SILICONE RUBBER IN NUCLEAR POWER PLANTS: PREDICTION OF SERVICE LIFETIME IN MOIST CONDITIONS

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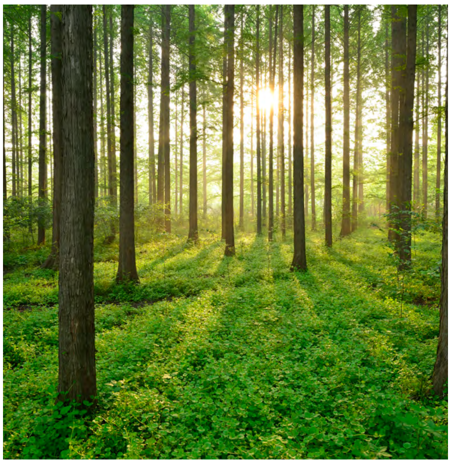
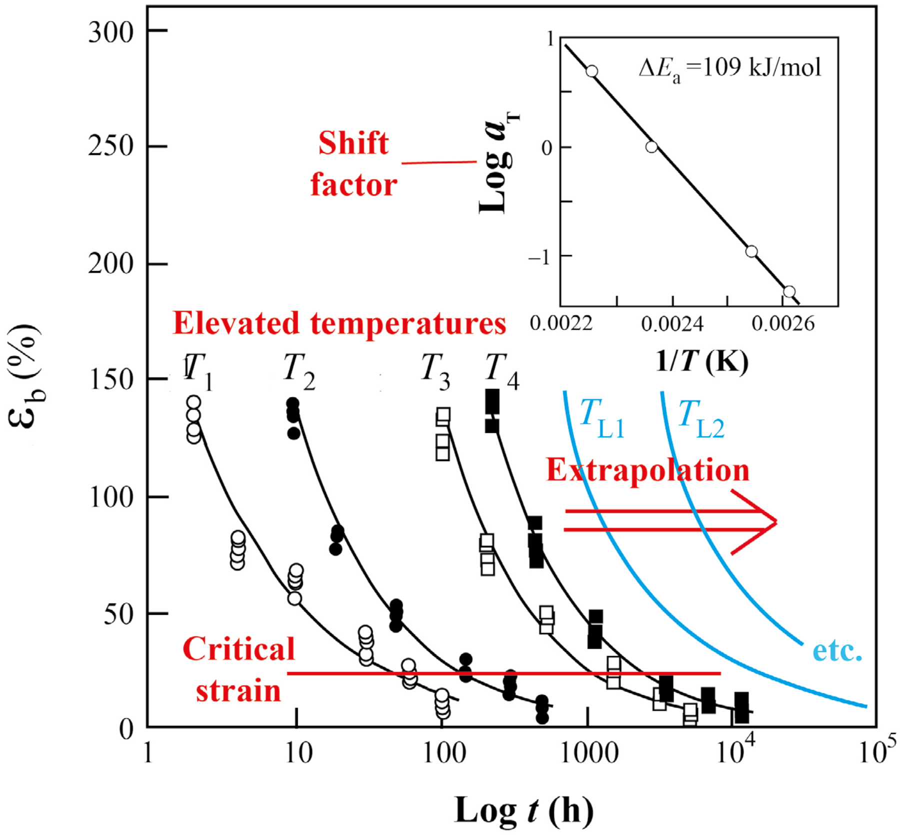


POLYMERIC MATERIALS IN NUCLEAR APPLICATIONS



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Silicone rubber in nuclear power plants: prediction of service lifetime in moist conditions

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Foreword

This report forms the results of a project performed within the Energiforsk Polymeric Materials in Nuclear Applications Program. The Polymers Program aims to increase the knowledge of aspects affecting safety, maintenance and development of components containing polymers in the Nordic nuclear power plants. A part of this is to investigate possibilities to facilitate and simplify the work that is performed in the nuclear business.

