

# Defining joint sealant condition in nuclear power plants

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<b>Summary</b> <p>Joint sealants are applied in nuclear power plants in between structural elements, such as concrete blocks. Their ageing is a less studied phenomenon. In this work the earlier obtained sealant samples from nuclear power plants are analysed with tensile test, dynamic mechanical thermal analysis (DMTA) and hardness measurement and the results were compared to a newly installed reference sealant. The sample preparation from the aged sealants was challenging. Most of the aged samples showed lower elongation at break, elastic modulus and hardness values than the new reference sample as it would be expected. However, one aged sample showed higher values for the mechanical properties measured compared to the reference sample. A detailed composition analysis of the sealant would be required to analyse the cause behind this. The elastic modulus defined with DMTA deviated from what was analysed based on the tensile test, indicating some complexity in elastic modulus measurement for these materials. Hardness seemed to be the most suitable material property to characterize the ageing out of the three methods applied in the study. Future work was proposed to develop hardness measurement to be used as part of on-site condition monitoring of these types of joint sealants.</p>	
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## Preface

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This work was completed as part of the Finnish Research Programme on Nuclear Power Plant Safety 2019 – 2022 (SAFIR2022) within the SAMPO (Safety criteria and improved ageing management research for polymer components exposed to thermal-radiative environments) project's work package 2 "Improvements in ageing management of polymer components".

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Authors

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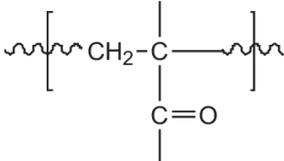
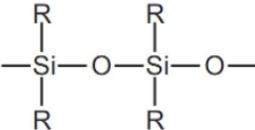
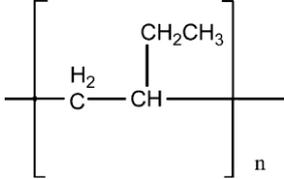
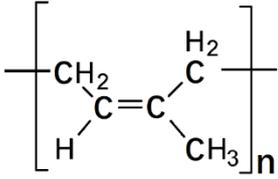
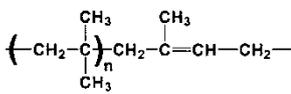
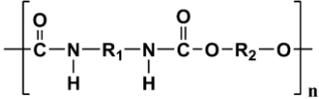
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## 1. Introduction

Joint sealants are polymer-based sealing solutions that are applied in between structures, such as floor and wall elements, thus joining them together and forming a gas tight structure. Such sealants are widely applied in constructions, including nuclear power plants (NPPs). However, their ageing in nuclear power plant environment is a less studied phenomenon. Table 1 shows the wide range of polymers, which can be used as a base polymer in joint sealants. Each type of joint sealant will have slightly different properties and target of application. The environment of use usually defines the joint sealant type as various different options are available for indoors, wet rooms (bathrooms and kitchen) or outdoor use.

Table 1. Summary on the most common joint sealant materials and their applications.

Sealant material	Application	General properties	Molecular structure
Acrylic	Wide range of building materials	Retains pliability, cannot be applied at low temperatures, good water resistance	
Silicone	Household use (kitchen and bathroom) and construction use (wood, stone, metal bricks)	Flexible, retains pliability, sticks to painted surface but cannot be overpainted	
Polybutene	Wide range of temperatures (e.g. road construction products)	Low cost, excellent durability and water resistance, good package stability and adhesion, will withstand cyclic joint movement, can stain adjacent surfaces and relatively poor recovery from extension	
Latex	Bathroom joints, cracks in plaster, tile, glass and plastic.	Easy to use, water resistant, not very flexible	
Butyl	Wide range of building and dissimilar materials	Good water resistance, very durable, poor adhesion to painted surfaces	 butyl rubber
Polysulphide	Basements, glazing frames, ceiling joints, floors, roofs, external walls, cladding, retaining walls, water retaining structures, joints in bridges, roads and aerodomes	Good water and chemical resistance	-S-S- (polymer containing a chain of sulfide atoms)
Polyurethane	Wide range of building materials, wider and irregular cracks	Retains pliability, good water resistance, overpainting recommended on foam (susceptible to UV-light)	

Heat, moisture and radiation are the most important ageing stressors that the joint sealants experience in NPPs. These stressors subject the sealants to oxidation and loss of plasticizers. Swelling and leaching become relevant if there is exposure to excess moisture or water.

