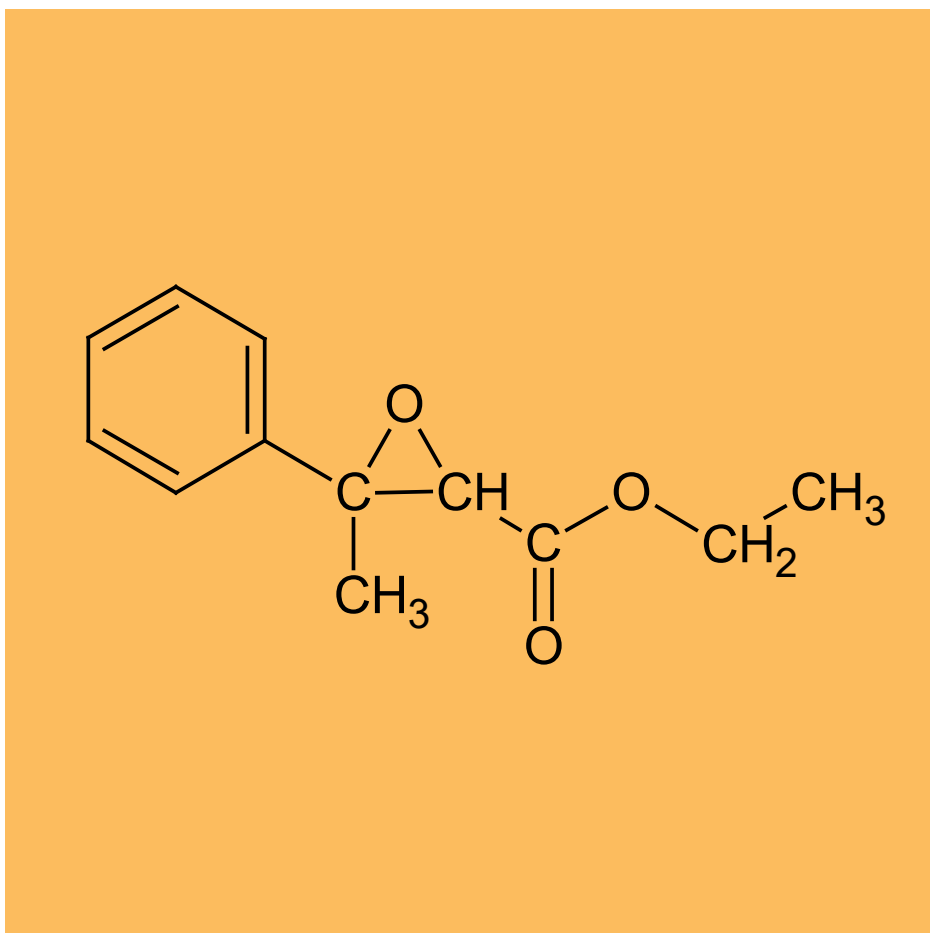
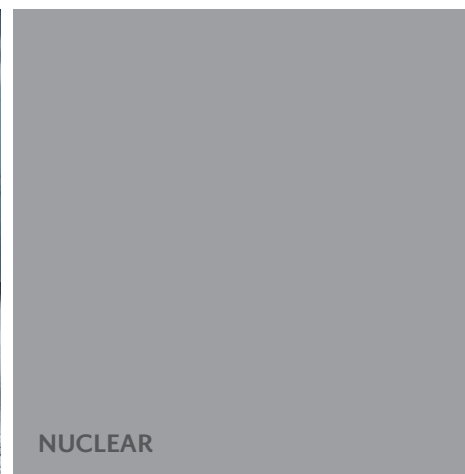


ACCEPTANCE CRITERIA FOR POLYMERS IN NUCLEAR

REPORT 2015:157



Acceptance criteria for polymers in nuclear

Feasibility study: Development of methodology and
guidelines for definition of acceptance criteria for
polymers

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Foreword

In order to maintain a safe and cost efficient operation of nuclear power plants, a detailed understanding of the ageing and degradation phenomena of various components is required. This is the case especially for components in the reactor containment. Studies of metallic materials and concrete components have been carried out for many years, and in the last decade interest has been drawn also to polymeric components. Polymeric materials have an important function in for example cables, insulation, membranes, linings and coatings. The main stressors for degradation of polymeric materials in nuclear power plant applications are temperature, radiation and the presence of oxygen.

During Energiforsk (Elforsk) workshops, research needs within polymeric materials in nuclear power applications was discussed. Following the workshops, a need for a better understanding of the status/remaining life length of polymer based components was identified. A feasibility study was initiated to investigate the prerequisites for a pilot study to develop acceptance criteria for polymeric materials.

The feasibility study was carried out by SP Sveriges Tekniska Forskningsinstitut. A reference group was formed to support the study, with participants from the nuclear industry in Sweden and Finland. The reference group are thanked for valuable comments, contacts and questions. The feasibility study was carried out within the Energiforsk Research Area Nuclear Portfolio, financed by E.On, Fortum, Karlstads Energi, Skellefteå Kraft, Teollisuuden Voima (TVO) and Vattenfall.

Sammanfattning

Bakgrund

Enligt IAEA, har drifterfarenheter visat att ineffektiv kontroll av åldersrelaterade försämringar på kärnkraftverkens huvudkomponenter kan riskera anläggningens säkerhet och livslängd. Åldrandet i kärnkraftverken måste därför hanteras effektivt för att säkerställa tillgången på konstruktionsfunktioner under hela anläggningens livslängd (IAEA, 2000). Som diskuterats vid ELFORSK seminarium om åldrande av polymera material vid NPP 2014 påpekades vikten av att definiera acceptanskriterier för polymera komponenter. Acceptanskriterierna bör baseras på funktionella krav, eftersom det är det första steget i processen av livstidsuppskattningar för både befintliga och nya material.

Vid ELFORSKs interna projektmöte enades det om ett gemensamt mål/vision att definiera aktiviteterna inom polymera material.

- Åldrande av polymera material har, tack vare ett effektivt underhållsprogram, ingen negativ inverkan på säkerheten eller tillgängligheten för driften av kärnkraftverket.

Man kom också överens om att fokus för gemensamma aktiviteter kan sammanfattas enligt följande:

- Förbättra utbyte av komponenter/upphandling - kompetent kund
- Förbättrad förståelse för status/återstående livslängd

Bland de elva intressanta områdena av gemensamma aktiviteter, bedömdes acceptanskriteriet som det mest intressanta för gemensamma insatser. (Widestrand, 2015).

Omfattning

Syftet med denna förstudie är att hjälpa till att fastställa/definiera de tillgängliga acceptanskriterier för en uppsättning komponenter som beslutats av de nordiska kärnkraftverken. Detta kommer att ske genom intervjuer med representanter från de olika anläggningarna, diskussion med referensgrupp och en litteraturstudie av internationella standarder och riktlinjer. Förstudien kommer att fokusera på systemkomponenter och specificera alla identifierade kunskapsluckor. En projektplan för en pilotstudie kommer också att sammanställas. Den kommer specificera hur man lär sig mer om de olika acceptanskriterierna för polymererna i de valda systemkomponenterna. Som en del av pilotstudien kan skrivandet av specifikationer också inkluderas.

Resultat

SP har utfört intervjuer tillsammans med de fem nordiska kärnkraftverken Loviisa, TVO, Forsmark, OKG och Ringhals. Med hjälp av intervjuerna har en uppsättning av komponenter identifierats (bilaga 3) och innehåller den information som erhöles och som finns tillgänglig för komponenterna. Detta inkluderar, men är inte begränsad till, typen av polymer och miljöförhållande, såsom värme och strålning. Baserat på komponenterna, och den typ av polymerer som används för det, har en litteraturstudie genomförts för att identifiera befintlig kunskap om åldrande mekanismer. Från den

kunskap som litteraturstudien gett och befintlig "know-how" vid SP, har tabeller med lämpliga metoder och föreslagna, om möjligt, acceptanskriterier noterats för varje komponent. Det var svårt att hitta redan definierade acceptanskriterier och därför har en pilotstudie föreslagits med mål att utvärdera acceptanskriterierna för komponenterna i appendix 3. Studien föreslår 7 arbetspaket som fortsatt forskning för att kunna ta fram acceptanskriterier för dessa komponenter.

Summary

Background

According to IAEA, operating experience has shown that ineffective control of ageing degradation of the major Nuclear Power Plant (NPP) components can jeopardize plant safety and also plant life. Ageing in NPP:s must be therefore effectively managed to ensure the availability of design functions throughout the plant service life (IAEA, 2000). As discussed at the ELFORSK Seminar on aging of polymeric materials in NPP 2014 the importance of defining the acceptance criteria for polymeric components was pointed out. The acceptance criteria should be based on functional demands since it is the first step in the process of lifetime estimations of existing or new materials.

At the ELFORSK internal project meeting a common goal or vision was agreed upon to define the activities within polymeric materials.

- Ageing of polymeric materials has, due to an effective maintenance management program, no negative impact on safety or the availability for operating the NPP.

It was also agreed that the focus for common activities can be summarized as:

- Improving exchanges of components/procurement – competent customer
- Improved understanding of status/remaining life

Out of the eleven interesting areas of common activities the acceptance criteria was rated as the most interesting for joint efforts (Widestrand, 2015).

Scope

The purpose of this feasibility study is to help define the available acceptance criteria for a set of components decided by the Nordic NPP:s. This will be done through interviews with the different sites, discussion with the reference team and through a literature study of international standards or guidelines. The literature study will focus on system components and detail any identified gap in knowledge. A project plan for a pilot study will also be compiled. It will detail how to learn more about different acceptance criteria for the polymers in the chosen system components. As a part of the pilot study writing of specifications can also be included.