The purpose of this study was to find a method for ageing silicone material in humid environments, since experience has shown that it is difficult to age silicone seals, especially in humid environments. The combination of moisture and high temperature has shown to be unfavorable for some silicone materials. It has been found that the ageing in high temperature and humidity in some studies led to very fast degradation of the silicone material and was thus found not to be representative of the expected degradation behavior in real service. An alternative method to determine the expected service life of silicone seals was needed.

The study was carried out by Ulf W. Gedde, Professor Emeritius KTH. The reference group consisted of Karin Jacobsson, technical expert. The study was performed within the Energiforsk Polymers Program, which is financed by Vattenfall, Uniper, Fortum, TVO, Skellefteå Kraft and Karlstads Energi.

Summary

Components made of silicone rubber are used in Swedish nuclear power plants. Elastic polymer sealants used in pipe transits and joints are examples of applications. It is known that silicone rubber does not resist LOCA. The question is how silicone rubber components behave at normal service conditions. Silicone rubber is thermally stable and it withstands solar radiation. Silicone rubber is sensitive to moisture at elevated temperatures. This report presents a background to the experimental work required to predict lifetimes of silicon rubber components subjected to moisture at 20 – 50 °C.

By moist ageing of silicone rubber samples at 60 to 100 °C and in neutral water and alkaline water (pH = 13.5; resembling fresh concrete) data are obtained that allows extrapolation and prediction of lifetime at lower temperatures. This report presents a fundamental research part that reveals the kinetics of degradation of the material. A simpler, less time-consuming method is also suggested that uses tensile testing at 23 °C to assess fracture strain and its dependence on temperature, relative humidity and pH during ageing. Structural integrity and preserved adhesion to the surrounding materials in a transit joint are important. The obtained mechanical data can be used to predict lifetimes at lower service temperatures (20 – 50 °C), also focussing on both fracture toughness and adhesion to surrounding materials.

Keywords

Silicone rubber; moist ageing; molecular mechanisms; lifetime prediction; project plan

Sammanfattning

Funktionella detaljer av silikongummi finns i svenska kärnkraftverk. Elastiska tätskikt av silikongummi i genomföringar för rör är exempel på en typisk tillämpning. Det är känt att silikongummi inte tål de höga temperaturer och ångtryck som föreligger vid LOCA. Frågan är om beständigheten hos silikongummi är tillräcklig för att klara förhållandena vid normaldrift. Silikongummi är termiskt stabilt och mycket stabilt mot solljus. Silikongummi uppvisar viss fuktkänslighet vid högre temperaturer. Rapporten presentera en vetenskaplig bakgrund till det experimentella arbete som behöver göras för att prediktera livslängd hos komponenter av silikongummi exponerade för fuktig luft i temperaturintervallet 20 till 50 °C.

Genom åldrande av silikongummiprov i fuktig luft vid 60 till 100 °C samt i neutralt vatten och alkaliskt vatten (*pH* =13.5; efterliknar nygjord betong) skapas en databas som möjliggör extrapolation och uppskattning av livslängden vid lägre temperatur. Rapporten presenterar ett projektavsnitt med grundläggande forskning för att i detalj beskriva nedbrytningshastigheten av silikongummi. Ett projektavsnitt med enklare tillämpad forskning ingår i forskningsplanen. Genom dragprov vid 23 °C av åldrade provstavar bestäms brott-töjningen som funktion av åldringsbetingelserna temperatur, relativ fuktighet och *pH*. Sambanden används sedan för extrapolation till lägre temperatur (20 – 50 °C). Strukturell integritet och bibehållen adhesion till omgivande material hos tätskiktet i genomföringar är avgörande för funktionaliteten hos komponent. Erhållna mekaniska data möjliggör en säker extrapolation till lägre temperaturer, med ett fokus på brottseghet och adhesion till omgivande material.