In the earlier work, various methods were listed for condition monitoring of joint sealants, and it seems that the most applicable ones include hardness measurement, differential scanning

calorimetry (DSC) and Fourier transform infrared spectroscopy (FTIR) [1]. In this work hardness measurement is applied to define the material condition and compared to the elongation at break (EaB) and modulus results obtained from tensile test. Modulus values are defined also by dynamic mechanical thermal analysis (DMTA) to compare the values obtained from tensile test.

## 2. Goal

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The goal of this work is to assess the condition of sealants obtained earlier from the nuclear power plants by the means of tensile testing, DMTA and hardness measurements. Based on the results, suitability of each method to be used as a condition monitoring technique for such joint sealants is evaluated and using the measured material properties as acceptance criterion is assessed.

## 3. Materials and methods

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The materials were obtained earlier from TVO and OKG. A commercial sealant (tradename Sikaflex® + Self Leveling Sealant) was studied as a “new” reference sample. The sealants from TVO were chosen based on their location at the plant. The highest floor of the reactor building was chosen emphasized as it was assumed that these samples might be the most aged ones. 4/5 of the tested TVO samples were located at the seventh floor of the reactor building and 1/5 on the fifth floor. More details on the TVO samples have been earlier reported in [1]. The OKG sealing tradename was Tremco and it had been used for ca. 30 years in service. The properties of the Sikaflex sealant can be found on the data sheet [2]. The total number of tested materials was thus seven.

The tensile tests were performed according to ISO 527, except the sample geometry was not according to the standard due to practical reasons. Instron 5500K8810/4505H2190 machine with 100 N load cell and pneumatic grips was used as the test equipment. Bluehill software (version 4.25) was used as a measurement software and data analysis.

Dynamic mechanical thermal analysis (DMTA) yields information about the mechanical properties of a specimen as a function of time and temperature under small, usually sinusoidal, oscillating force. With this method, mechanical stiffness (storage modulus) and damping (loss modulus) over a wide range of temperatures and different frequencies can be measured. In these measurements, measurement frequency of 1 Hz was applied and a temperature range from ca. -80 to 80 °C. The used equipment was Mettler Toledo DMA/SDTA861. Benefit of DMTA analysis in this case was its simpler sample preparation phase than in tensile testing.

Shore-A hardness was measured according to ISO 7619-1 standard.

## 4. Results and discussion

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Sample preparation from all aged samples was challenging. Their structure was soft and sticky and preparing a sample for DMTA or tensile test with homogenous dimensions was difficult. In case of some samples, the sample preparation was possible but attaching the sample between the tensile test machine grips was not possible as the sample was heavily deformed as it was pressed by the grip. In Figure 1 some of the prepared tensile samples can be seen. For example, it is clearly visible that sample 2.1 is deformed while prepared. Preparing a sample from the new Sikaflex material was clearly easier. The summary on the analyses that could be successfully performed to different samples are shown in Table 2.

This shows that preparing a suitable tensile specimen and attaching it to the testing equipment was more challenging than performing the DMTA measurements.

Table 2. Summary on the tests performed to different samples.

Test / Sample	TVO 1.5	TVO 1.6	TVO 1.9	TVO 2.1	TVO 2.2	Sikaflex	OKG
Tensile test		x	x	x		x	
DMTA	x	x	x		x	x	x
Hardness	x	x	x	x	x	x	x



Figure 1. Samples prepared from TVO material and the reference Sikaflex materials.

Figure 2 - Figure 5 show the stress-strain curves for the TVO samples and the commercial Sikaflex sample. Two tests per each TVO sealant were performed and average values of these two tests were used both for elongation and elastic modulus values in most cases. For the commercial sealant five tests were performed as there was more material available. The elastic modulus was defined based on the slopes of the linear part of the curves (see the two initial slopes in Figure 2 - Figure 5).