Result

SP has performed an interview together with the 5 Nordic nuclear power plants Loviisa, TVO, Forsmark, OJG and Ringhals. Through the interview a chosen set of components were identified (appendix 3) containing the available information for the components. This includes but is not limited to type of polymer and environmental aspects such as heat and radiation. Based on the components, and the type of polymers used for it, a literature study has been performed in order to identify existing knowledge on aging mechanisms. From the knowledge gained through the literature study and existing know-how at SP, tables with suitable methods and proposed, if possible, acceptance criteria is concluded for each component. It was difficult to find already defined acceptance criteria and therefore a pilot study is proposed with the aim to evaluate this for the components in appendix 3. The study proposes 7 work packages as continued research in order to identify the acceptance criteria for the components identified during the interview.

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1 Ageing by radiation and heat

The materials identified for this feasibility study are subject to different temperatures and doses of radiation. Radiation resistance is characterized by the half value dose of significant mechanical properties, for instance tensile strength. This is the absorbed dose that reduces a property by 50% (Wündrich K. , 1977). As written in *Prediction of service lifetimes of elastomeric seals during radiation ageing* (Burnay, 1984) historically radiation tests on polymeric material, samples have been irradiated at high dose rate ~10 kGy/h (1 Gy = 100 rad) to obtain "lifetime dose". However, it has been well established that many polymers exhibit dose rate effects or synergism between temperature and radiation making such predictions of limited use. An exposure at high irradiation also concentrates the ageing to the surface, which can generate poor correlation to real use. To be able to estimate the lifetime of a component a study of the functional relationship between the properties measured and the accelerating parameters can be used (Burnay, S.G, 2001).

The synergism in degradation of a polymer when exposed to heat and radiation is further supported in *Degradation of elastomer by heat and/or radiation* (Masayaki, 2007) for EPDM and at 140/70 C and 5 kGy/h / 3 kGy/h. The test showed synergism for the degradation with an increase in rate when the material was subject to both factors (Burnay, 1984). According to Kuriyama et al (Kuriyama, 1979), the value of E_a for nonirradiated samples is typical of thermo-oxidative degradation process, however for irradiated samples the values of E_a are more typical of oxygen diffusion in polymers. In the referred tests the oxygen penetration of the cross-linked polyethylene was limited to the surface (~0.6 mm at dose rate of 0.1 kGy/h).

Due to the morphology of the material, distribution of antioxidants, oxygen availability etc. ageing is a heterogeneous process. Diffusion of oxygen is often a rate determining step and surface degradation is observed in several studies. Decrease in mechanical properties such as elongation at break is often observed as a consequence of surface degradation (Wündrich, 1985). Most incidents caused by failure of polymeric materials subject to radiation are related to elastomeric seals and electric cables. Even though many studies show a synergy between heat and radiation most of the failures are caused by thermal ageing rather than radiation ageing (Kotthoff, 1994).

In many studies for evaluation of radiation resistance for polymers rather high dose rates have been used and the exposure was performed in nitrogen atmosphere. During use in nuclear power plants the dose rate is rather low and oxygen is present during outage. Hence studies have shown that the main degradation mechanism of polymeric materials is in general thermo-oxidation (Wündrich, 1985).

Fillers can influence the effect of irradiation and has to be considered both for thermosets and elastomers. Most of inorganic fillers increase resistance to radiation induced degradation and organic fillers decrease the resistance for thermosets (Wündrich, 1985). Antioxidants increase the resistance to degradation and this is expected since the degradation mechanisms are similar for thermo-oxidation and degradation initiated by nuclear radiation. Aromatic antioxidants have proven to have better protecting effect compared to aliphatic (Wündrich, 1985).

High energetic radiation causes changes to polymer materials. Polymer chain scission occurs with radical formation and oxidation as a consequence. This will eventually lead

to polymer degradation and cross-linking, the process will depend on the polymer type and working environment.

The dose rate for the components in this study ranges from none, low (mGy/h) to high (0,1 – 0,5 Gy/h). These dose rates are far below what has been used in experimental trials identified in the literature study. Values are often in the magnitude of 10^3 times higher than what is estimated as High dose in a nuclear power plant and 10^6 higher than low dose (Interview, 2015).

2 Interview

To decide on a set of components interviews were conducted with the 5 Nordic nuclear power plants Loviisa, TVO, OKG, Forsmark and Ringhals, see appendix 2 for interviewees. During the interview each site was asked to provide a few components of interest prioritized in the following order:

- Polymeric parts in system components
- Seals in building structures
- Electrical cables

They were also asked to provide information about the component; type of material, working environment etc. A complete list of the input received from the interviews can be found in appendix 3.

The following components and materials were identified during the interviews as major and were chosen for the literature study in order to define available acceptance criteria.

- O rings
 - × Nitrile rubber
 - × Viton rubber
 - × Silicone rubber
 - × EPDM
- Lining in tanks and pipes
 - × Rubber (natural)
 - × Epoxy
- Reinforced EPDM/Chloroprene - Membrane valve
- Teflon seal in valve
- Basin Door, EPDM seal
- Joint sealants

The following chapter will present the findings of the literature study and will in detail describe the selected components and materials in regard to existing knowledge on ageing mechanism.

3 Materials

3.1 O-RINGS

3.1.1 General description

O-rings are used in many mechanical devices as a seal between rigid parts to create a flexible as well as gas and liquid tight interface. Important properties of o-rings are flexibility and elasticity in order to provide good sealing performance as they can be exposed to both dynamic and static load (Parker, 2007). In the nuclear power plants subjected to this study it is used in the cooling systems as a component in many types of pumps both for reactor water, steam, hydrogen and seawater; it is also a common component in valves.

3.1.2 Material Characteristics

Several materials are found in o-ring applications, the identified materials in the interview are described below.

Nitrile Rubber

Nitrile rubber is a copolymer consisting of Acrylonitrile (ACN) and Butadiene. Other names for Nitrile rubber are Buna-N, NBR and Perbunan. Trade names are for example Nipol, Krynac and Europrene. The properties are tailored by altering the amount of the different building blocks and different additives.

Nitrile is used in o-rings and is resistant to aliphatic oils and acids. The material is also very common in the automotive industry. Since Nitrile is unsaturated i.e. contains double bonds, it is possible to modify and improve the material. Hydration i.e. addition of hydrogen to the double bonds, improves the long term stability of the material. The ACN content affects the glass transition temperature and the oil resistance. A low ACN decreases the glass transition temperature and high ACN content improves the resistance to hydrocarbons. 33% ACN is a common composition and a good compromise for the different requirements.

Nitrile can be used in the temperature range -40 °C – 108 °C and is generally resistant to long term exposure to heat. The material degrades through an auto-oxidation mechanism similar to other hydrocarbons. Fourier Transform Infrared Spectroscopy (FTIR) spectra were studied (Jestin, 2000) on vulcanized Nitrile Rubber after ageing at 100 °C. Celina et al studied Nitrile rubber at ageing temperatures between 85 °C and 140 °C. Both studies states that hydro- peroxides, alcohols, acids and ketones are formed upon thermal ageing of nitrile rubber (Celina, 1998). The ageing of un-vulcanized Nitrile rubber is associated with the butadiene parts of the rubber, the amount of double bonds decrease and absorption bands associated with 1,2- and 1,4-unsaturations decrease. Residual double bonds in vulcanized rubber are also affected by ageing.

Oxygen diffusion limits the oxidation rate in a nitrile rubber and depth profiles were plotted with carbonyl group concentration as a function of depth in the sample. The FTIR data were achieved on thin samples microtomed from the sample surface and into the sample center. The heterogeneous diffusion of oxygen into the material is explained by the morphology in the material (Celina, 1998).

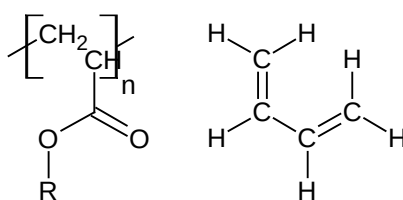


Figure 1 Co polymer of acrylic ester and butadiene.

Viton

Viton is DuPonts trade name for Fluoro elastomer rubber or FKM rubber. Other trade names for FKM are Dai-El and Technoflon. Fluoroelastomers are copolymers containing hexafluoropropylene (HFP) and Vinylidene Fluoride (VDF) or terpolymers of Tetrafluoroethylene, VDF and HFP. The fluorine content is typically 66-70%. They are used as o-rings and may replace NBR rubber when high performance products are needed.

Viton is very durable at high temperatures (up to 250 °C for 1000 hours) and in aggressive chemical environment. Viton is resistant to hydrocarbons and therefore often found in fuel systems in vehicles. The material is also durable in ozone and outdoor environment. It has been reported that Viton o-rings become hard and brittle upon high dose rates and soft and elastic upon low dose rates (Clough, 2003).

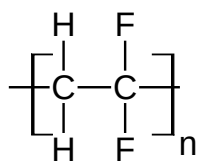


Figure 2 Viton monomer

Silicone

Silicone elastomers are based upon polydimethylsiloxane and a variety of reinforcing and extending fillers which are normally incorporated into the silicone rubber to achieve the desired set of properties (Toman, 2007). The main chain in silicone consists of silica and oxygen instead of hydrocarbons which is the common backbone in most polymeric materials. Silicone rubbers are mainly used when high temperature variation is expected and as an example a variation between -100 °C – 280 °C is possible. They have low strength but excellent flame, weather and ozone resistance. Silicone rubbers often have low tear strength and are not generally suitable for dynamic applications. Silicone swells in hydrocarbon oils and is severely attacked by sulphuric acid and water vapor at high temperatures. However weak alkalis, acids and alcohols leave silicon unaffected. The permeability rates of many gases are very high and are for instance 400 times higher than for butyl rubber.

