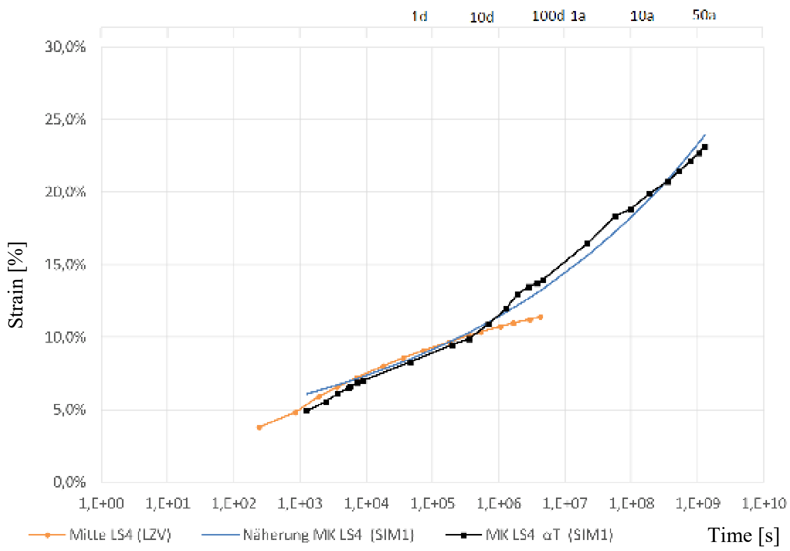

Figure 23: Evaluated curves of load level 4 [3]

4 CONCLUSION

The test results described in the paper in hands show that the time-temperature shift (TTS) of ETFE films can be mapped up to the viscoelastic strain range with the *Arrhenius*-equation. The *Arrhenius*-function and thus also the time-temperature-shift factors can be determined on the basis of short-term tensile tests at different temperatures and strain rates (see chapter 2), but also by application of the Stepped Isotherm Method (SIM) at gradually increasing temperatures (see chapter 3). If the master curves obtained with the SIM are calibrated with the creep curves derived from long-term tests done in parallel [3], both approaches presented in this article deliver a good concordance in the results [3].

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