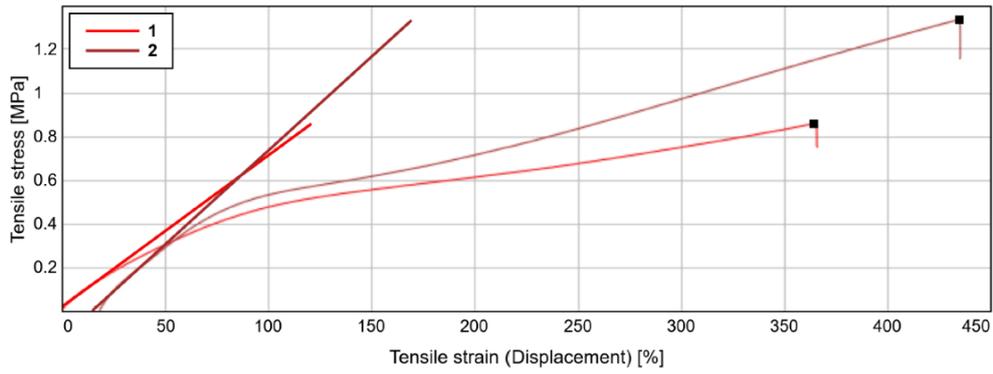


Figure 2. Tensile test curves for the two samples prepared from sealant TVO 1.6.

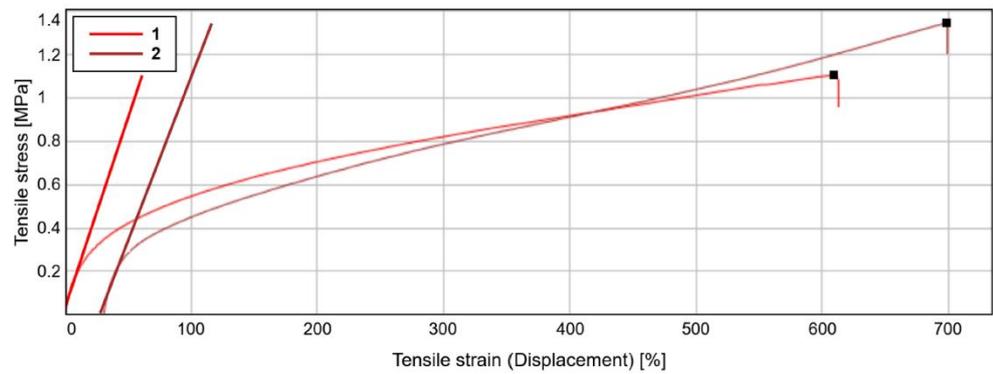


Figure 3. Tensile test curves for the two samples prepared from sealant TVO 1.9.

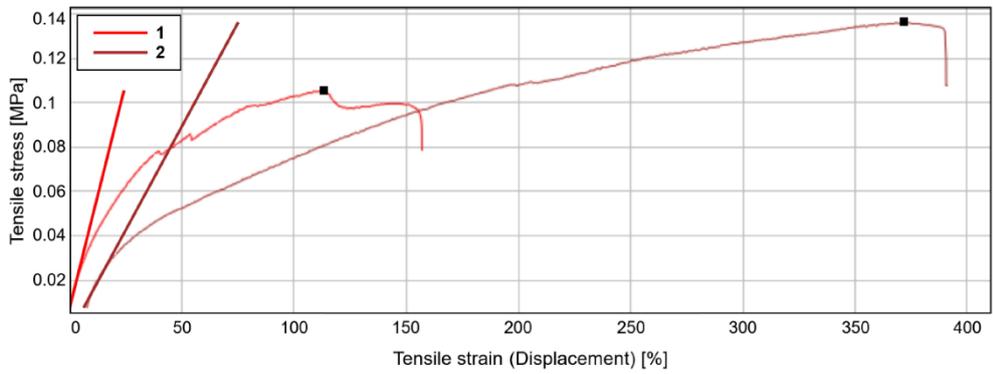


Figure 4. Tensile test curves for the two samples prepared from sealant TVO 2.1.

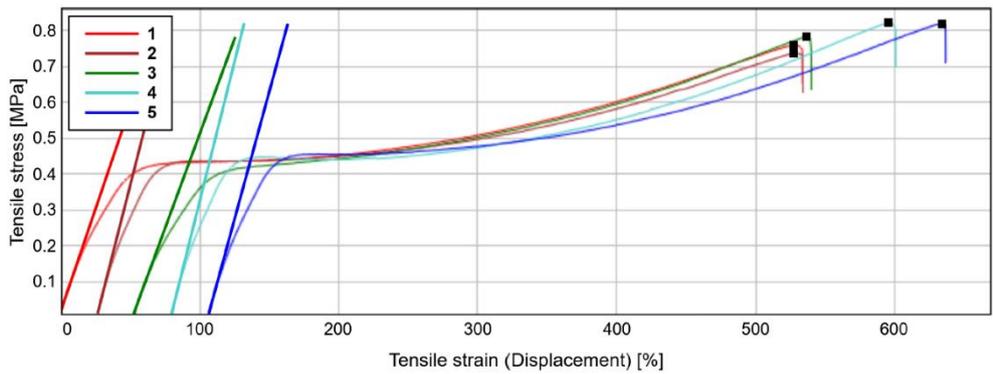


Figure 5. Tensile test curves for the five samples prepared from sealant Sikaflex® + Self Leveling Sealant.