Silicone elastomers can be engineered to work in different temperature spans or for a certain type of chemical which makes it important to specify the environmental conditions to the manufacturer.

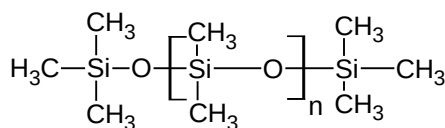


Figure 3 Silicone monomer

EPDM

EPDM (Ethylene propylene diene monomer) rubber is an elastomer and a terpolymer of ethylene, propylene and a smaller amount of diene monomers. EPDM is used in a wide range of applications because of its overall good performance in many different environments. And it is possible to design its properties by using different amount of the monomer and by using either sulfur or peroxide as vulcanizing agent. (Gummitechnologi, 2013)

EPDM is a commonly used elastomer for o-rings in nuclear power plants because of its good properties combined with low cost, but it is not normally used in the radiated zone (Toman, 2007).

EPDM shows high resistance to water and other polar fluids such as alcohols, ketones, esters, soaps, acid and alkali. It is also resistant to oxidation and ozone degradation. The performance in both high (up to 150 °C – 260 °C) and low temperature (down to -57 °C) is good and it has high resilience. It preserves its properties when exposed to up to moderate radiation. EPDM is a low cost material due to the ability to accept high amounts of oil extenders, and fillers (eg. silica, calcinated clay and calcium carbonate) (Toman, 2007)

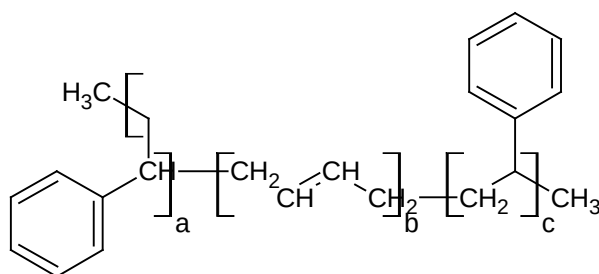


Figure 4 Ethylene propylene diene monomer

3.1.3 Working environment

Most o-rings are found in areas with low dose rate (app 1 mGy/h) and it could be assumed that thermal oxidative degradation is the dominant mechanism. It should be pointed out that the material used in nuclear power plants should be well stabilized with suitable antioxidant packages i.e. phenolic structures in order to function properly throughout the service life.

Literature data on Nitrile rubber exposed to high energetic radiation usually has the purpose to crosslink the polymer or modify the polymer surface. The irradiation was followed by degradation studies. The same structural changes of NBR polymer are observed both after electron beam irradiation and on thermo-oxidative ageing (Lopiteaux, 2003).

When installing o-rings an anti-seize lubricant can be used to help the installation. It is then important to be aware that a poorly chosen lubricant can accelerate the degradation of the o-ring. The proposed components are subjected to temperatures ranging from 40 °C – 100 °C and the media can be dry air, water, deionized water and nitrogen.

Different polymers have different resistance to high energetic radiation due to different molecular structure and additives e.g. antioxidants and stabilizers. Table 1 below shows relative radiation resistance for different rubber materials. This however depends on the quality of the rubber, the dose rate (Gy/h) and if it is exposed to oxygen. This means that values in Table 1 is valid for the material used for the referenced experiment. Most studies of radiation resistance of materials are made with high dose rates of about 1-10 kGy/h, although with the same total absorbed dose a lower dose rate at a longer exposure time is a tougher treatment (Toman, 2007).

Table 1 Table of relative resistance for different materials.

Material	Radiation resistance
Nitrile	Fair to good
Viton	Fair to good
Silicone	Fair to good
EPDM	Good to excellent
Natural rubber	Fair to good
Polychloroprene	Fair to good

3.1.4 Recommendations for the system component

Acceptance criteria and test method

To insure the quality of the o-rings, following properties can be tested on the o-ring: Hardness, Tensile strength, Elongation at break, Compression set, TGA, Stress relaxation etc. (see methods section). The standards SS 1587:2007 (based on ISO 3601-5:2002) - *Sealing elements – O-rings – Suitability of elastomeric materials for industrial application* or ASTM D1414 - *Standard tests for Rubber O-rings* can be used for the testing.

One possibility to measure the compression set on o-rings after usage is to measure the cross section thickness of a replaced o-ring 30 minutes after release. This would give an idea about compression set in a real usage of the product. Laboratory compression set measurements could be verified by leakage tests to correlate compression set values with a real function of the o-ring.

Table 2 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Ageing	Accelerated ageing with sampling.	Installation and buying	
Hardness	Can be measured directly on most o-rings, measures primarily the surface of the product	Installation, buying and during service of equipment.	Difference ± 10 from initial value
Tensile testing	Study cross linking. Method is sensitive to surface degradation, the product may function anyway.	Installation and buying	
Thickness	Can be both compression set and abrasion.	Installation, buying and during service of equipment.	
Compression set	Measuring the deformation of the cross section thickness of an o-ring and is a measure of the sealing performance	Installation and buying	50 % found in most standards, should be correlated to a leak test in a pressurized system.
Stress Relaxation	Measure counterforce of an o-ring under a specified deformation	Installation and buying	
Setting requirement	Correlation to compression set data, see project suggestion (sealing performance)	Installation and buying	
TGA/DSC	Antioxidant content	Installation and buying	Initial value

Technical specification

Properties that can be specified for elastomers:

Vulcanizing agent (EPDM), antioxidant content, maximum filler content, hardness, compression set, tensile strength, elongation at break, stress relaxation. Some rubber polymers (for example EPDM, NBR, Natural rubber and Chloroprene rubber) may be cross-linked or vulcanized by either sulfur compounds or peroxides. The different cross-link systems affect the final properties such as compression set and stability etc.

3.2 LINING IN TANKS AND TUBES (RUBBER)

According to the document Technical regulations for surface treatment edition 3 (TBY) a rubber lining can be used to protect tanks and pipes against corrosion and erosion from sea, fresh and deionized water. The lining should be able to manage the temperatures, fluid turbulence and pressure that the specific component will be subjected to. The document also specifies the rubber quality to be Natural rubber (also known as polyisoprene and Latex). This can be interpreted as being Natural rubber from rubber tree (*Hevea Brasiliensis*) and Natural rubber manufactured in a synthetic way.

Since the product rubber lining in tanks and tubes becomes a finished product when attached to the tank or tube it is not enough to study the material itself. It is also important to study the interface between the rubber and the carrier.

3.2.1 General description

Natural rubber is to be used for lining tanks and pipes subjected to water as written in Technical regulations for surface treatment edition 3. Since the knowledge on the type of rubber used at the different power plants was not detailed it is not possible to conclude that natural rubber was used. However in the below text an assumption has been made to that it is natural rubber both soft and hard and manufactured from tree and synthetic.

Natural rubber and polyisoprene share the same monomer chemistry. Both are found under the name natural rubber even though one comes from the rubber tree and the other from a synthetic process using different ways of polymerizing polyisopropene. Due to this there are many different qualities available on the market but they can be categorized into three main configurations: high cis content, high trans content and high 3,4 content. There are a number of important differences between natural and synthetic polyisoprene. Natural rubber has higher green (before vulcanization) strength and modulus, especially at higher temperature and strain. Synthetic polyisoprene is usually lighter in color, more consistent in chemical and physical properties and easier to process. It is also important to note that there are a variety of technical grades of natural rubber with different properties available (Hanser, 2001).

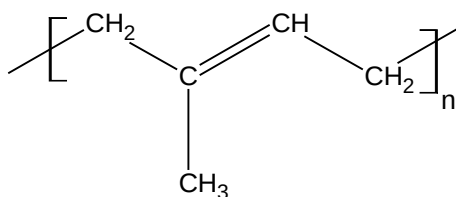


Figure 5 Polyisoprene monomer

3.2.2 Material characteristics

There are a variety of different characteristics that can be measured for natural rubber. For instance density, hardness, tensile strain – stress, low temperature properties, stress, relaxation, creep, adhesion and tear. Furthermore the characteristics can also be evaluated before and after the material is subject to heat, ozone, weathering and different chemicals to see how the characteristics changes.

In the TBY, edition 3 4.3.6.4 the quality of the rubber used for lining in pipes and tanks is specified and for instance thickness, times of calendered, adhesion and hardness is specified.

The quality control is most important during manufacturing and installation. Depending on the environment a time interval for checking the component is set. As for instance the quality control is now done every 7th year for the tanks at Forsmark.

3.2.3 Working environment

According to the interview the rubber lining is subjected to two different environments; sea water and deionized water. In both cases the material is also subject to oxygen. There is no radiation and the temperature is estimated to be at room temperature down

to inlet cooling water around 5-10 °C. During the interview it was not said how the surface was prepared prior to installation. However, TBY edition 3 states how the surface should be prepared which is assumed to the case for this tank.

In one of the tanks containing deionized water there has been an issue with an increased level of sulphate. It has not been possible to specify the origin of the sulphate.

Natural rubber has good elasticity as long as the temperature is kept above the glass transition temperature.

3.2.4 Recommendations for the system component

The current selection of natural rubber for using in tanks and pipes which is exposed to sea water or deionized water at temperature around room temperature ± 15 °C is a proper choice. However there are a few characteristics that are good to specify and verify when installing a new liner.

The product could be tested in accordance to the quality control time span used today, for instance every 7th year, although other industries often run in 5 year cycles in test programs. Recommended test methods before installing a new lining is hardness with Shore, tear, adhesion, thickness and TGA.