Nyckelord

Silikongummi; åldrande i fuktig miljö; molekylär mekanism; prediktering av livslängd; projektplan

List of content

1 Fundamental aspects and theory

Silicone rubber is based on poly(dimethyl siloxane), $– [Si(CH_3)_2 – O]_n –$. The polymer is abbreviated PDMS. Commercial silicone rubber materials contain additives, e.g. surface-treated amorphous silica (SiO_2) particles binding to the PDMS network polymer, and other inorganic fillers (also surface treated) such as aluminium trihydrate [1,2]. The repeating unit contains two hydrophobic methyl groups and a polar part, the SiO backbone. When PDMS is exposed to dry air, the surface becomes enriched in methyl groups. In water, the methyl groups fear the surface and the surface becomes enriched in the polar SiO-units [1,2]. Hence, PDMS has a double chemical nature.

PDMS is crosslinked by several methods: the high temperature method using peroxide and the low temperature process [3]. Both methods yield a polymer network with a fraction of dangling chain ends (cilia) and a soluble fraction with linear and cyclic oligomers [4]. At temperatures above 450 °C, the main degradation product is a cyclic oligomer with 3 repeating units, D_3 [5–7]. The oligomers are efficiently removed from the polymer by extraction in chloroform or a hydrocarbon solvent such as *n*-hexane [4]. However, after storage at moderate temperatures, small amounts of cyclic oligomers, mainly consisting of 4 to 9 repeating units (D_4 to D_9), are formed by a backbiting mechanism either from a chain end or randomly along a polymer chain.

PDMS is a polymer with a flexible backbone which is evident in many properties: low glass transition temperature (–123 °C [3]), hardening by crystallisation at low temperatures (– 60 to – 80 °C [3]) and poor barrier properties with high diffusivity of small molecule penetrants [8]. Both polar and nonpolar species have high permeability through PDMS.

Silicone elastomers are extra-ordinarily durable towards outdoor (sunny) conditions [9]. Exposures to partial discharges, which yield high-energy UV-light, oxidize PDMS and renders a thin surface layer of glassy SiO_x that is hydrophilic [1, 2, 10–14]. With time, the hydrophilic layer is covered by liquid dimethyl siloxane oligomers which contain cyclic oligomers (D_4 to D_9) and other low molar oligomers including linear molecules which change the surface from hydrophilic to hydrophobic [1, 2, 4, 10–14].

The presence of dissolved water in PDMS, particularly at high temperatures, splits the polymer chain by hydrolysis and yields two silanol groups according to Figure 1 [15,16].

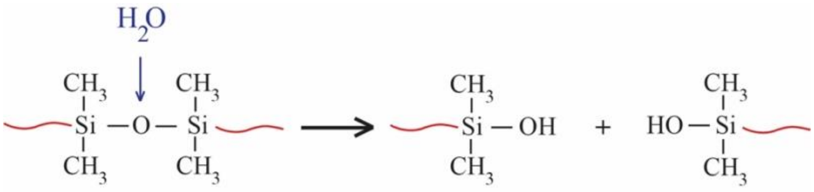

Figure 1: Hydrolysis of a PDMS chain

Hydrolysis also attacks the covalent bonds in the coating layer surrounding silica particles. A reversing condensation reaction is dominant at low water concentrations. The hydrolysis reaction is catalytically accelerated by alkaline and acidic conditions. In pristine water ($pH = 7.0$), the activation energy for the hydrolysis reaction is 96 kJ mol^{-1} [17]. In alkaline water ($pH = 13.5$; resembling fresh concrete), the activation energy is 60 kJ mol^{-1} [18]. Note that the catalytic effect is reducing the activation energy and also by increasing the pre-exponential factor in the Arrhenius equation. The hydrolysis reaction is accompanied by the formation of cyclic oligomers (back-biting).

Let us sketch what is required to describe the degradation rate by hydrolysis. Three factors are central: temperature (T), water concentration in the polymer component (C_{H2O}) and pH. The water concentration is given by Henrys law [8]:

$$C_{H2O} = k_h \times \mathrm{RH} \times p^*_{H20} \tag{1}$$

where k_h is the henry law coefficient, RH is the relative humidity (= p_{H20}/p^*_{H20}) and p^*_{H20} is the saturation water gas pressure at the particular temperature. Note that p^*_{H20} is a function of T. This is also true for k_h. The rate for the hydrolysis $(dn/dt)_{hydro}$ is given by:

$$(dn/dt)_{hydro} = C_{H2O} \times A \times \exp(-\Delta E_{a,h}/RT) \tag{2}$$

where A is a constant to be determined by fitting Eq. (2) to obtained experimental data. It should be noted that both A and ΔE_a are functions of pH.

