SERVICE LIFE EVALUATION OF NON-METALLIC CONTAINMENT SEALS

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ABSTRACT

A Service Life Evaluation Program (SLEP) was successfully completed for expansion joint seals used in the pressure relief duct of Pickering NGS, an eight unit CANDU station. These seals are part of the containment boundary, are difficult to replace and then only during station outages which are scheduled at ten year intervals. The SLEP, consisting of an accelerated aging program and a qualification test, was undertaken to determine the service life of the seals such that they would be capable of remaining functional following a combination of service life, a design basis accident and a subsequent seismic event.

The Arrhenius model of aging was used for the accelerated aging program. Samples of seal material were subjected to oven aging at five temperatures, 150, 160, 170, 180 and 190°C. Tensile properties and hardness were measured at various aging times and Arrhenius plots constructed. Based on changes in elongation, activation energies of 1.2 eV and 1.1 eV were calculated for the reinforcing fabric and the silicone cover rubber, respectively. Hardness measurements were also taken but, as expected, no precise quantitative aging relationship could be determined from material hardness.

For the qualification test, a representative length of seal was installed and field-spliced in a test frame built to simulate the installed configuration of the seal. It then underwent accelerated aging equivalent to the service life, followed by LOCA irradiation, exposure to LOCA conditions of humidity, temperature and pressure and a Design Basis seismic event. Finally, a pressure test to approximately five times design pressure was successfully performed to demonstrate the remaining margin of safety. Periodic air leakage tests indicated no deterioration in sealing performance and no physical deterioration was apparent.

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1.0 Introduction

It has long been recognized that non-metallic materials deteriorate when exposed to heat, radiation and other stresses. Such materials are of special concern when they are located in the containment boundary of nuclear generating stations. There are a number of non-metallic containment boundary components in the multi-unit nuclear stations at Ontario Hydro. These include airlock seals, pressure relief valve diaphragms, vacuum building roof seals, reactivity mechanisms deck seals and the joint seals in the pressure relief duct at Pickering NGS which are the topic of this report.

Pickering NGS is an eight unit CANDU nuclear generating station, with a negative pressure containment system having a 41.4 kPa(g) design pressure. The pressure relief duct (PRD) is a concrete tube which connects all eight reactors to the vacuum building via pressure relief panels and pressure relief valves. During a loss of coolant accident (LOCA) the PRD conveys steam and fission products to the vacuum building from the accident unit. The PRD is over 600 m long, mounted on piers approximately 20 m high and contains twenty-three expansion/contraction joints which are spanned by fabric-reinforced rubber seals. During a Pickering station outage in 1990, the existing PRD seals were replaced with polyester-reinforced silicone rubber seals.

Since the joint seals are part of a multi-unit containment boundary, they are difficult to replace and then only during station outages which are scheduled at ten year intervals. A service life evaluation program (SLEP) was undertaken to demonstrate the capability of the PRD seals to remain functional following a combination of normal service (preferably for the remainder of station life), a design basis accident and a subsequent seismic event.

2.0 Test Program for PRD Seals

The test program described here was initiated in 1989 and was planned so as to preclude the need for further testing when the formal qualification assessment is performed.

The test sequence was chosen to bound all normal service and design basis events in a single test, thus reducing cost. The service life included a 47.6kPa(g) commissioning test and forty years operation interspersed with pressure tests to 41.4 kPa(g) and scheduled at ten year intervals. The accident sequence included a bounding 'LOCA' which enveloped both main steam line breaks and a Loss of Coolant Accident coincident with Loss of Emergency Coolant Injection (LOCA/LOECI). Both a Site Design seismic Event (SDE) following a LOCA and an isolated but more severe Design Basis seismic Event (DBE) are also credible events. Therefore, the enveloping accident sequence included the more intense DBE following the bounding 'LOCA'. Finally the PRD seal and frame were pressurized for a 'burst' test to determine the remaining margin of safety. Throughout the entire sequence of normal service and post-accident service, the seal was left undisturbed in its mounting frame.

In order to simulate the normal service aging, accelerated aging was employed using the widely utilized Arrhenius model[2]. Since no relevant aging data existed for the materials used in the joint seal, an aging program was conducted to determine appropriate values for aging temperature and duration via calculation of the Arrhenius activation energy. The importance of the joint seals and the use of similar materials in other seals (vacuum building roof seals) made the considerable effort and cost acceptable.

All testing was performed at Siemens laboratories in Erlangen and Karlstein, Germany and using instrumentation traceable to German national standards.

2.1 Thermal Aging program

The Arrhenius model relates natural aging effects with accelerated thermal aging via an activation energy which is often specific to a particular physical or chemical property (eg tensile strength) of the material being tested. The joint seals consist of a fabric (polyethylene terephthalate filament) to provide tensile strength and a rubber cover (vinyl methyl silicone elastomer) to provide sealing and transmit the clamping and tensile load to the fabric. Therefore, the properties of interest were tensile strength of the polyester fabric, sealing backforce of the silicone elastomer and hardness (to monitor aging *in situ*). To ensure unusual interactions between the rubber and fabric were not overlooked, all samples were made from the composite material.

Ultimate tensile strength and elongation at break were determined for both the composite seal (ie. limited by the polyester fabric) and the cover rubber using 'dog bone' samples and the ASTM D378 test method (see Figure 1). Following rupture of the fabric, the load was dropped to 100 Newtons, the equipment range recalibrated and the test continued until failure of the silicone cover rubber. Samples in which the rubber was damaged during fabric rupture were not tested further. The tab portions of the 'dog bone' samples were used for hardness tests (ASTM D2240), prior to tensile testing.

The load/deflection characteristics of the seal were analyzed using ASTM D575 Part A. The compression shape factor of the seal was taken into account by using samples which duplicated the 15 cm span between clamping bolts in the Pickering NGS seal installation.