Acceptance criteria and test method

The following test methods are recommended. The standard can vary; see Description of test methods for more information. To be able to perform adhesion test the lining provider can line test plates (also used for thickness) on beforehand and send to a lab for testing of adhesion and TGA. The TGA test will then verify that the same material is used during installation (verification according to initial value).

Table 3 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Hardness	Used to specify the vulcanization of the material. Compare with value at installation.	Installation, buying and every 5 th year	Shore A 60 ± 5
Adhesion (Tank only) (ISO 4624)	In tanks the material need to stick to the surface (carrier) not to fall off.	Installation, buying and every 5 th year	>1 MPa
TGA	To verify the material data (fingerprint) Composition	Installation and buying	Initial value
Thickness	The thickness of the material is important for the tightness	Installation and buying	In accordance with supplier specified value
Bresle Soaking/leaching	Verify that substances are not leaching from the polymer. Surface cleaning, Adhesion	Installation	
Ageing	Accelerated ageing with sampling after 2, 4, 6 months in water and air.	Installation and buying	Adhesion
Test of water in tanks	The water is tested in concern of soaking leaching.	Installation and yearly	No foreign materials

Technical specification

In section 4.3.6 in TBY, edition 3 several important demands are made to the supplier of the lining material. In addition to these demands the requirements stated under Acceptance criteria and test method can be specified.

3.3 LINING IN TANKS AND TUBES (EPOXY)

3.3.1 General description

Epoxy polymers are thermosetting resins. Thermosetting resins create a thermal stable network with crosslinks, which are composed of covalent bonds between the molecules. The epoxy polymers are normally based on polyether formed in condensation reactions between bisphenol A and epichlorhydrin, and they require a large portion of curing agents. Most of the curing agents used are amines, thereby incorporating nitrogen into the molecular structure. The epoxy materials have a high concentration of polar groups in the molecular structure which gives these materials very strong adhesive forces. Due to the cross-linked nature of epoxy coatings, they have good mechanical properties, good chemical resistance, anti-corrosive properties and good thermal stability. Suitable applications for epoxy material are coatings, adhesives, encapsulation, impregnation and reinforcement (Parkinson, 1970).

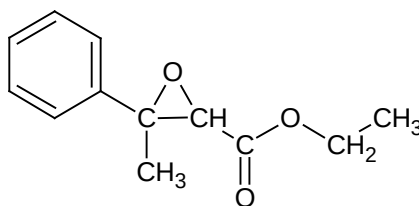


Figure 6 Epoxy monomer

3.3.2 Material characteristics

The formulation of the epoxy coating affects the chemistry of polymer chain formation and molecular weight. The final form of the polymer chain (its length, shape, and configuration) determines the properties and physical characteristics of the coating, such as flexibility, hardness, and adhesion. Another important characteristic of the epoxy coating is that they tend to absorb water over time. This will contribute to loss in adhesion to the metal. Another contributing factor to adhesion loss is discontinuities in the coating. It has been shown that the agents causing disbondment migrated to the coating-substrate interface through the coating defects rather than through the bulk of the coating (Parkinson, 1970) (Lee, 2006).

3.3.3 Working environmental

According to the interview the epoxy polymers are exposed to two different environments. OKG is using the epoxy material as lining of tanks made of carbon steel and in coolant systems. Ringhals is investigating the use of epoxy paint in tanks. These linings are subjected to water and the temperature is estimated to be between 5 °C and 50 °C. At Loviisa the environment the epoxy material is subjected to is sea water, oxygen and the temperature is estimated to be between 4 °C and 25 °C. In none of the two environments the material is subjected to radiation.

3.3.4 Recommendations for the system component

It is a known concern that the presence of moisture (water) around the epoxy can be harmful, since water is one of the most destructive agents for metal/polymer adhesion strength. If exposure period is sufficiently prolonged, the presence of water in the interfacial region is believed to produce large reduction in adhesion. (Vaca-Cortés, 1998). However the current selection of epoxy coatings for lining of tanks and pipes seems to be a proper choice. In Loviisa where the epoxy is used as corrosion protection in sea water pipes, it has been replaced with hard rubber

Acceptance criteria and test method

Ringhals states that they are concerned about hardness, lifetime, and soaking leaching. Hardness could be one of the characteristics that are suitable to use as an initial quality control in order to evaluate that the product has the correct formulation.

Electrochemical impedance spectroscopy (EIS) can be used to give indications of defects and electrolyte take-up by the coating.

Water tests are performed continuously due to the concern of soaking leaching.

For this reason control of discontinuities would be a good quality control at installation.

The following test methods are recommended. The standard can vary. As for the rubber linings in tanks the lining provider can line test plates (also used for thickness) in advance in order to be able to perform adhesion test (SigmaGuard).

Table 4 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Ageing	Accelerated ageing with sampling after 2, 4, 6 months in water and air.	Installation, buying and every 5 th year.	50% Bend test Adhesion
Hardness	To test that the formulation is properly mixed	Installation, buying and every 5 th year	Initial value
Bresle test	Test of soaking leaching, before filling the tank with water.	Installation and every 5 th year.	Not including chemical substances not allowed
Porosity/defects in coatings pinholes etc.	Ensure that the painted film is homogenous and free from defects (holidays, pores and craters)	Installation	
Film thickness	To assure enough thickness for water tightness.	Installation and buying	The mean value for each individual test surface must not exceed double the nominal thickness. In accordance with supplier specified value

Test method	Comment	Frequency	Acceptance criteria
Test of water in tanks	The water is tested in concern of soaking leaching.	Installation and yearly	No foreign materials
Adhesion ISO 4624 (tank only) In tanks the material need to stick to the surface (carrier) not to fall off. Installation and every 5th year	The adhesion is of important to test to analyze if any oxidation of the carbon steel has occurred.	Installation and every 5 th year.	>1 MPa
FTIR	To verify the material data (fingerprint)	Installation and buying	Initial value
Tensile test	Test the function of the material.	Installation and buying	Done before and after ageing. Degradation of <50%.

Technical specification

In section 4.3.6 in TBY, edition 3 several important demands are made to the supplier of the lining material when using rubber. These can partly be used for epoxy as well. The recommendation is to at least use the criteria stated under Acceptance criteria and test method.

3.4 REINFORCED EPDM/POLYCHLOROPRENE - MEMBRANE VALVE

3.4.1 General description

Membrane (diaphragm) valves are used at many locations in NPP:s as a component for flow control. These valves are linear movement types and it work as follows: The stem of the valve is used to push down a flexible membrane, which in turn blocks the path of the fluid. The main advantage of the membrane valve is that the membrane isolates the moving parts of the valve from the process fluid. They are therefore suitable for handling aggressive fluids and for those containing suspended solids. The possibility to use many different membrane materials enables membrane valves to be used in many different environments. Their application is however limited by the temperature that the membrane can withstand.

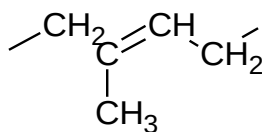


Figure 7 Chloroprene monomer

3.4.2 Material characteristics

Reinforced EPDM is used as membrane material in membrane valves of Grinnell type at Ringhals power plant. This membrane is reinforced with polyamide 6 (PA 6).

The characteristics of EPDM are described under the section o-rings above. The reinforcement PA 6 is a linear aliphatic polymer with high tensile strength and elasticity (Brydson, 1995). The reinforcement in this membrane is present as a single layer woven sheet completely incorporated in EPDM. It is placed near the surface opposing the flow side which shields it from the environment and is of less importance for the lifetime of the membrane. The adhesion between the rubber and reinforcement is important and lack of adhesion is a common failure mechanism.

Polychloroprene is used as membrane material in membrane valves at many locations in OKG, Ringhals and Forsmark power plants. Polychloroprene (Neoprene) is considered a high-performance multipurpose elastomer product due to its inherent balance of good mechanical and physical properties. It is resistant to oil, wax and grease, and can withstand temperatures from -50°C to 120°C. It is also resistant to ozone, weathering, and water immersion (Toman, 2007).

3.4.3 Working environment

The membrane valves are used in the power plants for control of water and gas flow. The material is normally exposed to temperatures of 40 °C to 100 °C and the media can be reactor water, deionized water or sea water. It is subjected to radiation in some cases but normally low dose (mGy). The membrane is subject to compression at the sealing edge, dynamic pressure and flexing when operated and vibration and pressure from turbulent flow.

Anti-seize lubricants can be used to help the installation of the membranes and therefore it is important to know that the agent will not accelerate the degradation of the material.

3.4.4 Recommendations for the system component

Acceptance criteria and test method

The same methods and standards that were specified for o-rings in the section above can be used for setting a value to insure the quality of the material in the membranes

Table 5 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Hardness	Can be measured directly on the membrane, measures primarily the surface of the product	Installation and buying and during service of equipment	Difference ± 10 from initial value
Tensile testing	Study cross linking, method is sensitive to surface degradation,		
Compression set	Measuring the deformation of the cross section thickness of a membrane and is a measure of the sealing performance	Installation and buying and during service of equipment	50 % found in most standards, should be correlated to a leak test in a pressurized system.
Stress Relaxation	Measure counterforce of a membrane under a specified force	Installation and buying	
TGA/DSC	Antioxidant content DSC TGA composition	Installation and buying	Initial value

Technical specification

Properties that can be specified for elastomers:

Vulcanizing agent (EPDM), antioxidant content, hardness, compression set, tensile strength, elongation at break. Some rubber polymers may like EPDM be cross-linked or vulcanized by either sulfur compounds or peroxides. Peroxide cross-linked materials have better compression set properties.