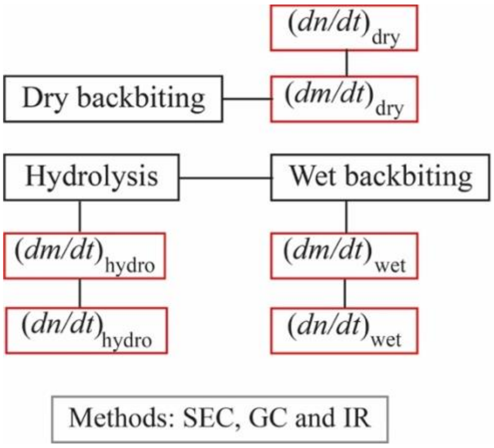

Figure 2: Kinetics of PDMS degradation including experimental assessment methods. SEC: size exclusion chromatography; GC: gas chromatography; IR: infrared spectroscopy

Degradation of silicone rubber occurs also by oxidation even in the absence of corona discharges or high-energy radiation. Oxidation has a much slower pace than hydrolysis and is here disregarded. More significant are the backbiting reactions. Dry backbiting refers to a reaction from the original structure. The extent of this reaction can be obtained by gravimetric and chromatographic analyses of samples after ageing at elevated temperatures under dry conditions. The kinetics of wet backbiting can be assessed by gravimetric and chromatographic analyses after ageing in moist conditions.

How is $(dn/dt)_{hydro}$ assessed? The mole-number of events per gram sample and time is one parameter to assess. Gravimetry provides *dm*/*dt*-data and by measurement of the molar mass distribution of the extracted soluble fraction, the number of soluble molecules can be assessed, which can be used to calculate the number of degradation reactions per time unit (*dn*/*dt*). Soluble high molar mass species are due to hydrolysis whereas the low molar mass species (oligomers) originates from the back-biting reaction. Hence, by using SEC and GC, polymer degradation under moist conditions can be revealed.

The general task is to assess the degradation rates as a function of *T*, C_{H2O} and *pH*. Data taken at high temperatures with a systematic change in the relative humidity should be obtained and the extent of structural and property change should be revealed. The rates of structural change rates should be monitored as a function of *T* and C_{H2O} at constant *pH*. The trends are finally extrapolated in order to obtain the rates at service conditions. The samples to be used in the fundamental science study should be pristine samples of silicone rubber with precise molecular structure. Commercial silicone materials contain fillers. A few prominent materials of this category should be included. They should be part of the fundamental study (Section 2) and the applied study (Section 3). The kinetics of the hydrolysis depend on *pH*. The water concentration dependence of the hydrolysis is assessed only at $pH = 7.0$ at different temperatures. Another studied case is $pH = 13.5$ (alkaline conditions), which resemble fresh concrete. The temperature shift factor (activation energy) and the preexponential coefficient are assessed. Together with the data obtained at neutral pH conditions, a data set is obtained that can provide the base for extrapolation in temperature, water concentration and *pH*.

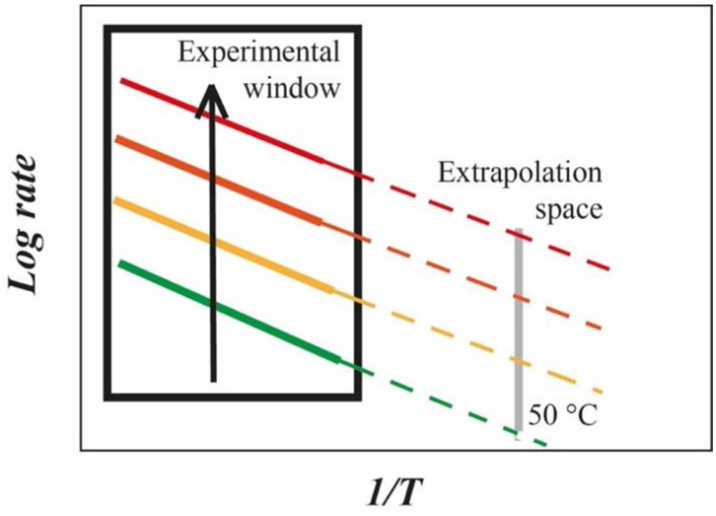

Figure 3: Method sketch showing prediction at constant *pH*. The arrow indicates increasing C_{H2O}; the green line shows data for the lowest C_{H2O}-value

