EVALUATION OF THE LIFETIME BEHAVIOUR OF RUBBER SEALS FOR USE IN ALPHABUS THRUSTER VALVES USING ACCELERATED TECHNIQUES

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ABSTRACT

An investigation has been performed to evaluate rubber seal materials and techniques for determining their lifetime behaviour under known loads and temperatures. Initial measurements were performed on silicone samples using dynamic mechanical analysis (DMA) to estimate shift and acceleration factors for subsequent use in a representative test programme to determine accelerated lifetime data. This script presents the principles used for the evaluation, a description of the test apparatus and the presentation of results obtained to date.

1 INTRODUCTION

Seals are commonly used in valves for a variety of space applications including as part of the thruster control system for the Alphabus telecommunications satellite design. Valve seals are frequently produced from a number of different polymeric materials such as polytetrafluoroethylene (PTFE), Viton rubber, silicone rubber, ethylene propylene diene M-class rubber (EPDM) and polyimides. The operational lifetimes and parameters of modern spacecraft mean that these valve systems are expected to withstand longer lifetimes both on ground and in orbit as well as more extreme operating parameters. Today’s requirements are typically 5 year storage plus 15 years flight which could potentially be extended to up to 15 years ground storage and 20 years on orbit. In most cases the valve consists primarily of metallic components and therefore the life-limiting material is usually the polymeric valve seal. The longer durations mean that lifetime issues are not able to be fully covered by the use of heritage data alone which is often limited to the geostationary earth orbital (GEO) conditions and a typical 5 year mission lifetime.

To cover this gap a programme of work has been performed to evaluate ageing effects on silicone seal materials using accelerated empirical techniques. Samples of a silicone seal material supplied by Ampac ISP have been evaluated using dynamic mechanical analysis to determine the response of seals to mechanical stress and elevated service temperatures. This data has then been utilised to develop an empirical prediction for the response of the seal over extended lifetimes using the time temperature superposition (TTS) technique. The TTS analysis derives a number of acceleration factors for a range of test temperatures. This data is then used to perform accelerated tests under representative loading conditions.

The accelerated tests are performed in a bespoke test apparatus which accurately monitors small changes in displacement down to a few microns. The data collected is continuous and is corrected for changes associated with the apparatus to produce a direct measurement of the changes in dimensions of the seal under a given load and temperature environment. Since the data stream is continuous a snapshot can be taken at any given time which can then be used to extrapolate out to the lifetime of the seals under investigation. This prediction can continuously be revised and improved as more data becomes available.

A typical valve will consist of a seat, end stop and a closing spring. To seal the two parts a seal is positioned between the two parts of the valve to ensure a hermetic connection and minimal leakage of propellant. A generic design is shown in Fig. 1 where
it can be seen that the seal would typically be under a predefined spring-load. The continuous application of both temperature and load means that any seal material could be susceptible to creep or deformation over a prolonged period resulting in changes to the functional performance. The temperature ranges considered for the application in question are shown in Table 1[1]. From Table 1 a weighted reference temperature of +80°C was agreed by taking into account factors such as the nominal conditions, the average temperature, margins etc. The material investigated as part of the alphabus programme was a peroxide cured silicone rubber specifically procured for this application by Ampac ISP. Whilst the generic properties of silicones are well known each formulation will have characteristics and behaviour specifically associated with the particular formulation; these can include hardness, modulus, susceptibility to operational environment as well as long term performance such as creep.

<table>
<thead>
<tr>
<th>Table 1: Temperature ranges for valve seal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flight Non-operating</td>
</tr>
<tr>
<td>Flight Operating (with thermal control failure)</td>
</tr>
<tr>
<td>Flight operating (nominal)</td>
</tr>
<tr>
<td>Acceptance</td>
</tr>
<tr>
<td>Qualification</td>
</tr>
</tbody>
</table>

2 PRINCIPLES OF DMA AND TTS

Dynamic mechanical analysis (DMA) is used to measure the mechanical and viscoelastic properties of materials as a function of temperature, time and frequency when they are subjected to a periodic stress. The types of materials that can be analyzed with this technique include thermoplastics, thermosets, composites, elastomers, ceramics and metals. During a DMA test a sinusoidal load is applied and the displacement response of the material is accurately measured (see Fig. 2). The phase difference ($\delta$) between the applied stress and the materials response corresponds to the ratio of the storage ($M'$) and loss modulus ($M''$). The moduli are calculated from the measured stiffness according to the following equations:

$$\left| M' \right| = Sg = \frac{F_0}{L_0} \Rightarrow S = \frac{F_0}{L_0}$$

where $g$ is the geometry factor calculated from the sample dimensions and $S$ is the stiffness of the sample. The storage and loss modulus can then be derived from the following equations:

$$M' = \left| M' \right| \cos \delta \quad M'' = \left| M' \right| \sin \delta \quad \tan \delta = \frac{M''}{M'}$$