The thermal aging program was performed using circulating-air ovens with temperature controllers. Ideally, accelerated aging temperatures should be as close as possible to the service temperature. However, accelerated aging duration must be reasonable (typically several hundred hours) for practical and economic reasons. Based on unpublished data from testing of similar compounds (data supplied by elastomer and filament manufacturers), a trial aging run was performed at 160°C. Based on these results, five aging temperatures, 150, 160, 170, 180 and 190°C, were selected to produce measurable degradation of the silicone rubber within a reasonable time period and without high temperature decomposition of the polyester. Since aging duration at 150°C exceeded one year, temperatures below 150°C were rejected

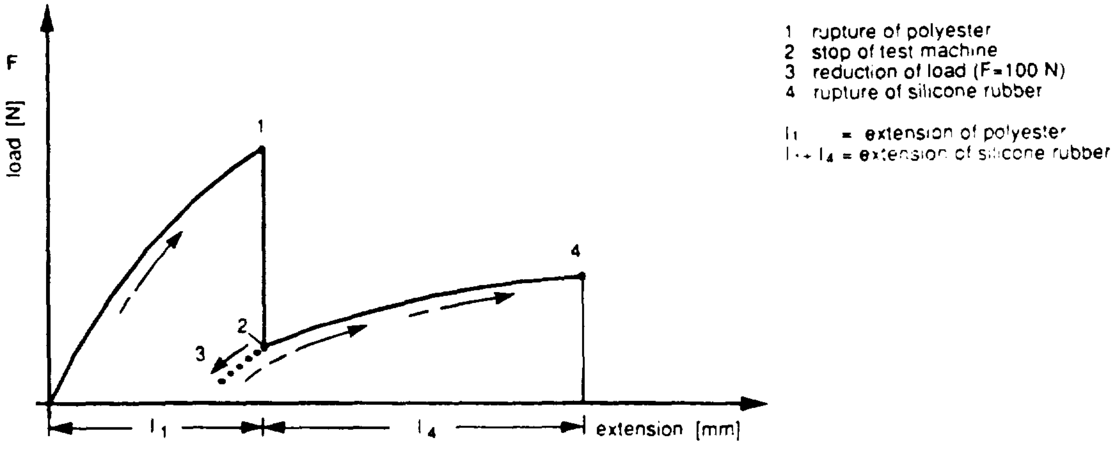


Figure 1: Load-extension curve for tensile test of composite seal.

because of excessive aging periods. Typically, five aging intervals were used at each temperature with three specimens per sample point for a total of over seventy-five samples.

2.11 Aging and Tensile Properties

Samples were removed from the ovens at specified intervals and tensile tests performed, breaking first the polyester fabric, and then the silicone elastomer. Typical results for elongation at break (UE) are provided as relative elongation for silicone-covered polyester (polyester fabric) and silicone rubber in Figures 2 and 3, respectively. For the polyester fabric, both ultimate tensile strength (TS) and elongation at break (UE) were reduced as aging time increased. Thermal aging at higher temperatures (180°C, 190°C) led to immediate and quick reductions in tensile strength followed by a period of slow reduction. At the lower temperatures (150°C), the commencement of deterioration in TS was significantly delayed (eg. 5% reduction after 2500 hours) before decreasing and over 9000 hours were necessary to achieve a 70% reduction in tensile strength. In contrast, the relative elongation results showed a more sustained reduction as time increased and did so over all temperature ranges.

For the silicone rubber, reductions in relative elongation were more evident than reductions in tensile strength over the same time period. The elongation results showed trends similar to those for polyester (seal as a whole) although the measurements for silicone rubber were more variable than for the polyester since the failure of the fabric will cause some damage to the silicone rubber surface and thus influence the tensile properties of the rubber.

Overall, UE exhibited a greater sensitivity to thermal aging than did tensile strength. Elongation is widely utilized as the most sensitive indicator of aging and one that exhibits a steady decline with progressive aging[2]. Data from Barbarin[3] indicates that the sensitivity of elongation to aging exceeds that of tensile strength and is comparable to that of compression set for several high-performance elastomers including silicone rubbers. Therefore, among the parameters investigated here, elongation at break appears to be the most useful parameter for constructing Arrhenius plots representative of the aging characteristics of the seal material.

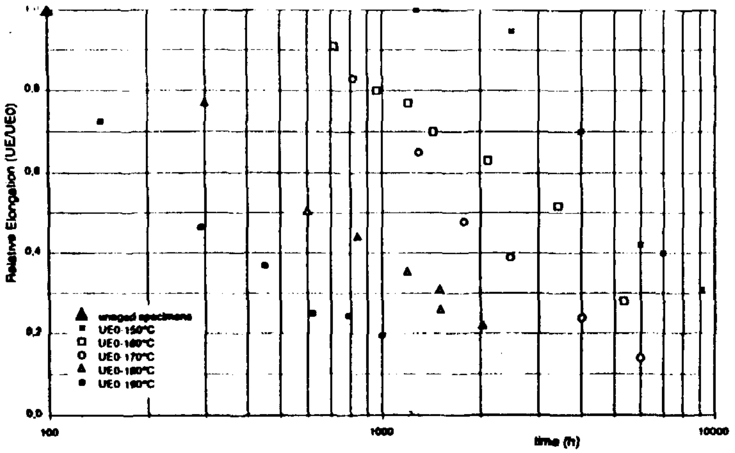
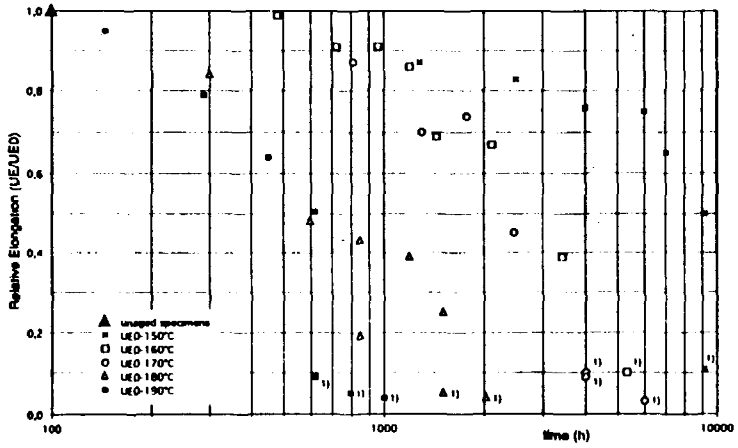