Let us just go through how this study should be made. Step one is to prepare thin film samples. We recommend the use of four different PDMS materials: two materials with exact network structure with no soluble fraction and two commercial materials. Asses the water uptake (expressed by Henry's law) at different temperatures and different *RH*. The second part is to select the proper *RH*-values for the different temperatures in order to obtain a useful spread in the C_{H2O}-values.

The third part is to carry out ageing at 60, 80, 90 and 100 °C. Samples are taken out after 0, 10, 100 and 1000 h at 100 °C. A shift factor corresponding to 96 kJ mol^{-1} ($pH = 7.0$) is used to calculate the required times at 60, 80 and 90 °C. Extrapolation to 20 – 50 °C is carried out by using the Arrhenius equation. The moist ageing experiments is first carried out at $pH = 7.0$ (liquid water). In a second series of experiments, the sample are exposed to alkaline water ($pH = 13.5$) and the degradation rate data taken at 60, 80, 90 and 100 °C is extrapolated to the service temperatures using the Arrhenius equation.

Now we come to the question what to measure. One piece is used to measure the mass change accompanying extraction in a good solvent. The change in mass after extraction and drying includes the mass of the sorbed water and the extractable oligomers. A limited number of chemical analyses (SEC and GC) are carried out, mapping direct hydrolysis products being higher molar mass compounds and backbiting products being low molar mass products. Dumb-bell specimens are obtained by punching and the initial modulus and the strain at break (ε_b) are determined. Both quantities have a relationship to the extent of the reaction.

Chain scission yield a looser network and lowering in the initial modulus. The obtained ε_b -data is expressed as a function of ageing parameters (T, RH and pH). This function is extrapolated to lower temperatures (20 – 50 °C) and specified values of RH and pH. The life time assessments require the establishment of a critical property value, e.g. a critical value for ε_b. This require functional tests focussing on tightness of sealing which in turn requires assessment of adhesion between silicon rubber and potential materials in contact (e.g. concrete and metal). Both adhesive and cohesive fracture modes are sensitive to polymer degradation and the required stress to obtain crack propagation should be firmly related to ε_b. Establishment of this function thus finally permits a lifetime prediction under given input parameter values (T, RH and pH).

2 Experiments and analyses of data concerned with fundamental science

2.1 MATERIALS

Pristine crosslinked PDMS materials are prepared as follows: vinyldimethyl-terminated PDMS (two types: $M_n \approx 7000$ g mol^{-1} and $M_n \approx$ 20 000 g mol^{-1}; M_w/M_n should be in the range 1.5 to 2.0) are crosslinked by a hydrosilylation reaction, using a (30 – 35 %) methyl-hydro – (65 – 70 %) dimethylsiloxane copolymer with $M_w = 2100$ g mol^{-1} as crosslinker. The ratio of hydride to vinyl groups should be 2:1 in order to obtain a fully crosslinked structure. A platinum divinyltetramethyl-disiloxane-complex is used as catalyst at a low concentration (30 ppm). These chemicals can be obtained from United Chemical Technologies, USA (web: unitedchem.com). Sheets being 1 mm thick are obtained by compression moulding at 135 °C (time ≈ 15 min) and then post-curing performed in an oven at 120 °C for 12 h. The soluble fraction is removed by extraction in boiling *n*-hexane. The films are finally dried in a vacuum oven and stored in a desiccator.