With TVO 2.1 samples, the two stress-strain curves deviate from each other. It is suspected that the first sample with shorter elongation had defects that have caused the smaller elongation. A likely source of the defects is the sample preparation phase as preparing a dumb bell-shaped sample was very challenging for this material. Thus, the first curve was not considered to represent the actual material condition and the elongation at break and elastic modulus values were extracted only from a single curve.

Figure 6 and Figure 7 summarize the elongation at break and elastic modulus values extracted from the tensile tests. Interestingly one of the aged sealant materials has a higher elongation at break value than the new commercial material. It should be noted that the materials differ from each other by their composition affecting the comparison of the results. However, the result show that the aged sealant may still show significantly high elongation at break values, indicating that the material property in question is not necessarily the most representative property to describe the condition of the sealant. From the elastic modulus values the same trend can be observed as from the elongation at break results. TVO 1.9 sample shows larger elastic modulus value than the new commercial sealant.

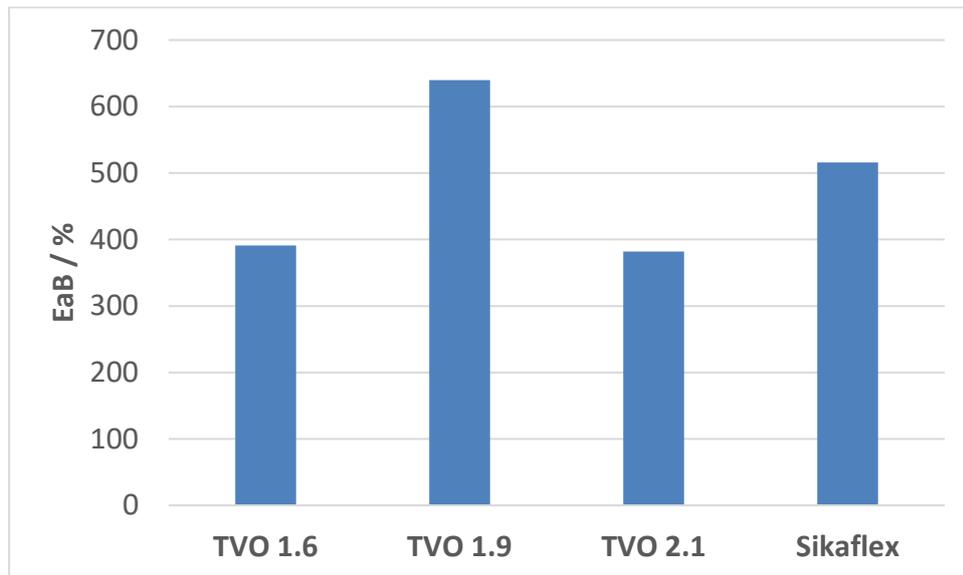


Figure 6. Elongation at break results.

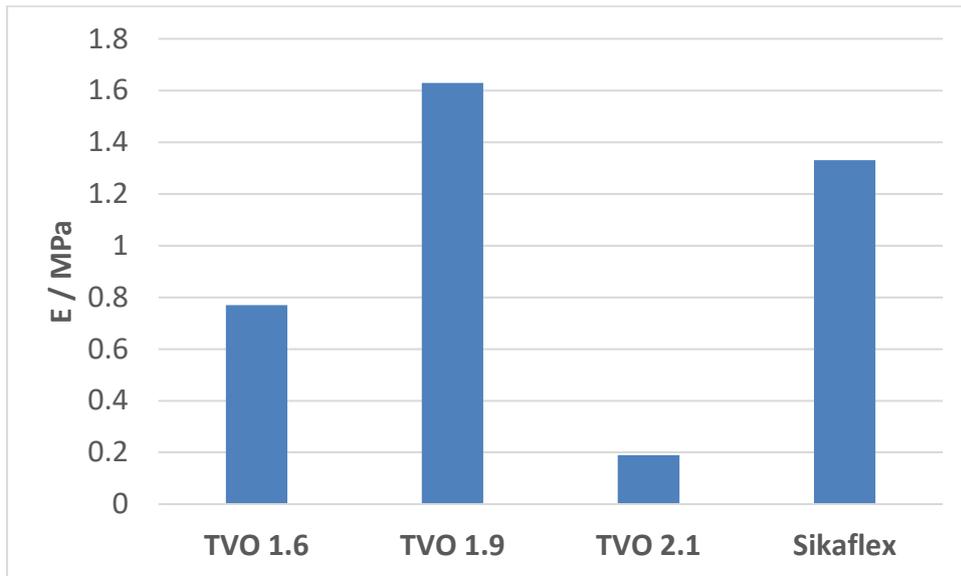


Figure 7. Elastic modulus values calculated from tensile test data.