Figure 2: Thermal aging data - relative elongation (UE/UE$_0$) for silicone-covered polyester



1) rupture of polyester and silicone simultanously

Figure 3: Thermal aging data - relative elongation (UE/UE$_0$) for silicone rubber

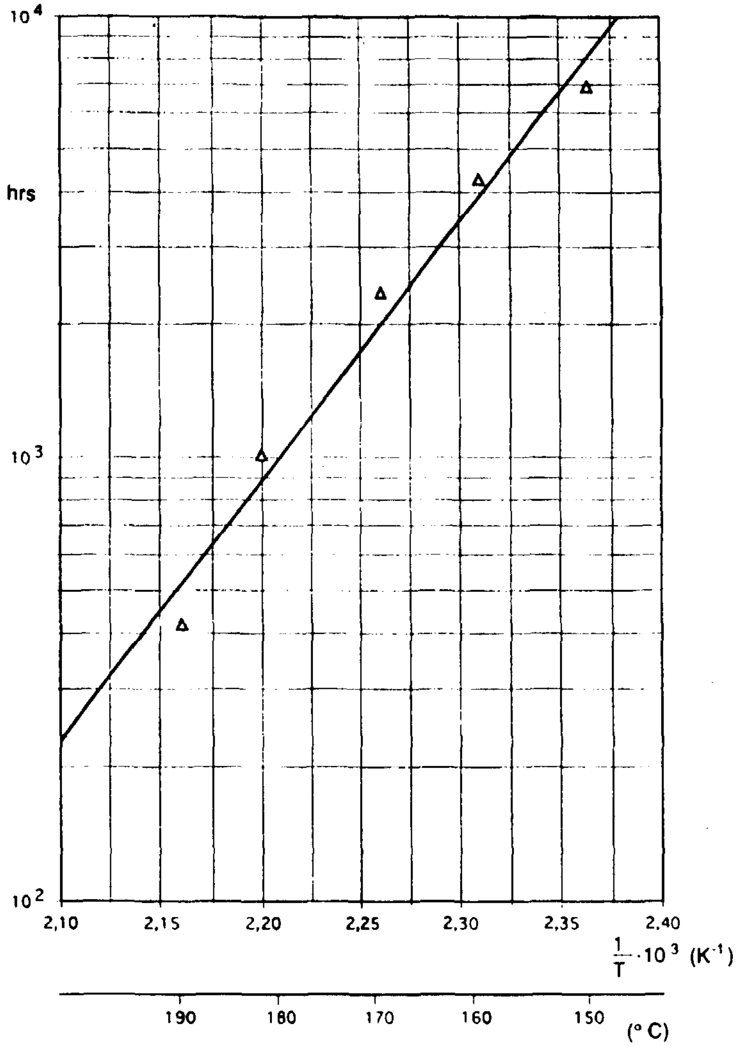


Figure 4:

Arrhenius - Plot for Polyester (UE/UE0 = 0,4)

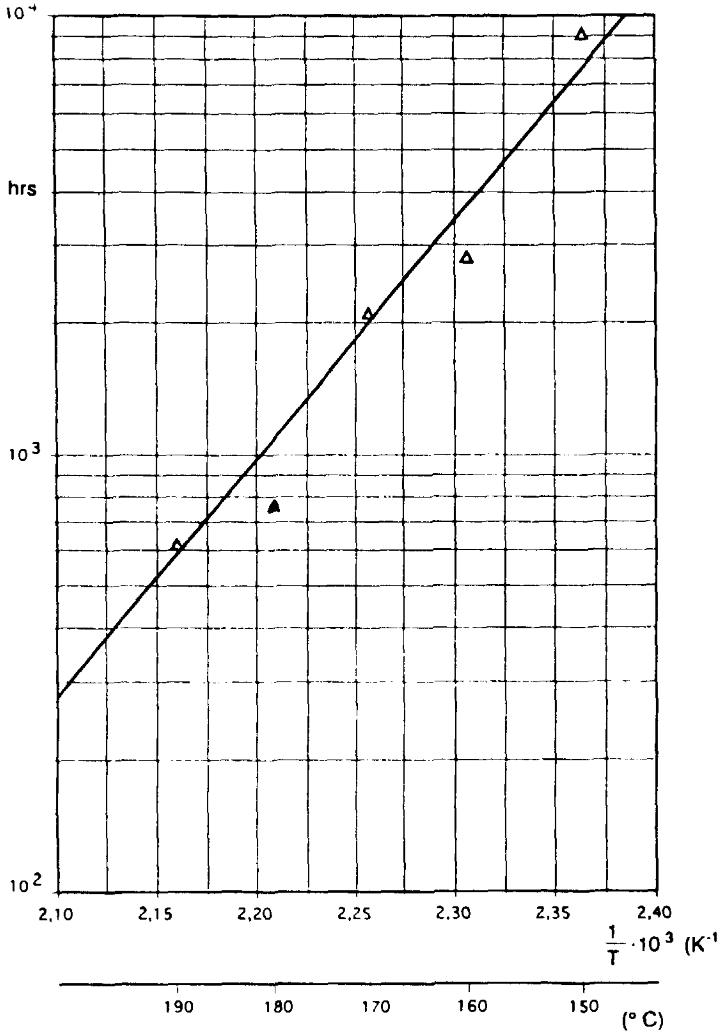


Figure 5:

Arrhenius - Plot for Silicone Rubber (UE/UE0 = 0,5)