3.5 TEFLON SEAL

3.5.1 General description

Teflon is a trade name for polytetrafluoroethylene (PTFE) and is a completely halogenated ethylene form. The PTFE has a simple molecular structure and moderately strong intermolecular forces. It is used as a common engineering material for small high-performance parts where their inertness and high temperature resistance is of importance. Examples of applications are; tubing, gaskets, seals, containers and for electrical coatings. PTFE has low coefficient of friction and it is almost impossible for other materials to adhere to, which makes it useful for unlubricated bearings, sliding surfaces and industrial processing technology where ease of cleaning is important. When PTFE is sintered with wear reducing compounds, an industrially important class of bearing materials is formed. One aspect of PTFE that has held it back from more extensive industrial and engineering use is its high melt viscosity. This prevents injection and blow molding from being possible and only expensive sintering and extrusion manufacturing processes are available for part production (Parkinson, 1970) (Rae, 2004).

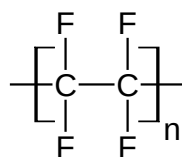


Figure 8 Polytetrafluoroethylene monomer

3.5.2 Material characteristics

PTFE exhibits useful properties over the widest temperature range of any polymer and it is useful to exceptionally high temperature up to 250 °C. PTFE has good tensile strength and is tough, and also extremely chemically inert. It is insoluble in all common solvents and is resistant to almost all acidic and corrosive materials. PTFE has amongst the highest resistivity of any material, a very high dielectric strength and low dielectric loss.

PTFE and other fluoropolymers behave differently compared to their hydrocarbon similarities in radiation chemistry. This is mainly due to the distinctive characteristic of the C-F bond. The stability of the C-F bond makes a transfer of the fluorine atom highly unlikely when exposed to radiation. Of this reason PTFE undergoes mainly chain scission when irradiated at room temperature in air and to less extent, in vacuum, after exposure to very low doses. This is characterized by a dramatic drop in molecular weight and since the mechanical properties are only maintained at a high molecular weight the mechanical properties are lost.

When PTFE is irradiated in absence of oxygen and above the crystalline melting point of 330 °C crosslinking occurs, which results in an improvement in mechanical properties, radiation stability and optical properties. (Parkinson, 1970) (Rae, 2004) (Forsythe, 2000).

3.5.3 Working environment

According to the interview the environment the Teflon polymer is subjected to is de-ionized water in the temperature range of 50 °C and 224 °C, and the pressure between 62 – 95 bar. It is not subjected to radiation. It has however been asked to study the effect of radiation on Teflon materials used in the sites due the fact that Teflon is highly sensitive to radiation.

3.5.4 Recommendations for the system component

Acceptance criteria and test method

Among the mentioned properties some are measurable characteristics of Teflon; for example tensile strength and coefficient of friction. According to the interview the Teflon material is used as a coating for a carbon steel spring used in sealing for a drag valve, therefore it is possible to think that it is exposed to abrasion in this application. It is also known that Teflon is a material that sets over time. Both abrasion and setting are characteristics that are of importance when it is used in sealing applications, since this will decrease the sealing force. There are tests to measure setting in a material, but it is also possible to measure a dimensional change. The abrasion loss can be measured by means of weight loss or visual control.

Table 6 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Abrasion (mass loss/visual)	A character related to sealing force	Service of equipment	
Tensile strength		Installation and buying	
Ultrasound/x-ray	Verify internal cracking.	Service of equipment	
Compression set/Abrasion test	Compression set/abrasion causes dimensional changes and leakage	Service of equipment	Compare dimensions to initial value

Technical specification

The recommendation is to at least use the criteria stated under Acceptance criteria and test method. However it could be good to include the abrasion resistance as well.

3.6 BASIN DOOR

The basin door has a valve using EDPM as sealing material. This is used when loading fuel into the reactor and is subjected to water and radiation. Since the elastomer itself is EPDM and has been described in chapter 3.1.2 this chapter excludes General description and Material Characteristics.

3.6.1 Working environment

The valve is subjected to deionized water at temperatures between 30 °C and 50 °C. At times the valve is also subject to high doses of radiation (0,5 Gy/h) during a period of 40 hours.

3.6.2 Recommendation for the system component

The recommendations are very much similar to the ones for EDPM in o-rings. It may be the environment that differs but still the following properties should be taken into account: Hardness, Tensile strength, Elongation at break, Compression set, TGA, stress relaxation etc. (see methods section). Laboratory compression set measurements could be verified by leakage tests to correlate compression set values with real function of the EPDM seal.

Table 7 The table demonstrates recommended test methods to be used for the polymer and its application.

Test method	Comment	Frequency	Acceptance criteria
Hardness	Can be measured directly on the product depending on placement, measures primarily the surface of the product	Installation, buying and during service of equipment.	Difference ± 10 from initial value
Tensile testing	Study cross linking, method is sensitive to surface degradation, the product may function anyway.	Installation and buying	
Thickness	Compression set.	Installation, buying and during service of equipment.	

Test method	Comment	Frequency	Acceptance criteria
Compression set	Measuring the deformation of the cross section. It is a measure of the sealing performance	Installation and buying	50 % found in most standards, should be correlated to a leak test in a pressurized system.
Stress Relaxation	Measure counterforce under a specified deformation	Installation and buying	
Setting requirement	Correlation to compression set data, see project suggestion (sealing performance)		
TGA/DSC	Antioxidant content, TGA composition	Installation and buying	Initial value

3.6.3 Technical specification

Properties that can be specified for elastomers:

Vulcanizing agent, antioxidant content, hardness, compression set, tensile strength, elongation at break.

3.7 JOINT SEALANTS

3.7.1 General description

Joint sealants are installed everywhere in the building structures of the plants. They are used to seal expansion joints between concrete building elements, around doors and for sealing cable entries and pipe penetrations through walls. The advantage of sealants compared to other sealing products is the very tight sealing through a chemical bond. A rubber sealing product cannot become that tight.

The functions of the sealants installed in the plants are to stop air, water, steam, heat and fire gases to spread between rooms.

Joint sealants are thermoset plastics produced on site. Replacing old seals is time consuming and dusty as it requires grinding of the adhesion surfaces. Good durability and sufficient properties are therefore very important.

It should always be considered if another sealing product can replace a sealant if it is likely that the seal needs to be opened again, typically for cable penetrations.

3.7.2 Material characteristics

Mainly four different basic materials are found in installed sealants:

- *Silicone*
Mainly found between concrete wall elements and as fire seals. High quality silicone sealants can be very durable. Unfortunately there are also low quality products on the market. Their disadvantage is that they can never be replaced with another type of sealant, as nothing else will adhere to silicone residues.

- *Polyurethanes*
PU is common in old installations between concrete elements. Polyurethanes can be sensitive to high temperature and humidity, making them unsuitable for some environments. PU-sealants in general need a primer as protection against concrete. A disadvantage with PU for installing personnel is the allergenic properties.
- *Acrylic*
Acrylate sealants are mainly found as fire seals. They are in general less flexible than other sealants. Some acrylics can be applied directly on concrete, some need a primer.
- *Hybrid*
Hybrid sealants is a relatively new group. The most common products are based on MS-polymer (trade name Kaneka for Silane terminated polymer). The general chemistry is that the polymer backbone is terminated with silane groups, making it possible to cure it with humidity as a silicone. As the polymer backbone varies a lot between different products, the durability properties also varies a lot. MS-polymer has showed to have good general durability properties. A disadvantage with hybrid sealants is often high viscosity, making them harder to install perfectly. High viscosity is bad for the wetting of the surfaces and thereby the adhesion.

3.7.3 Working environment

Many sealants can be sensitive to higher temperatures and/or humid environments. That must be considered when installing new seals but it can also be a problem for installed seals during some kind of possible accident conditions. Radiation resistance of sealants is barely studied at all. It is not likely that this is needed but this has not been investigated in detail. In general, results from studies of radiation properties of silicone rubber can be applied on silicone sealants.

3.7.4 Recommendation for the system component

The possibility that a joint sealant cannot resist the conditions under possible accident conditions needs consideration. It is relatively easy to find the installed products and evaluate these properties in laboratory.

Monitoring status of installed sealants is possible. In most cases the adhesion is the reason for failure of a joint. There is a standard adhesion test prescribed by the Swedish branch organization SFR (SFR, 2012) that can be used.

If the adhesion is sufficient, the polymer can still be quite degraded. This affects the possibility to withstand pressure differences over the seal. In this case the sealant often becomes sticky and soft. Therefore manual and visual investigations are relevant completing activities.

3.7.5 Technical specification

The European standard EN 15651-1 specifies façade sealants. The product class for indoor use, should not be considered relevant for industrial use. The classes F 25 LM and F 25 HM are more suitable. Those are classes for high mechanical strength and flexible products. EN 15651 is the product standard for CE-marking.

If the seal has a fire protection function there is a separate CE-marking according to ETAG 026 for these products.

There is no real consideration of long-term properties in the above mentioned standards. This must be evaluated in some other way. If only a few products are installed in Nordic NPP:s this might be a subject for a research project.

4 Descriptions of test methods

4.1 RADIATION EXPOSURE

To simulate the effect of long term radiation it is possible to carry out laboratory exposures on material samples. Diffusion limited oxidation (DLO) effects needs to be taken into consideration when setting up the radiation exposure test. Since it is more dominant on the surface of the material the sample thickness can affect the long term radiation properties.

In literature dose rates are usually given for specific samples. This is relatively easy to measure with an error of $\pm 10\%$. The absorbed dose on the other hand is more difficult to measure due to the morphology, structure and composition of the samples. The error of measured absorbed dose rate may be up to $\pm 20\%$ (Wündrich, 1985).

The effects of radiation are evaluated typically with some mechanical test method, measuring material strength and spectroscopy methods to study changes in chemical structure.