Two different commercial crosslinked PDMS materials representing the materials used in Swedish nuclear power plants are obtained. The preferred shape: films being 1 to 3 mm in thickness. Two different samples are obtained: (i) Films without any further change; (ii) Films with removed soluble fraction by extraction in boiling *n*-hexane.

Two different samples/specimens are prepared: small squares (20×20 mm) aimed for the reaction kinetics study. This includes both the model materials (pristine crosslinked PDMS) and the commercial composite materials (including both types). Dumb-bell shaped specimens are punched from the four different film samples. Details: narrow section = 12 mm (length) × 2 mm (width), punch die part no 4 (ISO 37:1994 E).

2.2 AGEING

Dry ageing exposures are carried out at 60, 80, 90 and 100 °C for different periods of time. These studies provide information about the kinetics of dry back-biting. Ageing in moist air (different *RH*) of samples are carried out at

60, 80, 90 and 100 °C. This includes both the small squares and the dumb-bell shaped specimens (Details: narrow section = 12 mm (length) × 2 mm (width), punch die part no 4 (ISO 37:1994 E)). Ageing of silicone rubber is also carried out in pristine water (pH = 7.0) and alkaline water (pH = 13.5). The latter is prepared according to the following recipe: 0.6 g $Ca(OH)_2$, 72 g NaOH, and 58.36 g KOH in 4 L distilled water, according to ASTM D7705.

Table 1: Ageing procedures in fundamental study[a,b,c,d]

60°C ($H_2O(g)$)	80°C ($H_2O(g)$)	90 °C($H_2O(g)$)	100 °C($H_2O(g)$)	80 ($H_2O(l)$)	100 ($H_2O(l)$)
0 %RH (2 t_a)	0 %RH (2 t_a)	0 %RH (2 t_a)	0 %RH (2 t_a)	pH =7.0, 13.5	pH =7.0, 13.5
30 %RH (4 t_a)	30 %RH (4 t_a)	30 %RH (4 t_a)	30 %RH (4 t_a)	3 t_a	3 t_a
60 %RH (4 t_a)	60 %RH (4 t_a)	60 %RH (4 t_a)	60 %RH (4 t_a)		
100 %RH (4 t_a)	100 %RH (4 t_a)	100 %RH (4 t_a)	100 %RH (4 t_a)		

[a] Includes 4 materials with ageing temperatures and surrounding medium ($H_2O(g)$ in air or ($H_2O(l)$)

[b] Ageing times (t_a) are calculated based on activation energy values from the literature

[c] Approximate relative humidity values (%RH)

[d] In liquid water case, two different pH values

The fundamental study uses 246 samples, of which 4 are from the fresh (unaged) silicone rubber materials. A preferred maximum ageing time is 6 to 12 months. To this, 928 dumb-bell specimens are used for the mechanical study. A preferred maximum ageing time is 6 to 12 months.

Table 2: Ageing procedures in applied (simple method) study[a,b,c,d]

60°C ($H_2O(g)$)	80°C ($H_2O(g)$)	90 °C($H_2O(g)$)	100 °C($H_2O(g)$)	80 ($H_2O(l)$)	100 ($H_2O(l)$)
	0 %RH (2 t_a)		0 %RH (2 t_a)	pH =7.0, 13.5	pH =7.0, 13.5
30 %RH (5 t_a)	30 %RH (5 t_a)	30 %RH (5 t_a)	30 %RH (5 t_a)	5 t_a	5 t_a
60 %RH (5 t_a)	60 %RH (5 t_a)	60 %RH (5 t_a)	60 %RH (5 t_a)		
100 %RH (5 t_a)	100 %RH (5 t_a)	100 %RH (5 t_a)	100 %RH (5 t_a)		

[a] Includes 2 materials with ageing temperatures and surrounding medium ($H_2O(g)$ in air or ($H_2O(l)$)

[b] Ageing times (t_a) are calculated based on activation energy values from the literature

[c] Approximate relative humidity values (%RH)

[d] In liquid water case, two different pH values

The simple method uses 464 dumb-bell specimens, of which 20 are from the fresh (unaged) silicone rubber. A preferred maximum ageing time is 6 to 12 months.