Figure 8 shows the DMTA curves for the measured samples. The storage modulus  $G$  was extracted around the temperature of 20 °C. The elastic modulus is known to be a function of  $G$  and Poisson's ratio  $\mu$ :

$$E = 2(1 + \mu)G \quad (1)$$

In such a soft materials as the studied sealants, the Poisson's ratio is very close to 0.5. Thus, Equation 1 can be simplified in form of:

$$E \approx 3G \quad (2)$$

This equation was applied as the elastic modulus  $E$  was extracted from the DMTA data. The results are shown in **Error! Reference source not found.** TVO 1.9 and 2.2 sealant materials show clearly higher  $E$  values compared to other samples. The  $E$  value for TVO 1.9 material was roughly 4-times larger than was defined based on tensile tests, for TVO 1.6 material 3-times larger and for the commercial Sikaflex material roughly 2-times larger. The modulus values obtained for TVO 1.5 and OKG samples were the lowest. These were the two materials that could not be tested by the tensile test.

It is possible that the test samples were not representative enough for the DMTA tests, which may cause error in the  $E$  values. Also, the approximation of Poisson's ratio could contain some error, causing higher calculated  $E$  values. Furthermore, the measurements were performed only with a single sample and additional measurements should be performed to obtain more reliable estimations for  $E$  (this concerns the  $E$  values calculated from the tensile tests as well).

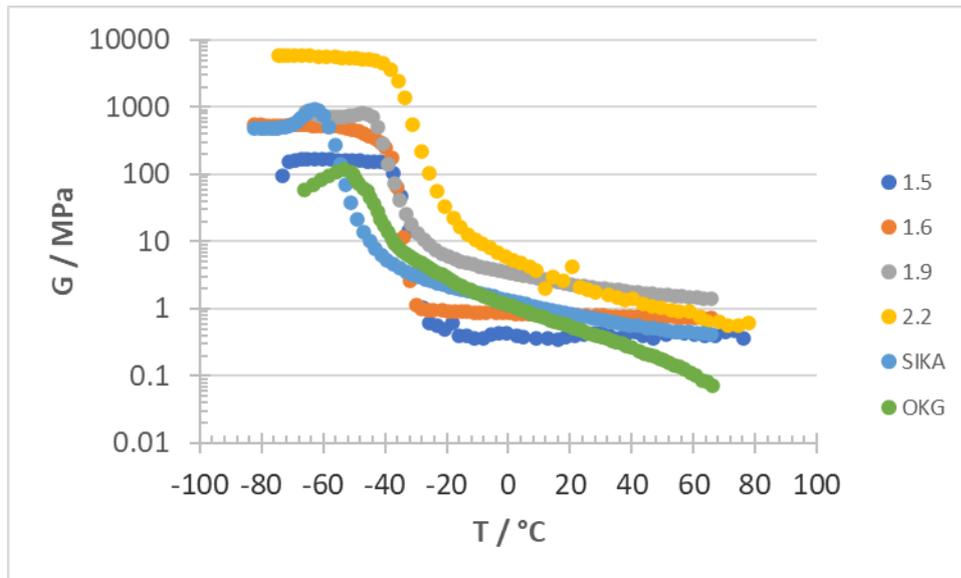


Figure 8 DMTA curves for the measured samples.

Table 3. Temperatures at which G values were extracted from the DMTA data and the calculated E values.