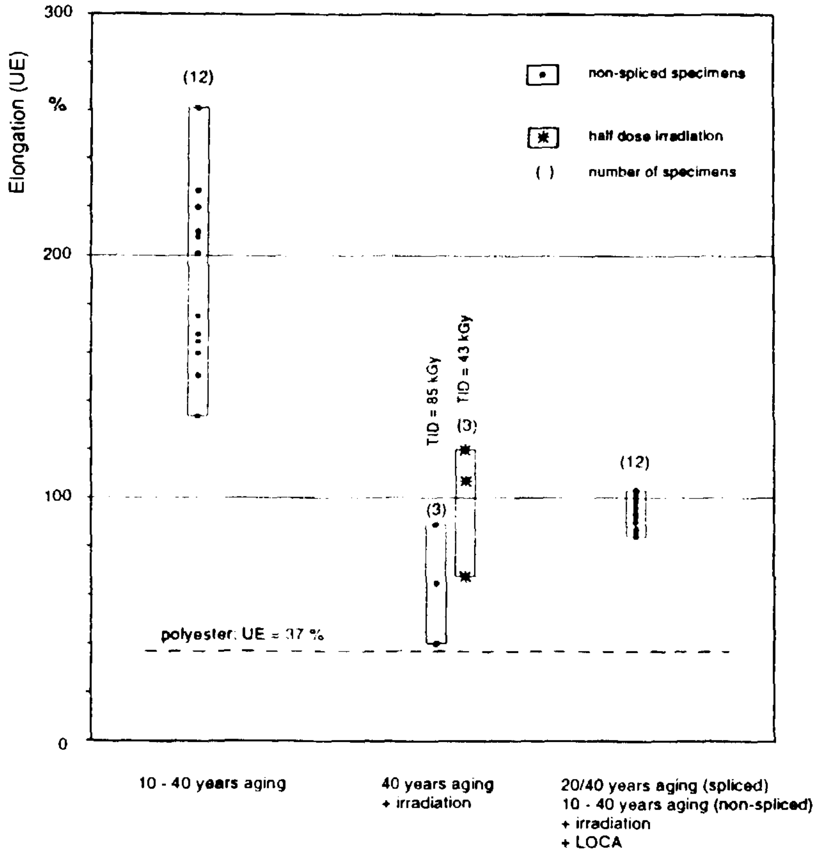


Figure 6: Effect of irradiation and thermal aging on ultimate elongation of silicone rubber.

From Figures 2 and 3, the time for a specified level of degradation was determined, Arrhenius plots were constructed from time/temperature data and an activation energy calculated from Equation 1, below.

$$\ln\left(\frac{t_1}{t_2}\right) = -\frac{E_a}{K_b} \times \left(\frac{1}{T_1} - \frac{1}{T_2}\right)$$

Equation 1

t_1, t_2 ..... *times for service life and equivalent life at elevated temperature*
E_a ..... *Arrhenius activation energy in eV*
K_b . .... *Boltzman constant = 0.8617 E-4 K/eV*
T_1, T_2 ... *Temperature for service life and for accelerated aging life*

The level of degradation can be a specific failure criteria (eg 45%). In this case, engineering judgement indicated that there was ample margin in tensile properties and the degradation levels were selected to ensure that they represented as much data as possible and, given some variability in slope, that they provided the most conservative activation energy. For the polyester, this was 40% degradation, and for the silicone rubber, 50% was selected due to limited data below this point. Levels of 50% have been used elsewhere[4,5]. From the Arrhenius plots (see Figures 4 and 5), activation energies of 1.17 eV and 1.07 eV were calculated for the polyester fabric and silicone rubber, respectively.

2.12 Aging of Compressive Properties

Aging studies of compression properties often use compression set as the critical property.[3] However, this approach does not provide directly a sealing backforce measurement. Here, the aging of compressive properties was attempted by examining load/deflection curves (ASTM D575-Part A) as a function of aging time. Samples were prepared to resemble a 15 cm section of the bearing clamp, used at Pickering NGS, in order to account for the compression shape factor. Preliminary aging trials were conducted at 110°C and 160°C for periods varying between 50 and 700 hours.

The results showed very little variation in the load/deflection curve for the seal and attempts to determine absolute sealing backforce were not encouraging. For the relatively short aging times available for the compression samples, it was difficult to separate the effects of relaxation and actual aging-induced changes in load/deflection properties. No further compression testing was attempted.

2.12 Aging and Hardness

Hardness has been the traditional non-destructive method for determining rubber aging *in-situ*. However, as found in some other studies[4,7], the results shown in Figure 7 show no precise relationship between aging time and hardness, although there is clearly a trend towards increasing hardness with increased aging time. Because of the lack of a quantitative