4.2 COMPRESSION SET

The most central property of sealing rubber products is compression set. Compression set is a test method (ISO 868) in which a circular rubber piece or an o-ring is compressed by typically 25% of initial thickness. The compression set rig may be stored at temperature relevant to the service environment. After a specified time the test piece is released and allowed to relax. The thickness is measured before and after compression and exposure. The material of good quality should retain its initial thickness or almost the initial thickness. Compression set is normally caused by subsequent crosslinking reactions and occurs during relatively short period depending on the temperature. Upon polymer degradation, which is the long term process the elasticity decreases and as a consequence, the sealing performance decrease. Poor compression set may also be caused by crystallization or high filler concentration.

Knowing the dimensions of installed sealing products like o-rings, it is possible to measure actual compression set by measuring the dimensions after demounting. It does take some practice to perform this in a repeatable way but it could be a usable method. The timing is important, meaning that the sample must relax a specific time after demounting typically 30 minutes.

4.3 STRESS RELAXATION

A complementary measurement to compression set is stress relaxation. This is a more direct way to measure sealing properties of rubbers. Instead of dimension change, the sealing force is measured over time. When the sealing force is zero there are no physical sealing. Compression set is more common to measure as it is easier, but stress relaxation gives a better picture of sealing properties. In dynamic environments with changing temperature or dimensional fluctuations, or in chemical environments that can give physical changes in the material, stress relaxation is preferable.

It is possible to carry out an approximate measurement of actual stress relaxation on demounted sealing components by compressing the component to the actual seat height and measure the force after the initial relaxation. Another possibility is to run

stress relaxation in cyclic temperatures if the working temperature is varying in the product application.

A common standard is ISO 3384.

4.4 HARDNESS

Hardness is actually measured on the surface part of a product and may be used for surface degradation studies. The ageing of rubber is correlated to increasing hardness. Some plastics can also show such correlations.

The technique is based on the measurement of how deep a certain needle can penetrate a surface within a certain time. The most common standards are Shore (ISO 868) for plastics and IRHD (ISO 48) for rubbers. There are handheld “durometers”. Those tend to generate results with poor repeatability. Education is needed. When it is possible to remove some material and measure the hardness in laboratory, it is preferable.

Hardness can sometimes correlate to measurements of modulus in tensile tests. An advantage is that small samples can be used and it can be done on one final product, for instance o-ring.

4.5 TENSILE STRENGTH

A tensile test is to pull a test sample until it breaks and record the force. It can be carried out on both products and more commonly material samples.

Tensile test results are very common on product data sheets. One must have in mind that it is hard to know under which circumstances and parameters those results were obtained. When tensile testing a plastic, parameters like temperature and test speed can easily affect the result 50 %. Reference values are therefore preferably obtained from own measurements but can still often be found elsewhere and used with some caution.

The most common standard for plastics is ISO 527 and for rubbers ISO 37. Bending tests are often correlating to tensile tests and can be used as an alternative when preparation of test samples can be difficult. ISO 178 is a common standard for bending tests of plastics.

4.5.1 Ultimate strength

The maximum measured force divided by the minimum cross section area is the ultimate strength of the material. For plastics and rubbers it is measured in MPa.

Ultimate strength can be used to measure degradation and ageing but it is seldom to recommend as the correlation is weak and a decrease is often quite sudden.

4.5.2 Elongation at break

A better property to correlate to ageing is elongation at break. It is the maximum possible elongation of the material before it ruptures, measured in percent of the initial length. Normally a polymeric material has decreasing elongation at break when ageing. During ageing of semi-crystalline polymers the tie molecules between the crystalline regions break and cause a significant decrease in elongation at break. An exception can be thermosets that are not fully hardened in production.

One must keep in mind that elongation at break is sensitive to sample preparation for reproducible results. Samples can be mill cut, water cut, stamped, cut with knife and so on.

4.5.3 Modulus of elasticity

The modulus or stiffness of the material can be measured at small deformations when the material is still elastic and not plastic. The modulus of elasticity is the increase of stress (MPa) at a certain increase of strain (mm/mm), commonly measured in MPa. Most polymeric materials have increasing modulus with ageing. Modulus can often be tricky to measure. There are a lot of practical parameters that affect the results.

An alternative to modulus of elasticity, that are commonly used for rubber materials, is to measure stress at a certain strain, like stress at 100 % elongation. Even if this can be far above the elastic region it can generate reliable reproducible results. An elongation of 100 % is commonly chosen for rubbers but for plastics an elongation of 1-10 % can be used.

An advantage with measurement of modulus or some other stiffness is that it is far less sensitive to sample preparation than elongation at break and ultimate strength.

Modulus in tensile correlates to modulus in compression. It can sometimes be easier to prepare samples for compression tests.

The modulus can sometimes also correlate to hardness.

4.6 TEAR STRENGTH

Tear strength is commonly used to evaluate the strength of rubber materials. Some materials can be specifically sensitive to tearing. A common test specimen is a sheet that is stamped to a trousers-shaped test piece. The “legs” are pulled in a tensile tester. The average force per sheet thickness is recorded. A common standard is ISO 34.

Tear strength can correlate to ultimate strength in tensile tests.

4.7 ADHESION

The adhesion of coatings and linings to the substrate can be measured in different ways. The most common method is a perpendicular tensile test with a circular “dolly” glued to the coating. This can be carried out according to ISO 4624 using a hydraulic device to apply tensile force. The maximum force is converted to adhesion per area (MPa).

An alternative for hard and thin coatings is the cross-cut test according to EN 2409. A cutting device is used to produce a right-angle lattice pattern. An adhesive tape is applied and removed from the pattern. A visual inspection is carried out and the loss of adhesion is compared to a picture scale.

Soft and thick materials like joint sealants and thick rubber linings can be tested for adhesion in a quite practical manner. A strip of the material is cut loose. One end of the strip is also cut loose from the substrate. The free end is slowly pulled. If the strip breaks but not the adhesion it is a good result. This method is prescribed for façade sealants in Sweden (SFR, 2012).

All methods for adhesion measurements have poor repeatability. There are modern devices for ISO 4624 producing acceptable results but still the measurements will be approximate, $\pm 25\%$ is normal.

4.8 IR-SPECTROSCOPY

IR-radiation excites molecular bonds causing absorbance of the radiation. Scanning through the IR-band generates an absorbance diagram. From this a lot of chemical information about a material can be obtained. This is carried out using an IR-spectrophotometer, usually designated Fourier Transform Infrared Spectroscopy (FTIR). When it comes to ageing, oxidation can be studied by selection frequencies that are absorbed by oxygen-carbon bonds. Ageing generates increased absorbance for those frequencies. It is necessary to have reference values from new material as well as some kind of correlation to physical properties.

Using a reflective absorbance technique like ATR it is possible to measure IR-absorbance on very small samples ($2 \times 2 \times 1$ mm), making the method useful for installed components.

For coatings intended for corrosion protection monitoring oxidation can be a technique to early indicate water absorption and penetration. An oxidized material is more hydrophilic.

Radiation causes surface degradation and therefore depth profiling is valuable to get information about how deep the degradation has progressed. It might be possible to slice a sample with a microtome for this purpose. On thicker materials a cross section can be analyzed.

A common problem with IR-analyses is interference from additives like carbon black and fillers absorbing too broad in the IR-range. Black rubbers can be pyrolyzed prior to analysis to avoid this problem.

4.9 DSC

Differential scanning calorimetry (DSC) is a thermal analysis technique. A sample is heated at a constant temperature rate and the energy consumption is recorded. All processes that require or generate heat, like crystallization melting or chemical reactions, can be detected.

It is also possible to study oxidation reactions. To protect polymers from ageing by oxidation anti-oxidants are added. How well they work or if they are consumed can be analyzed using DSC with oxygen atmosphere. Something between a very fast ageing and a very slow combustion is carried out, measuring OIT (Oxidative Induction Time). A common standard is ISO 11357-6. Reference values are necessary.

A very small sample is needed for DSC, about 5 mg, making it possible to sample installed components.

4.10 TGA

Thermo-gravimetric analysis (TGA) is another thermal analysis technique. A sample is heated according to a constant temperature ramp while the loss of weight is recorded. The technique is common for rubbers as they are blends of components. The relative

content of the components can be measured. A standard component of rubbers is a plasticizer, which is a low molecular weight chemical making the rubber softer. They can be lost by diffusion, evaporation and extraction during the service life of the rubber, making the material shrink or lose function in some other way.

Plasticized PVC can also be analysed for loss of plasticizer. This is a main mechanism in the ageing of soft PVC products.

It might be possible to find reference values for plasticizer content on installed components. Plasticizers evaporate around 300 °C and polymers pyrolysis at around 600 °C.

4.11 BLISTERING, CRACKING AND FLAKING

Coatings are often visually evaluated for cracking, flaking or blistering. When the damages are of those types it is often time to replace the product. There are standards to evaluate the degree of damage, like the ISO 4628 series.

4.12 ABRASION

Abrasion can be measured in laboratory as a material property. That can be useful for material comparison. There are standardized methods.

On installed components it is more suitable to try to measure the actual material loss. Components that are subjected to an abrading environment, like valve seals or dynamic o-rings, could be weighed or a material thickness could be measured.

4.13 COMPRESSION SET

Compression set is a test method (ISO 868) in which a circular rubber piece or an o-ring is compressed by typically 25% of the initial thickness. The compression set rig may be stored at temperature relevant to the service environment. After a specified time the test piece is released and allowed to relax. The thickness is measured before and after compression and exposure. The material of good quality should retain its initial thickness or almost the initial thickness. Upon polymer degradation, the elasticity decreases and as a consequence, the sealing performance decreases. Poor compression set may also be caused by crystallization.

Compression set is a measure of sealing performance of o-rings and gaskets. It is however somewhat weak. A heavy compression set can sometimes still result in good sealing properties. It is valuable to correlate compression set values by function tests of tightness in specific test rigs.