Sample	T / °C	G / Mpa	E / Mpa
1.5	20.1	0.4	1.2
1.6	19.4	0.81	2.43
1.9	20.4	2.32	6.96
2.2	20.4	2.36	7.08
Sikaflex	20.4	0.83	2.49
OKG	19.6	0.56	1.68

The hardness measurement results are shown in Figure 9. TVO 1.6, 1.9 and Sikaflex have higher hardness values compared to the other ones. TVO 1.6 and 1.9 have both higher hardness than the fresh Sikaflex sample. To define reason why some of the aged samples showed higher hardness than the fresh sample, additional composition analyses would be required. For TVO 1.5 two separate values are displayed as it was noticed that the sample contained two different areas with clearly different hardness value. TVO 2.1 and 2.2 as well as OKG material seem to have rather low hardness values compared to the new material, indicating severe ageing. This partly supported by the fact that no tensile test samples could be prepared from TVO 2.2 and OKG materials.

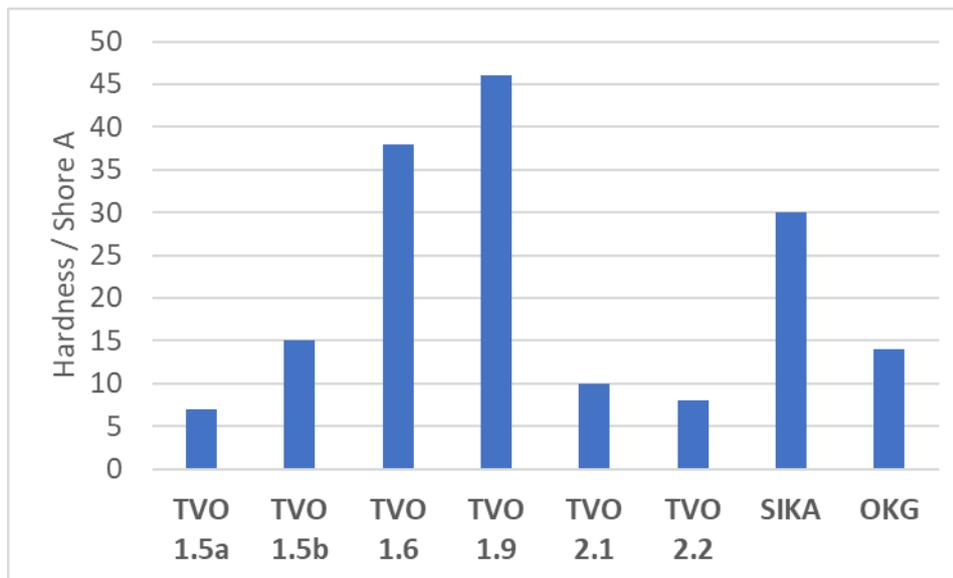


Figure 9. Hardness measurement results.

Based on the obtained results hardness result seems to be the most promising method to measure joint sealant condition. It is less laborious than tensile test and the hardness seemed to decrease more in aged samples than elongation at break (excluding TVO 1.9 sample). Hardness measurement can be considered as non-destructive which is an obvious benefit when sealant condition is tested onsite. Despite the fact that the DMTA method sample preparation and performing the test was somewhat easier than the tensile test, the obtained elastic modulus values deviated from those ones obtained from the stress-strain curves. Still, some of the most aged samples could be tested by DMTA but not with tensile test, indicating that DMTA could be suitable in testing severely aged samples.

## 5. Future work

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The following aspects should be considered as part of future work:

- Hardness measurement could be developed further for these types of materials as a non-destructive condition monitoring method.
- There are still plenty of aged sealants to be tested, possibly in better condition than the ones tested within the framework of this report. Measuring these samples could provide more data which would enable to analyse the sensitivity of the hardness measurement.
- Testing with artificially aged Sikaflex® + Self Leveling Sealant could enable prediction of lifetime of the sealant.
- Setting up an acceptance criterion for the hardness measurement would require correlation to sealing capability.
- Hardness measurement setup should be possible to put in practise for onsite condition monitoring relatively easily.

## 6. Summary

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The condition of joint sealant materials obtained from nuclear power plants and a reference new sealant material was determined in this work. The applied methods were tensile test, DMTA and hardness measurement. Tensile test and DMTA samples were challenging to prepare from the delivered sealant materials due to the quite severe ageing. When the results were compared, one of the aged samples showed higher elongation at break, elastic modulus and hardness values than the new reference sample. Hardness was concluded to be the most convenient material property to measure and representative as an ageing indicator for these types of materials. Future work was suggested to develop hardness measurement further to be applied on-site conditions.

## References

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- [1] Sipilä, K. 2022. Joint sealants and their ageing in nuclear power plants. VTT-R-00094-22. 14 pp.
- [2] Sika group. [Sikaflex®+ Self-Leveling Sealant | SIKA](#) (18.1.2023)