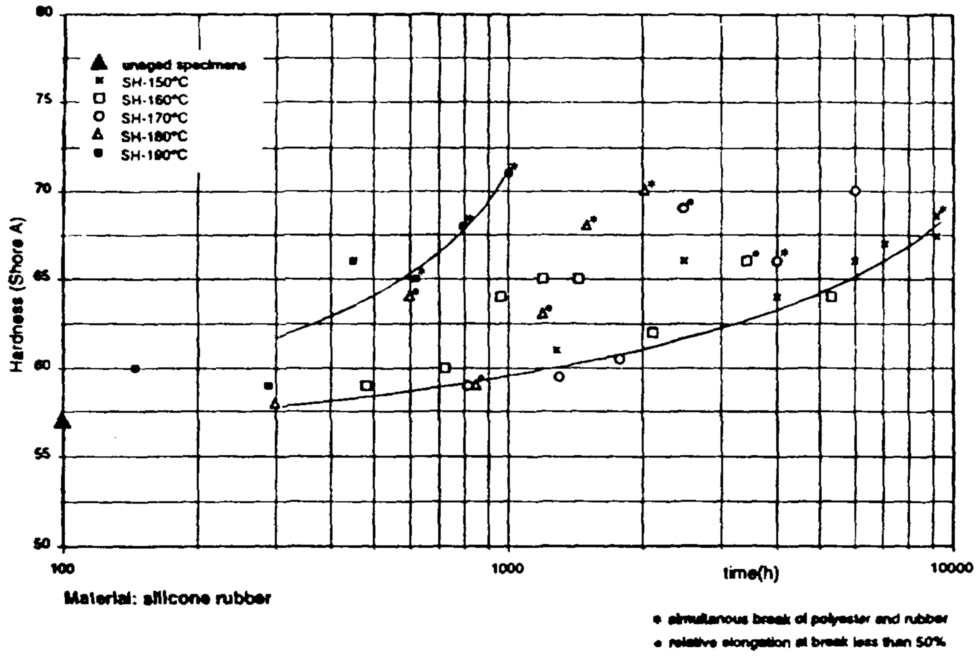


Figure 7: Hardness of thermally-aged specimens

relationship, and in order to confirm the accelerated aging results and monitor the installed seals, it will be necessary to rely on destructive testing of sample coupons of seal material which were mounted beside each PRD joint during seal installation.

2.13 Summary of Aging Program

In Table 1, the activation energies calculated here are compared with typical activation energies for another silicone rubber (1.5 eV)[8], ethylene propylene rubber (1.05 - 1.28 eV) which is often considered to be similar to silicone in aging resistance, and neoprene (0.87 eV), an older and less aging resistant elastomer[9] used in some of the original seals. The Ea of 1.07 eV for silicone rubber was selected for further aging of the composite seal in order to be conservative. It is the lowest for the composite seal and would provide an approximate factor-of-two safety margin against catastrophic failure of the polyester fabric (seal rupture). Lack of an activation energy for the loss of sealing backforce was not considered critical. The majority of the clamping load (1.9 MPa) is necessary to provide deflection which can compensate for unevenness in the clamping bars. The clamping load requirements to retain containment pressure (41.4 kPa(g)) and prevent pull-out of the seal (69 kPa(g)) are considerably less. Relaxation of the seal will compensate for much of the clamp unevenness and reduce the load requirement even as the sealing backforce is reduced due to compression set. Furthermore, leakage due to aging of the silicone rubber, if aging of the compressive property were significantly different from that for elongation, can be detected via routine leakage testing.

Material/Property	Activation Energy (eV)
Polyester fabric elongation (this study)	1.17
Silicone rubber elongation(this study)	1.07
EPR elongation[9]	1.05-1.28
Neoprene rubber[9]	0.87
Silicone rubber range;average [11]	0.86-1.97; 1.22

Table 1: Summary of Activation Energies

2.2 Phase II Testing - Radiation Effect on Coupons

A second phase of testing was performed to quantify the effect of the accident radiation on the seal material and to ensure that sequential application of accelerated aging and the accident dose (85 kGy) would not cause failure in the qualification test. No significant changes in tensile properties were observed for the composite seal (ie. the polyester fabric) after irradiation or accelerated aging. However, there was a significant reduction in elongation at break for silicone rubber (see Figure 6).

Virgin silicone material exhibited an ultimate elongation (UE) of 210%. Elongation at break for samples exposed to 196 hours of thermal aging (100°C) plus either 43 kGy or 85 kGy retained an UE of 95% and 61%, respectively. The additional reduction in UE at 85 kGy is consistent with reports of threshold radiation damage for silicone elastomers[10] occurring at 10-50 kGy total integrated dose. However, none of the reductions in elongation at break is even close to the limiting ultimate elongation of the polyester fabric (37%) and therefore, no compromise of the seal properties (eg flexibility) is expected.

2.3 Qualification test

2.31 Test Sequence and Service Life Aging

The qualification test was composed of a simulated service life followed by a conservatively modelled design basis accident/seismic event sequence (see Figure 8). The post accident mission time of the seal is six months. However, the pressure differential (and stress on the seal) drops to essentially zero after 48 hours.

The seal was installed in an octagonal frame with full-scale dimensions except the length of the straight spans between corners, which were much reduced to accomodate the test vessel.

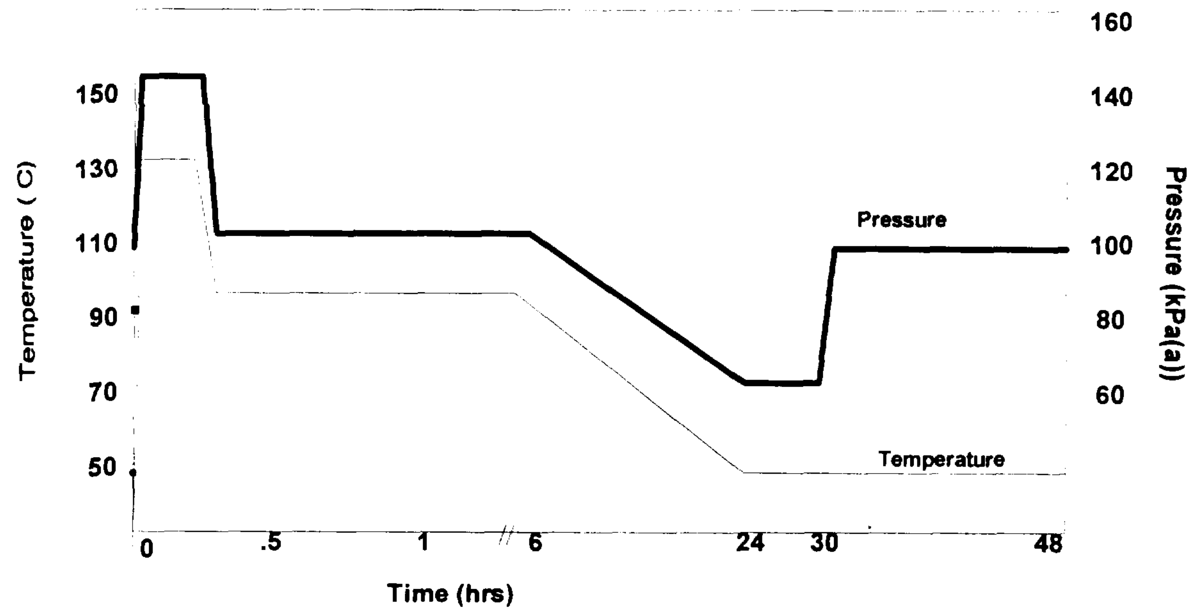


Figure 8: Post accident temperature and pressure profile.