4.14 WATER ABSORPTION

Linings are mainly corrosion protection and an important property is water tightness. When the material oxidizes it becomes more hydrophilic and can absorb more water, which can lead to corrosion of the substrate and loss of adhesion.

To measure water uptake or oxidation can be a good early method to avoid corrosion problems.

Water uptake can be measured by sampling the material, weight it and dry it in laboratory. There are standard methods like ISO 62 for this.

An even smaller sample is needed to measure oxidation with FTIR and measure peak height for carbon-oxygen bonds. The suitability of this method is depending on polymer type and interfering additives.

4.15 POROSITY

Linings are produced on site. To check the produced polymer film for pores and too thin layers a porosity meter can be used. A metal brush is swept over the whole surface. High voltage is connected to the brush and the lining substrate is connected as the other terminal. Pores will give electrical discharge through the material.

The method is normally used at installation but can also be used searching for cracks in older linings.

An established standard is ISO 29601.

4.16 FILM THICKNESS

There are some possibilities to measure thickness on linings and other coatings. ISO 2808 contains over 20 methods to measure coating thickness, both destructive and non-destructive methods. Different methods can generate somewhat different results. One method should be consequently used.

5 Pilot study

5.1 MATERIAL SELECTION

The material that has been selected for the pilot study is specified under each work package.

5.2 PROJECT PLAN AND BUDGET

Overall aim is to help develop new methods for nuclear power plants to determine the acceptance criteria for a polymeric component. It is also to teach in regards to using the methods and the compilation of technical specifications. Since there are gaps in knowledge verifying tests are needed and proposed in the work packages below.

The project is planned to be done in cooperation between institute, university and industry and leads by SP Technical Research Institute of Sweden. The work is divided into different work packages (WP) where WP1-WP3 has a main focus in developing acceptance criteria.

The other WP:s were identified as interesting but lay somewhat outside the scope of this feasibility study. The intention of WP4 is to use it as a real comparison and correlation to the test done in WP1-WP3 but a pre study is needed to see if that is the case. WP5 could give a deeper understanding in the ODL effects from radiation and how much effect it has on degradation of polymers during operation. WP6 is proposed since a poor choice in lubricant may change the life time for the polymeric component and WP7 intends to educate staff involved in developing technical specification in existing knowledge at SP and the knowledge gained from the work in WP1-WP3.

Budget (kSEK)

WP	Year 1		Year 2		Year 3		Total	Comments
WP1		910	910	910			2 730	3 components (130 is one time cost)
WP2		175	175				350	10 components
WP3				150	150		300	1 epoxy, 1 rubber
WP4			125	125			250	5 components
WP5			50	80			130	1 component
WP6				125	125		250	3 components
WP7			110				110	

WP0: Coordination and project management. This package also includes regular discussions with the reference team.

WP1: Development of condition monitoring methods for o-rings and seals including low dose radiation exposure. Perform accelerated ageing through heat and radiation to components. Test for a specific property, compression set for instance, and correlate it to a function test for tightness of an o-ring. By doing so a correlation between

compression set and tightness can be achieved. To be able to better compare the effect the radiation has on degradation a parallel test in heat will be done.

The o-rings will be mounted in compression set test rig as well as in tube connectors during exposure. Compression set after certain exposure times will be measured and the pipe connections will be tested by mounting them in our hose testing equipment and the sealing performance will be measured as water pressure without leakage.

By testing the correlation between compression set and tightness of an o-ring an understanding of the function (tightness) based on a material property (compression set) can be made. The aim is to be able to use this to set acceptance criteria for an o-ring using compression set as a property.

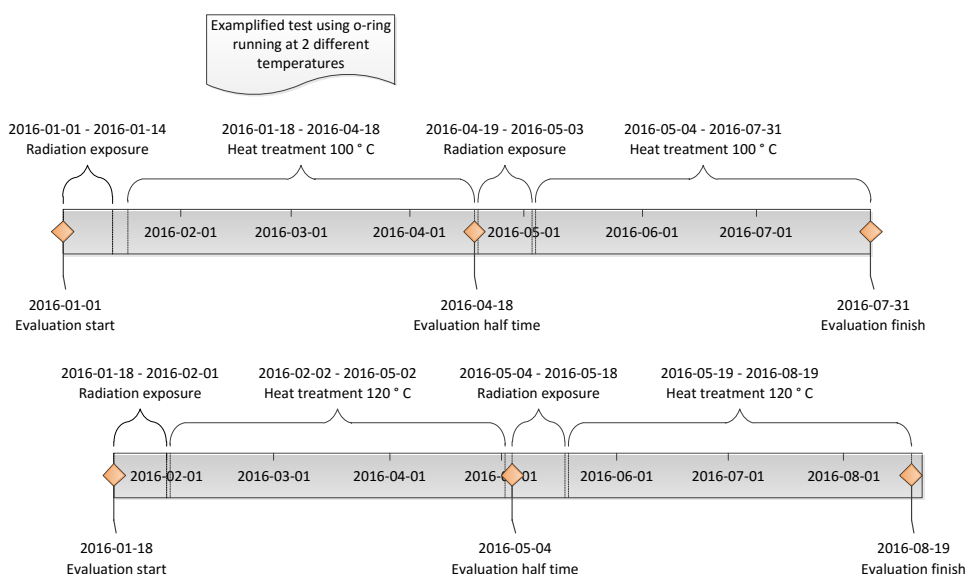
Other evaluation methods are tensile testing, thermal analysis (TGA, DSC) and if necessary chemical analyses such as Gas-Chromatography Mass-Spectrometry (GC-MS) and elemental analysis. DSC has been identified as a valuable method since antioxidant content is indirectly measured and indicates the residual service life of a product. Another benefit is that only small samples (5 mg) are needed. Since the knowledge about the material composition is often limited it is valuable to analyze composition (amount of plasticizer and filler), type of antioxidant and vulcanization system.

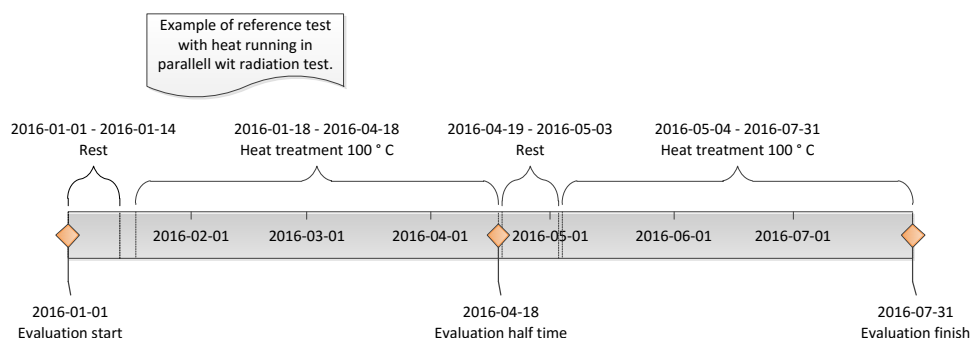
To be able to expose samples to realistic low radiation doses a modification/new build of a radiation chamber is necessary to be able to run in doses from 1 Gy/h to 10 kGy/h.

The following materials are recommended to be included in WP1:

- O-rings (Nitrile, Viton, Silicone, EPDM), subject to low radiation.
- Teflon seal, subject to low radiation
- Reinforced EPDM, subject to low radiation

Below time lines shows the sequence of tests to be completed for one component including three different test temperatures, two different radiation levels and a reference test without radiation exposure. The reference test will be completed for each chosen temperature.





The result may be possible to use in an update of the document Tekniska Bestämmelser för Mekaniska anordningar (TBM). Depending on the components identified in WP4 a comparison can be made to the accelerated test here in WP1. Presentation and discussion of the results is included in the work package.

WP2: Joint sealants are present in our nuclear plants everywhere in the building structures. Their function is to seal off different rooms to stop gases, water or fire from spreading. The knowledge around installed sealants is low. Their ageing properties are mostly unknown and acceptance criteria have seldom been set. Only products directly intended for fire protection are well documented. The WP will focus on screening installed sealants for ageing properties. Also the behavior under accident stresses, with high temperature and humidity, will be evaluated. Possible methods to evaluate status of installed sealants will be developed together with acceptance criteria. Methods and theory can partly be derived from the previous Energiforsk study "Fogmassor i betongkonstruktioner – typer, åldringpåverkan och inspektion". This work package will focus on actually installed sealants at indoor applications and criteria for replacement sealants. A smaller similar project has been carried out by SP for Ringhals.

The practical work will be simulations of ageing in a realistic time perspective, simulation of accident stresses and evaluation of the sealing function of test joints.

Presentation and discussion of the results is included in the work package.

WP3: Linings. The purpose of this WP is to develop a method to early detect problems with linings before the adhesion problems starts. It is important to detect a failure early since this directly affects the function of the lining, i.e. lose its corrosion protection. The water tightness of a lining is the most important property to delay corrosion of the substrate and thereby adhesion loss. The water tightness of a lining is affected by localized water uptake of the lining which represents failure points in the coating. Therefore the study will focus to evaluate correlation between oxidation of the material, water uptake and corrosion of the substrate with the aim to find an oxidation measurement that can be carried using a small sample instead of destructive tensile tests of the lining. If this is possible checking the status of the lining will be much easier. The materials include both rubber and epoxy-linings and the work will also need to include a literature study to find out what type of the materials that has been used and is in use today. This could be done on material that has been exchanged in the NPPs. Presentation and discussion of the results is included in the work package.