2.3 ANALYSIS OF WATER UPTAKE

The equilibrium water uptake is assessed as a function of T and RH. The dry sample mass is first measured. The sample mass is measured in controlled RH as a function of time. Different equations, primarily Henry's law (cf. Eq. (1)), are fitted to the experimental data. The low solubility of water in PDMS suggest that Henry's law is applicable.

2.4 ANALYSIS OF REACTION RATES

Samples of initially measured mass are aged in moist air (covering a wide *RH*-range) at 60, 80, 90 and 100°C at different periods of time and two different *pH*-values (7.0 and 13.5) at 100 % *RH* (liquid water). The sample mass is measured directly after the moist ageing. The samples are then extracted in boiling *n*-hexane and dried to constant mass. The difference in mass before and after moist ageing and extraction in *n*-hexane followed by drying provide a mass rate estimate of the reactivity. Analysis of extracted species leaving the aged samples is performed with GC and SEC in accordance with the methods used in Ref. [4]. The concentration of silanol groups can be assessed by IR. The mass loss rate data (dm/dt) are analysed by fitting the Arrhenius equation to the experimentally found data:

$$(dm/dt) = A \times \exp\left(-\Delta E_a/RT\right) \qquad (3)$$

where A is an adjustable constant (pre-exponential factor), ΔE_a is the activation energy, R is the gas constant and T is the absolute temperature (in kelvin).

3 Simple method to assess long-term performance

3.1 TENSILE TESTING

This part can be used in conjunction with the experiments presented in Sections 2.1 – 2.4. The tensile testing presented may also be solely used on the commercial materials. This approach is believed to provide useful empirical data for extrapolation and prediction of lifetimes at lower temperatures (20 – 50 °C). Unaged and aged dumb-bell shaped specimens (details: narrow section = 12 mm (length) × 2 mm (width), punch die part no 4 (ISO 37:1994 E) are tested in a tensile testing machine at 23 ± 1 °C and 50 % *RH*. The elongation rate is 30 mm min^{-1}. The initial modulus and the strain at break are recorded (3 specimens are tested for each ageing temperature and ageing time). Only fractures in the narrow section should be included in the statistical analysis. The further analysis of the data is illustrated by Figure 4.

The data presented in Figure 4 (shown as an educative illustration) can be extrapolated to any lower temperature by extrapolation of the isotherm to a higher $1/T$-values. All the data obtained at all ageing temperatures can be used to generate a master curve at for instance 150 °C and this master curve can be shifted to any chosen service temperature. All the data points displayed in the graph would appear in this master curve. The use of this methodology is recommended.

The advice is also to compare the shift factors (ΔE_a-values) obtained for the different processes, i.e. the degradation processes and the fracture data. Note that in the current case, the degradation process and the strain at break data are functions of both temperature (expressed by (ΔE_a (log a_T)) and *RH* (expressed by another shift factor).