The corners are under the greatest stress during overpressure and are therefore reproduced in full scale (see Appendix A). The frame was built to the original construction standards and the seal was installed and field-spliced according to normal installation procedures with clamping bolt torques of 67 N.m. The seal was left undisturbed for the entire qualification test sequence.

The test frame containing the seal was then mounted in the LOCA test vessel in the Karlstein laboratory (see Appendix B). For the aging portion of the program, a cover was mounted outside the seal to ensure homogenous temperatures both inside and outside the seal. Between phases of the test sequence, leakage tests were performed at design pressure (41.4 kPa(g)) and torque measurements were taken on sets of ten bolts, selected at random.

The service life was simulated by a commissioning pressure test to 47.6 kPa(g) followed by accelerated aging equivalent to forty years (design life of the station) divided into four 'ten-year' intervals equivalent to the periods between test outages. To simulate each 'ten-year' interval at a conservative PRD temperature of 25°C, accelerated aging was performed for 100 hours at a temperature of 90°C. This gives a factor of three margin when combined with the use of an activation energy of 1.07 eV instead of 1.17 eV. Each of these 'ten-year' equivalent periods was followed by four pressurizing cycles plus a pressure test to determine seal leakage at the design pressure of 41.4 kPa(g). The additional pressure cycles allow for variations in test frequency during the balance of the service life of the station. The PRD containing the joint seals is normally isolated from the operating reactors and the cumulative radiation dose is negligible. Homogeneous temperatures were ensured by electrical heating elements in the wall of the vessel and circulating externally heated air via fan.

Following the service life aging, no significant leakage could be detected and bolt torques had reduced from the inital 67 Nm to 20 Nm due to relaxation of the seal.

Ideally, the bounding 'LOCA' should consist of a simultaneous application of radiation, steam and high pressure. For practicality, the seal frame was irradiated first to the full accident dose and then exposed to accident temperature, steam and pressure in the LOCA test vessel. This is consistent with findings elsewhere which conclude that irradiation prior to thermal stress (as part of accident or independently) is the most conservative sequence[1]. Furthermore, the irradiation was conducted at accident temperature to include potential synergistic effects.

2.32 Irradiation

The appropriate gamma dose, in the bounding 'LOCA' of the accident sequence, was determined to be 40 kGy. The initial estimate of the beta dose was approximately 350 kGy, potentially damaging for silicone elastomers. However, only 35 kGy of this is sufficiently energetic to penetrate to the reinforcing fabric. One side of the silicone will sustain damage from substantial amounts of less energetic beta. However, the two sides of rubber are not connected and cracks or damage to one side will not propagate past the fabric to the shielded side of the seal. Therefore, the fabric and one layer of rubber will be exposed to, at most, 35 kGy of penetrating beta radiation. The gamma plus penetrating beta plus a ten percent margin led to a total dose of approximately 83 kGy.

The seal and frame were removed from the LOCA test vessel, transported to the irradiation laboratory and mounted in a room-sized irradiation chamber with Co-60 sources delivered to the center of the test frame via guidepipe (see Appendix D). The test frame was mounted inside a sheet-metal inner chamber which was continuously supplied with heated air delivered to both sides of the seal to maintain the test temperature of 100°C. During irradiation, temperatures were measured via thermocouples and resistance temperature detectors (RTD's). All parameters were continuously recorded on chart recorders for audit purposes.

Dose rates were kept as close as possible to expected doses and are much lower than typical rates in other studies[1]. Actual dose rates varied from 280 to 350 Gy/hour. Typical accident dose rates are estimated to be approximately 1000 Gy/h during the first day to an average of 40 Gy/h over the next 30 days. The use of an extended heating period (309 hours versus 48 hours specified in the test profile), employed to accomodate the low dose rate (300 Gy/hour), resulted in additional conservatism in the test.

2.33 LOCA test

The seal and frame were remounted in the LOCA test vessel. The test was started at 35°C and superheated steam was injected into the vessel up to an overpressure of 0.49 bar.

Temperature rise from saturation to superheated at 135°C was slow due to the thermal inertia of the test specimen and heat transfer to the test vessel walls. Thus the overpressure phase lasted 30 minutes (twice specification) and the superheated interval was 15 minutes as specified. The seal was sprayed with soap solution during the overpressure period in order to identify leaks. Small amounts of leakage were measured during the underpressure but no leaks could be found in the seal or frame via soap spray or inspection. It was periodically necessary to activate a vacuum pump to maintain the underpressure transient. After 30 hours, the pressure was increased to reach atmospheric pressure at 32 hours and the test was continued up to 48 hours at 50°C. The seal was leaktight at the end of the LOCA test and torque measurements once again indicated a range of 20 - 25 Nm.

2.33 Seismic Testing

For Pickering NGS, a LOCA and Site Design (Seismic) Event is design basis. However. to envelope the case of a more serious Design Basis (Seismic) Event, the latter was used here. A seismic event will result in movement between adjacent sections of the pressure relief duct. The magnitude and frequency of movement to be experienced by the joint seal, which spans the gap between adjacent sections, are listed in Table 2, above. Testing was performed by mounting both the seal and its frame on a large servohydraulic shaking table (SH9) located in the Siemens laboratory in Erlangen (see Appendix D). Prior to beginning the test, a torque measurement of 40 Nm for several bolts indicated some corrosion of the test frame and hardware.