Fig. 2. How DMA works

Once a sinusoidal load is applied and the response monitored then the relative values of $\tan \delta$, $M'$, $M''$ can be determined. The values of these parameters then become dependent on the state of the material at a given temperature. For instance the ability of the material to flow will mean a higher level of loss.
modulus - i.e. more viscoelastic character. This value in turn will be governed by the state of the molecular interactions of the material. Typical changes which can be observed by DMA are shown in Fig. 3. From Fig. 3 it can be seen that a frequency abscissa scale is utilised indicating that the processes described and indeed all molecular behaviour is frequency dependent. This forms the basis of the time-temperature superposition (TTS) technique.

The time-temperature superposition principle describes the equivalence of time and temperature. This means that for a given temperature molecular effects will occur at a given frequency however at a different temperature the same molecular effects will occur at higher or lower frequencies dependent upon the temperature change. The change occurs in a systematic way governed by the Williams-Landel Ferry (WLF) equation [2]:

$$\log(a_T) = \frac{-c_1(T - T_0)}{c_2 + T - T_0}$$

where, if applied near the glass transition temperature $C_1 = 17.4$ and $C_2 = 51.6$ typically.

Using the WLF equation, a superposition can be used to describe temperature dependent behaviour in stress relaxation (or modulus). Relaxation curves made at different temperatures across a range of frequencies are superposed by horizontal shifts along a logarithmic time scale to give a single master curve. Using the shift factors (SF) generated from this exercise and assuming that Arrhenius treatment is acceptable, then the acceleration factors (AF) can be considered as the ratio of shift factors and thus derived by using the equation [3]:

$$AF = \frac{SF(T + \Delta T)}{SF(T)}$$

where $SF(T)$ is the shift factor at a temperature of $T$ and $SF(T + \Delta T)$ is the corresponding value at a temperature rise of $\Delta T$.

DMA analysis provides the first estimate of an acceleration factor which should be subsequently verified by a suitable set of experiments. This is the principle behind the second phase of this investigation where a lifetime test is subsequently performed on materials using a representative test configuration and conditions. This then allows verification of the acceleration factors as well as an actual measurement of the materials change.

3 TTS ANALYSIS

Time-temperature superposition analysis was performed using DMA in a shear mode. Frequency sweeps were performed from 700Hz to $1 \times 10^{-3}$Hz with ten steps per decade using a force amplitude of 5.0 Newton’s, a displacement amplitude of 10µm across a temperature range of -100°C to +120°C at steps of 20°C. Additional frequency sweeps were then performed on new samples across the temperature range of 10-200°C using temperature intervals of 5°C.

![DMA raw data and temperature profile](image)

**Table 2: Acceleration factors based on an 80°C reference temperature**

<table>
<thead>
<tr>
<th>Test Temperature</th>
<th>Conservative</th>
<th>Optimistic</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>90</td>
<td>2.78</td>
<td>3.05</td>
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<td>110</td>
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</tr>
<tr>
<td>120</td>
<td>45.1</td>
<td>50.9</td>
<td>48.0</td>
</tr>
<tr>
<td>130</td>
<td>74.6</td>
<td>138</td>
<td>95.4</td>
</tr>
<tr>
<td>140</td>
<td>155</td>
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<tr>
<td>150</td>
<td>668</td>
<td>1080</td>
<td>814</td>
</tr>
<tr>
<td>160</td>
<td>1960</td>
<td>2900</td>
<td>2320</td>
</tr>
</tbody>
</table>

The silicone material was tested in the DMA by applying frequency sweeps at different fixed temperatures to obtain a series of curves for inclusion in the TTS analysis. The use of different temperatures allows a shift along the frequency scale (and hence time scale) so that very slow processes – such as creep
can be mapped to the relevant temperature. The delta in temperature between adjacent values can then be utilised to derive an acceleration factor for utilisation in a more specific configured test. The raw data utilised for this analysis is shown in Fig. 4 along with the temperature program.

So as to provide a range of estimates for a best and worst evaluation both a conservative and optimistic estimate was performed. The conservative and optimistic DMA evaluations are provided in Fig. 5 and Fig. 6 respectively. Both of these analyses are performed on the same data set from Fig. 4 and attempt to fit the data as accurately as possible. The conservative values however, generally have a larger overlap of the frequency curves than the optimistic ones resulting in a shorter extension into the low frequencies. Since this technique depends upon a manual best fit of the curves this approach is used where the data does not generate an obvious concise fit but instead shows appreciable noise and requires the use of vertical shifts in the data. It should be noted that all of the curves are interdependent and move according to the position of each other therefore fitting of the data is a compromise of all of the curves.