WP4: Barsebäck. Study materials from Barsebäck that have undergone ageing during operation for many years. If possible use existing data for specific component to be able to see how a material has degraded. Investigate if the degradation is on the surface or

in the bulk of the material. Preferable choose the same components as in WP1 to be able to correlate to accelerated trials. Therefore the focus will also be to find components that have been exposed to radiation and high temperature to avoid the effect from storage after close down.

A pre study is needed to identify the polymeric components that can be available to study, the amount of data that can be found for these components and to know if it is possible to gain access to these/possible to take them off site. Likely, the possibility to find information about the components will be limited. Chemical analysis will be used to identify and characterize the materials further. Presentation and discussion of the results is included in the work package.

WP5: Synergy effects between radiation and heat. Study how Oxygen Diffusion Limit (ODL) changes with temperature. Diffusion increases with increased temperature according to the Arrhenius equation but a synergy is seen in degradation when combining heat and radiation. The question is whether this is due to changes in diffusion temperature dependence. Does the damage of the material caused by the radiation allow oxidation in the bulk material below the surface? Surface oxidation of irradiated material has previously been studied by using FTIR spectroscopy. Studies on surface oxidation on black rubber materials are somewhat complicated since the carbon black filler absorbs the IR light completely. It may be possible use analysis methods such as TOF-SIMS to better study the oxidation profile from the surface to the bulk material and find out how deep the radiation penetrate the material. In this work package the cross section of two materials exposed to radiation and heat according to the test program described in WP 1 will be studied by TOF-SIMS.

WP6: Lubricants. The intention for this work package is to provide a screening of compatibility between different materials in contact. This can be based e.g. on the existing lubricants used in the nuclear power plants when installing new components. To do so input is needed from the NPP:s in regards to what lubricants are used for montage of new components. Some Nordic plants do have data of safe combinations; others do not but try to keep the number of lubricants low. A test matrix to cover the need of compatibility data therefore do not need to be too large. A possibility is to use screening of how different materials work together with the suggested rubber components can be carried out through theory (Hansen solubility parameters) and lab tests. Presentation and discussion of the results is included in the work package.

WP7: Training of staff involved in developing technical specification. The intention is to use the experience SP has in setting technical specifications and the knowledge gained during the pilot study as input to technical purchase specifications. This will provide a help when ordering new components containing polymeric materials. This will also give a better ability to set the correct quality for a new material. A work shop and demonstration in SP laboratory is included.

To be able to keep the knowledge gained from the education and help the process in developing technical specification for purchase a upgrade of section 3.1.2 Plastic and rubber in document Tekniska bestämmelser för Mekaniska Anordningar (TBM) is included in WP7. The upgrade will be done during and after the training of the staff to better understand the gaps needed to be filled.

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7 Appendix 1, Reference team

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8 Appendix 2, Interviewees

Power plant	Interviewee	Roll	email
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9 Appendix 3, Interview chart

The colour code provides a sorting for different components and materials.

NPP	Type of component	Function	Component name	System#	Polymer
Ringhals/ OKG	Epoxi-lining tank of carbon steel, emergency cooling systems	Corrosion, erosion protection	Sigmaguard CSF650		Epoxi
Ringhals	Rubber-lining tube, tank	Corrosion, erosion protection		715 (713)	Rubber
Ringhals	Valve, seal	Basin door for fuel loading	Orbinox, Uddeholm		EPDM
Ringhals	O-rings for charge pump	pumping reactor water	many	30/40 334	EPDM
Ringhals	O-ring for residual heat removal pump	Removes heat water during start and stop and LoCA	136 P	30 321	EPDM
Ringhals	Membrane valve	flow control	Grinell		Reinforced EPDM
Ringhals	Teflon seal, plug	Sealing drag valve	FCV 478, 316/GM/	30/40 415	PTFE
TVO	O-ring for valves			3-54	EPDM (sulphur vulc)
OKG	Membrane valve		many		EPDM/bunarubber/ Chloropren
OKG	O-ring for valves		many		EPDM/Viton/Silikonummi
OKG	Rubber lining				
Forsmark	Sealing EPDM, O-ring	Scram valve, keep tightness		3-54	EPDM
Forsmark	Sealing EPDM	Drivdon, keep tightness		2-21	EPDM
Forsmark	Valve vapour tube	Will shut down when closing containment		30-3-11	Nitrilrubber
Forsmark	Lining tank	Corrosion protection		733	Natural rubber (most likely)
Forsmark	Membrane in cooling pump	Tightness			EPDM
Loviisa	Lining epoxi	corrosion protection in seawater pipes			Epoxi
Loviisa	Rubber lining	corrosion protection in seawater pipes			
Loviisa	O-rings for primary pumps	pumping reactor water			EPDM
Loviisa	Man door seals	sealing			Silicon rubber

NPP	Type of component	Miljö							
		Temp °C (min/max)	Moisture	Radiation	Relaxation	Oxygen	Pressure	Grease	Loss of Coolant Accident
Ringhals/ OKG	Epoxi-lining tank of carbon steel, emergency cooling systems	5/50	Water	No	No	Sometimes	No		No
Ringhals	Rubber-lining tube, tank		Sea water	No	No	Yes	No		No
Ringhals	Valve, seal	30/50	Deionised water	Yes, high during 40 h	No	Yes	No		No
Ringhals	O-rings for charge pump	40/70	Reactor water	Not normally	No	No	Max 174 bar	Yes	Yes
Ringhals	O-ring for residual heat removal pump	(40?)70/174	Reactor water	Possible	Yes	Yes	Yes, max 465 PSIG		Yes
Ringhals	Membrane valve	40/70	Water	Yes	Yes	Sometimes	Sometimes		No
Ringhals	Teflon seal, plug	50/224	Feed water (deionized)	No	No	No?	62-95 bar		No
TVO	O-ring for valves	-/100		Yes					
OKG	Membrane valve	Operating temp (45 C)	Yes	Yes (75-80% located outside containment), membranes located in purification system, can be exposed to radiation outside containments as well.	Yes, vibration				
OKG	O-ring for valves	Operating temp (45 C)	Yes	Yes (75-80% located outside of containment)	Yes, Vibration				
OKG	Rubber lining								
Forsmark	Sealing EPDM, o- ring	Not high	No		Vibration	Nitrogen			No
Forsmark	Sealing EPDM	50-100, to 140		Low radiation (gamm)	Vibration				No
Forsmark	Valve vapour tube		No	Radiation	Vibration	Air, dry			Yes
Forsmark	Lining tank	~20	Yes, deionized water	No	No	No			No
Forsmark	Membrane in cooling pump	For high temp		No					
Loviisa	Lining epoxi	4/25	Sea water	No	No	Yes	Yes	No	No
Loviisa	Rubber lining								
Loviisa	O-rings for primary pumps		Reactor water	Yes	Yes			No	Yes
Loviisa	Man door seals		Water	Yes	No	Yes	Yes	No	Yes

NPP	Type of component	Material data	Quality control	Method	Replacing procedure	Comments
Ringhals/ OKG	Epoxi-lining tank of carbon steel, emergency cooling systems	Yes	Not installed	Visual	-	Tested at SP. OKG has this. Worry in regards to hardness life time and leaching.
Ringhals	Rubber-lining tube, tank	Unknown	Yes	Visual, hardness	Changed when rubber is loose	Water contains hypochlorite (locally and consumed quickly), have issues with barnacle.
Ringhals	Valve, seal	Unknown	No	-	-	Used only for loading fuel into reactor containment.
Ringhals	O-rings for charge pump	peroxide vulc., hardness 70-80	No	-	Specified number of years	Continuous low flow, higher flow and radiation at LOCA. Normal compression of o-rings are 20%.
Ringhals	O-ring for residual heat removal pump	relatively unknown	At installation	Visually	8 years	Normally turned off, active cooling to 70 °C, compression set is critical
Ringhals	Membrane valve	maybe			Leakage	Compression set problems, compressed 20-30%, in many different systems.
Ringhals	Teflon seal, plug	maybe	-	-		Teflon coated steel spring
TVO	O-ring for valves		Dimension+visual+specification	Measuring manually, visual.		
OKG	Membrane valve	Origin manufacture may sometimes not exist anymore. Batch# provided at delivery incl hardness and type of rubber.	At suspicion of lack of quality products has been sent to a lab for testing.	Visual	Replacement is based on experience (every 6th year), components is compared to similar environments and material.	Great difference in the quality between different EPDM materials.
OKG	O-ring for valves		At suspicion of lack of quality products has been sent to a lab for testing.	Visual	Replacement program (for instance rubber tube) exist for some components but often replacement is based on experience.	
OKG	Rubber lining				Change program exists	
Forsmark	Sealing EPDM, o-ring				Every 4th year	
Forsmark	Sealing EPDM	Peroxid vulc			Service, replace /4 year	
Forsmark	Valve vapour tube		Quality 70 shore A		Replace / 2 year	Used at reactor start
Forsmark	Lining tank				Control every 7th year.	Amount of sulphur in tank is increasing with time. Leaching? Water is used in a accident scenario.
Forsmark	Membrane in cooling pump					Pumps with to high power that has been reduced. This leads to a higher working temperature then estimated for the seals in the pump.
Loviisa	Lining epoxi		Yes	Visual		Replaced with hard rubber
Loviisa	Rubber lining					
Loviisa	O-rings for primary pumps					
Loviisa	Man door seals					More information from Loviisa

ACCEPTANCE CRITERIA FOR POLYMERS IN NUCLEAR

This report contains a feasibility study for acceptance criteria for different polymeric materials in system components in nuclear power plants. The report proposes different methods for evaluating acceptance criteria and identifies where there are gaps in knowledge. Different work packages are proposed to set acceptance criteria for the polymeric materials in the system components identified during interviews with the Nordic nuclear power plants.

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