The seal specimen survived the seismic test with no visible indications of physical damage or displacement. After remounting of the seal and frame in the LOCA test vessel, a leakage test was performed at the design pressure of 41.4 kpa(g). A few small bubbles were detected in some corner sections but the leakage was insignificant (less than 0.5 kPa in 20 minutes).

test section excitation	seismic testing	
type of excitation	fixed sine	
direction of excitation	X	Z
frequency	5 Hz	8 Hz
excitation level	± 5 mm	± 9 mm
duration	≥ 20 sec	

Table 2: Seismic testing parameters[11]

2.34 'Burst' Test

Finally, the joint seal was exposed to a high pressure 'burst' test to establish, for the end-of-life condition, the remaining margin of safety relative to the design pressure of 41.4 kPa(g). Test vessel pressure was increased in increments of 25 kPa until a final pressure of 200 kPa(g) was reached (limited by constraints of test rig and safety considerations) and held for 50 minutes. The seal was observed to stretch to accomodate an increase in diameter of up to 16 mm along the midline. This expansion translates into approximately 14% elongation of the fabric in the seal. The full 200 kPa(g) is approximately 19% elongation, significantly below the 37% elongation capability of the seal material.

No significant leakage could be detected during the pressurization test. The seal and test frame were visually inspected following completion of testing. There was no indication of slipout of the seal from the clamping arrangement. The colour and surface condition of the silicone rubber were unchanged. This test confirms that the margin of safety for tensile strength of the seal is approximately a factor of five without taking credit for the full elongation capability of the seal. This confirms the engineering judgement in Section 2.11 that there was ample margin to avoid using a specific failure criteria. The availability of further elongation capability indicates further significant margin. Further, leakage due to seal degradation or stressing does not appear to be a problem.

3.0 Service Considerations and Summary

Completion of this test program demonstrates the capability of the expansion joint seal to fulfill its pressure-retaining containment function for the balance of plant life (up to 40 years). Following the full test sequence, the pressure retaining capability of the seal is at least five times the required values and leakage was found to be insignificant. Torque measurements indicated an initial drop from 67 Nm to 20 Nm followed by no further changes in torque. This implies relaxation is the only significant contributor to reductions in sealing backforce and that that process is substantially complete shortly after installation.

It has been suggested that attempts to predict natural aging based solely on accelerated aging, with no correlation to natural aged samples, are inappropriate[2]. Therefore, part of the ongoing qualification effort in the joint seal service life evaluation involved placement of sheets of expansion joint seal material beside each joint seal in the PRD. During each 10-year test and maintenance outage, portions of the sheets will be removed to be examined and destructively tested for comparison to the accelerated aging program data.

4.0 Conclusions

1. An experimental program for determining the Arrhenius activation energy of the seal materials was completed. Activation energies of 1.07 eV and 1.17 eV were found for

the ultimate elongation property of silicone rubber and polyester, respectively.

2. A representative model of the PRD joint seal was installed, 'commissioned' and successfully completed tests for: 40 years of service life, scheduled pressure tests, exposure to a bounding 'LOCA' simulation, a Design Basis seismic event for 20 secs. and an overpressure test up to approximately five times design pressure.

   Leakage tests between each phase of the test program indicated no change in the leaktight initial state of the seal.These results demonstrate the integrity and capability of the seal for the balance of plant life. Torque measurements, after an initial drop due to seal relaxation, remained constant for the remainder of the test, indicating no apparent reduction in sealing backforce due to test conditions/ stresses.

3. Irradiation beyond threshold doses during accidents is likely to cause more damage in high performance silicone elastomers than either extended aging or high accident temperatures.

4. Accelerated aging of high-performance elastomers such as silicone will necessarily involve lengthy testing and high test temperatures and may require ongoing testing to ensure the accelerated aging was representative of lower service temperatures. To relate accelerated aging and natural aging and monitor the correlation, coupons were placed in the duct for removal at 10 year intervals.

5. Service Life testing is involved and costly. However, this is acceptable for nuclear safety system components which are largely inaccessible for replacement.

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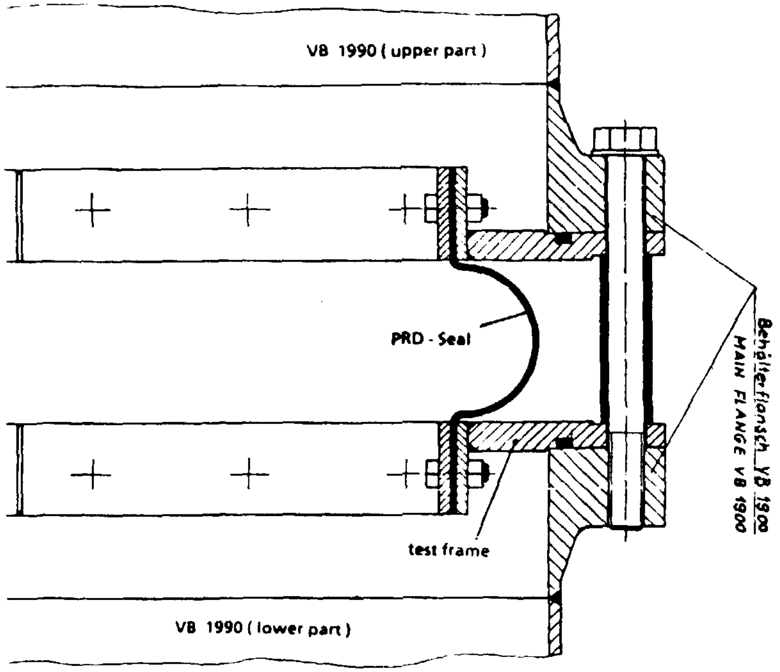
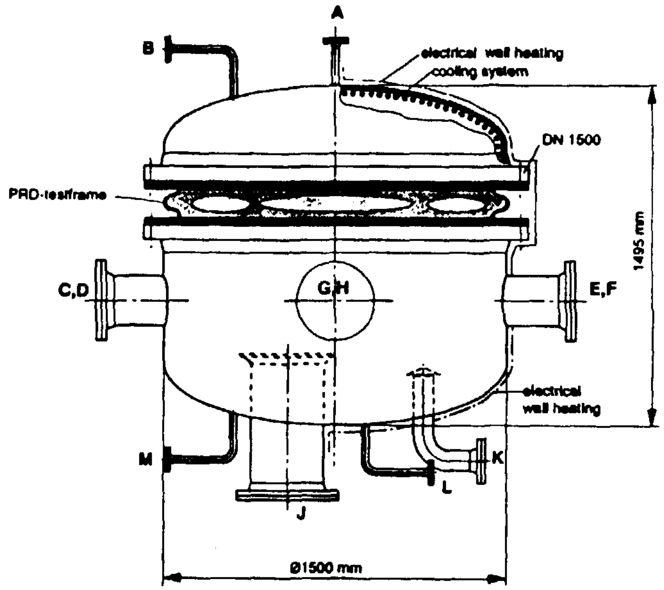