Fig. 5. TTS analysis using conservative shift factors

Fig. 6. TTS analysis using optimistic shift factors
Where necessary vertical shifting was also performed to optimise the fit. The vertical shift corresponds to a subsequent correction of the geometry factor for the sample concerned and has no effect upon the frequency shifts.

The acceleration factors derived are summarised in Table 2. The derived average acceleration factors were utilised in the ongoing ageing campaign.

4 AGEING CAMPAIGN

Samples of the silicone seal materials were initially measured for thickness and then mounted on the test fixture shown in Fig. 7 and Fig. 8. The seal sits on a steel base plate with a steel cylinder mounted above to apply the correct load of 4.71N. For each temperature two seals were tested utilising three cylinder sets. The third cylinder set was a “dummy” set to correct for expansion/contraction effects associated with the test fixture. Each cylinder has three probes mounted radially to accurately monitor expansion/contraction. Compression was determined by averaging the response from each of the probes and subtracting the fixture response as determined by the dummy cylinder. Each environmental chamber and test fixture was monitored by a series of thermocouples mounted at various locations to ensure accurate temperature control was maintained throughout.

The compression response raw data of the seals with time is shown in Fig. 9. It can be seen from Fig. 9 where a chamber reset occurred for 140°C/150°C. This reset was associated with problems identified in the test data at 140°C which showed anomalous reading and steps in height which are believed to be associated with sticking of the displacement probes at the elevated temperatures. These effects prevented the
data from being utilised and an investigation is presently underway to identify the fault with the probes.

Fig. 10 gives the seal compression data showing all the cylinders except for the 140°C cylinder 7 data which is off-scale. All of the data shown is normalised to a zero reference point. From the data in Fig. 10 it is clear that the 140°C cylinder 9 (and the off-scale 140°C cylinder 7) shows significantly different behaviour to the other cylinders. Large steps are observed in this data as well as little or no compression between the instrument generated steps. Whilst it cannot be excluded that the low compression levels at 140°C are material related it seems more likely that the effect is generated by something in the test configuration. Fig. 10 also shows a delta between the two 80°C reference samples. Despite this delta the curves show very similar behaviour. The delta corresponds to approximately 7µm of displacement which is similar in magnitude to the level of error associated with the probes.

To apply the TTS acceleration factors the data needs to be manipulated in a logarithmic scale (see Fig. 11). The use of the logarithmic scale tends to emphasise the shape of the early lifetime data and again it can be seen from Fig. 11 that the 150°C test shows a different behaviour to the 110/80°C tests. On the basis of this anomaly it cannot be determined if the 150°C data is valid. Nevertheless the latter data does show a trend which could be extended back to the 80/110°C data and possibly will become clearer as the test campaign proceeds. For this reason the data is included in this script.

Fig. 12 shows the compression data after the average acceleration factors from Table 6 are applied. It can be seen that there is a good correlation between the 80°C cylinder 3 and the 110°C data and an off-set with the cylinder 1 data. Despite the difference in the two cylinders the correlation is good. On the basis that there is a reasonable correlation between the 80°C and 110°C data the lifetime campaign presently provides data for 1264 days at 80°C. Further it can also be stated that the 80°C and 110°C exposure campaigns show a good correlation using the average acceleration factors and that no modification of the acceleration factor values is required at present. Evaluation of the 140°C and 150°C test campaigns shows significant anomalies in the test data. There are some indications that for the 150°C corrected data, if the initial first days test data is discounted, the remaining information gives an upward trend similar to the lower temperature data however there is insufficient overlap in the other
temperatures to ascertain if this trend is consistent with the thermal ageing of the rubber or purely coincidence.

5 FURTHER INVESTIGATIONS

The data reported here is part of an ongoing campaign. The key to successful TTS experiments is multiple testing at different intermediate temperatures to enable overlap of multiple curves and thus gain confidence in the acceleration factors. This campaign will include a number of additional temperature point over a 1 year campaign up to 150°C. Once confidence is gained in the acceleration factors a similar campaign is to be performed at valve level.

6 CONCLUSIONS

The ageing campaign has been successfully initiated and progressed to 67 days for 80°C and 110°C. Additional data has been obtained at 140°C and 150°C however this data is incongruous with that seen in the lower temperatures. It is believed that the deviations seen here may be associated with the probes sticking at the higher temperatures. The average acceleration factors from DMA estimates produced a good correlation between the 80°C and 110°C test runs. By applying the acceleration factors for 110°C the lifetime behaviour of the silicone seal under compression can be extrapolated to 1264 days (approximately 3.5 years) at 80°C.

7 REFERENCES


Fig. 12. Compression data shifted with average acceleration factors