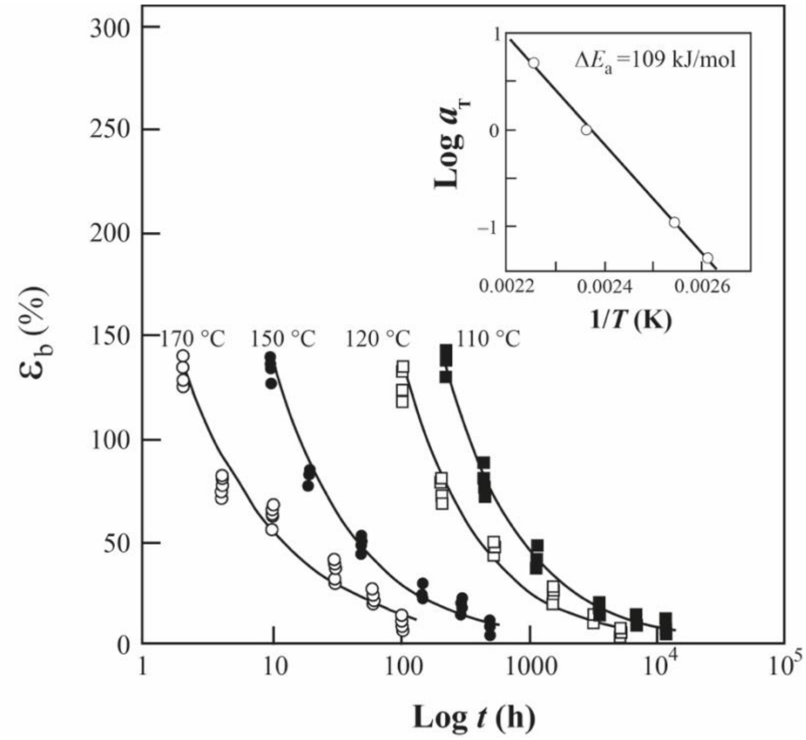

Figure 4: Strain at fracture (ε_b) as a function of ageing time (t; note the logarithmic axis) at different ageing temperatures. By horizontal shifting (log a_T) to obtain maximum overlap, the insert Arrhenius diagram was obtained. Note that 150 °C data is the reference (log a_T = 0). The data are corned with EPDM rubber aged in air [19]

Strictly, the rate of the degradation reaction should be a function of temperature and the water concentration in the polymer component. I recommend that the (*dm*/*dt*)-data given as a function of *T* and *RH* is changed to be a function of *T* and C_{H2O} in accordance with Eq. (2).

A common use of silicone rubber is in transits and joints where the adhesion between the rubber and surrounding materials; the latter can be concrete or metals. Measurements of adhesion is much more complicated and the measurement of fracture strain is simpler and more reliable to determine the temperature shift factor. Furthermore, the loss of mechanical integrity expressed in fracture strain is expected to scale with resistance of both adhesive and cohesive failure modes. My proposal is to age a sandwich consisting of silicone rubber and concrete according to the fracture strain data as a function of temperature, reactive humidity and *pH*. A correlation between adhesive strength and fracture strain should be established.

3.2 CRITICAL STRAIN AT BREAK ASSESSMENT

The establishment of a critical ε_b requires functional testing of samples that has been aged under accelerated conditions. Several duplicates are required to have a useful statistical ground to establish a safe assessment of the critical ε_b. Figure 5 shows a potential sample geometry.

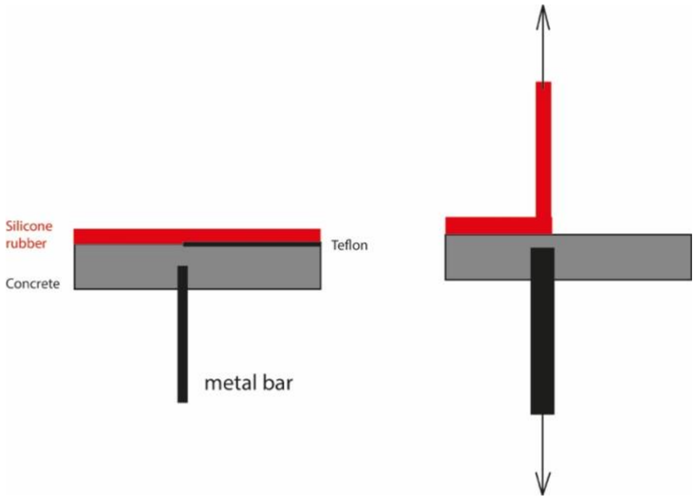

Figure 5: Specimen geometry aimed for testing adhesion of aged samples

A test similar to that used in LOCA is to age a full-sized system, a pipe going through concrete with a silicone rubber sealant to different levels of degradation and then measure the functionality of the sealing (tightness).

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SILICONE RUBBER IN NUCLEAR POWER PLANTS: PREDICTION OF SERVICE LIFETIME IN MOIST CONDITIONS

Silicone rubber materials are used in Swedish nuclear power plants in pipe transits and joints. This report describes the polymer science background and the ageing mechanisms under moist conditions. A project plan is presented that include both a fundamental science approach and a more time-efficient empirical method that enable lifetime prediction valid for typical service conditions.

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