TEST VESSEL VB 1900 WITH PRD-SEAL

Appendix A



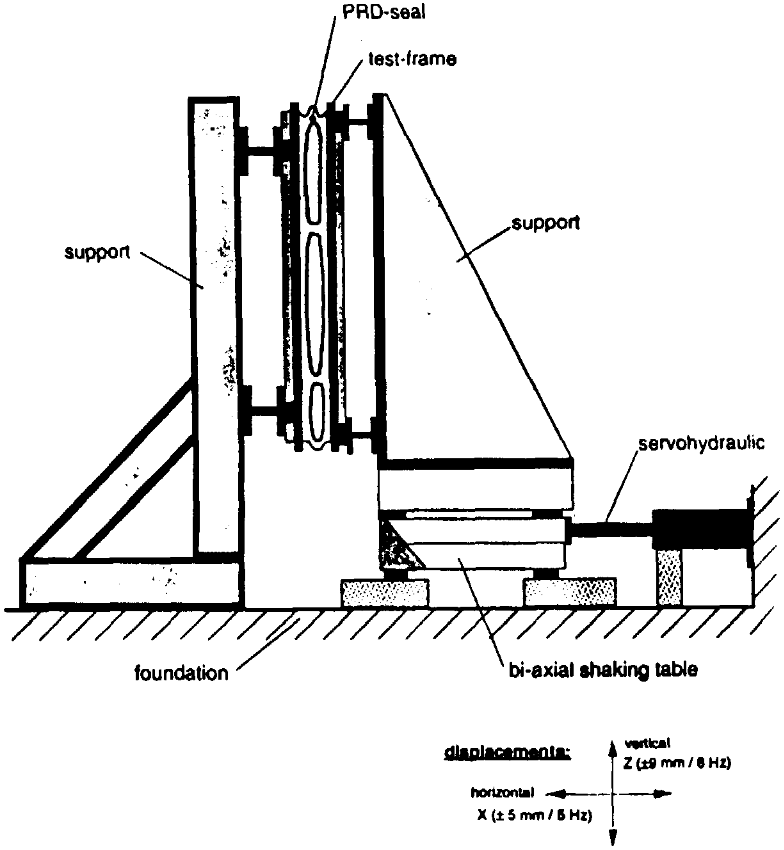
Design Data :

dimensions are millimeters

A,B .... L,M ⟶	nozzles
design temperature	250 °C
design pressure	9 bar
volume (without PRP-testframe)	1900 liters
weight of VB 1900 (without PRP-testframe)	2000 kg
weight of specimen	406 kg

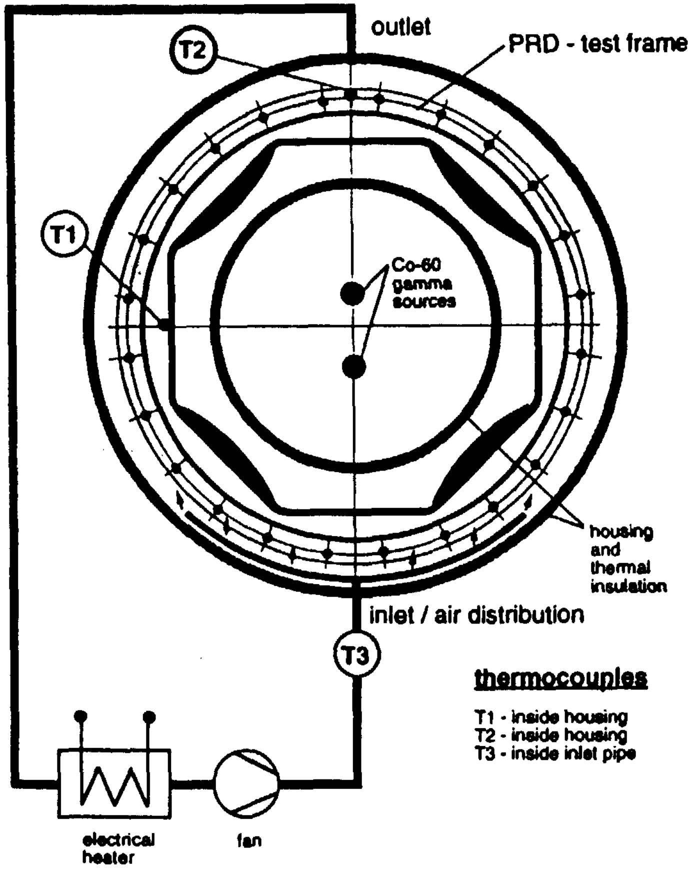
TEST VESSEL VB 1900

Appendix B



Test-Set-Up (Seismic Test)

Appendix C



Irradiation TEST SET-UP

Appendix D