



AEROSPACE INFORMATION REPORT

AIR1412™**REV. E**

Issued	1976-11
Revised	2018-01
Stabilized	2023-07

Superseding AIR1412D

Designing for Long Life with Elastomers

RATIONALE

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FOREWORD

The properties of elastomers change with time and temperature; in some cases, these changes are substantial. As a result of long-term storage stability problems with some early elastomeric materials, the aerospace industry to a large extent has become accustomed to the application of age controls on O-rings, hoses, and certain other rubber products. This has proven to be very costly, time-consuming, and unwieldy. Additionally, elastomeric materials qualified for service based on the results of short-term simulation tests conducted only at service temperature extremes have not always performed adequately in the field. Accelerated tests, when required, should be performed significantly above the continuous service temperature to provide a meaningful estimate of life at reduced temperatures. However, the aging temperature must not be so high as to change the degradation mechanism and thereby give misleading results (see references 8 to 18 below).

Replacement and reassembly of parts have been found to lower reliability. Maintenance on very complex aerospace products is difficult to carry out because of compactness of these products and disassembly required to gain access to seals or other rubber goods. The reliability and cost requirements of aerospace components are very high, hence short life, unreliable elastomeric parts cannot be tolerated. Long life elastomers are available for use in aerospace designs. It, therefore, follows that designing for long life is a much more viable approach.

The designer must convey a specific requirement to all concerned that he is building critical aerospace equipment intended for long life and high reliability. This can be as straightforward as a detailed drawing note citing the life requirement in years and the expected environments. This overall requirement has to be backed up by specific elastomer material performance and mechanical property specification requirements, as well as due consideration to application requirements. Moreover, the designer cannot assume that published specifications or proprietary callouts will automatically provide elastomeric performance to meet his specific needs.

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1. SCOPE

This document lists those guidelines recognized as being essential for consideration by the designer who is preparing to select an elastomer as part of an aerospace design.

1.1 Purpose

To provide guidelines to the aerospace designer in the testing and selection of elastomers so that long life service will be realized in critical components.

2. APPLICABLE DOCUMENTS

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1 Related Publications

The following publications are provided for information purposes only and are not a required part of this SAE Aerospace Technical Report.

1. F. R. Eirich, ed., "Science and Technology of Rubber", Academic Press, New York, 1978.
2. A. V. Tobolsky and H. F. Mark, ed., "Polymer Science and Materials", John Wiley and Sons, New York, 1980.
3. H. Liebowitz, ed., "Fracture - An Advanced Treatise", Vol. VII, Academic Press, New York, 1972.
4. H. F. Mark and N. G. Gaylord, N. J. Bikales, ed., "Encyclopedia of Polymer Science and Technology", Vol. 8, p. 419, Interscience Publishers, New York, 1968.
5. U. Meier, J. Kuster, and J. F. Mandel, "Rubber Chemistry and Technology", Vol. 54, 254 (1984).
6. L. P. Smith, "The Language of Rubber", Butterworth-Heinemann Ltd., Oxford, UK, 1993.
7. A.N. Gent, ed., "Engineering with Rubber – How to Design Rubber Components", 2nd Edition, HanserGardner Publications Inc., Cincinnati, Ohio, 2001.
8. K. T. Gillen, J. Wise and R. L. Ciough, "Novel Techniques Applied to Polymer Lifetime Predictions", <http://www.osti.gov/scitech/servlets/purl/10113498/>.
9. K. T. Gillen, "Modulus Profiling of Polymers", Polymer Degradation and Stability, Vol. 17, pp. 31-47, 1987.
10. J. Wise, "An Ultrasensitive Technique for Testing the Arrhenius Extrapolation Assumption for Thermally Aged Elastomers", Polymer Degradation and Stability, Vol. 49, pp. 403-418, 1995.
11. K. T. Gillen, "Extrapolation of Accelerated Aging Data – Arrhenius or Erroneous?", Trends in Polymer Science, Vol. 5 (8) pp. 250-257, August 1997.
12. M. Celina, "Oxidation Profiles of Thermally Aged Nitrile Rubber", Polymer Degradation and Stability, Vol. 60, pp. 493-504, 1998.
13. K. T. Gillen, "The Wear-Out Approach for Predicting the Remaining Lifetime of Materials", Polymer Degradation and Stability, Vol. 71, pp.15-30, 2001.
14. K. T. Gillen, M. Celina and R. Bernstein, "Validation of Improved Methods for Predicting Long-term Elastomeric Seal Lifetimes from Compression Stress-Relaxation and Oxygen Consumption Techniques", Polymer Degradation and Stability, Vol. 1, pp. 25-35, 2003; <http://www.sciencedirect.com/science/article/pii/S0141391003001599>.

15. K. T. Gillen, "Methods for Predicting More Confident Lifetimes of Seals in Air Environments", Rubber Chemistry & Technology, 73, 2, pp.265-283, 2000; <http://rubberchemtechnol.org/doi/abs/10.5254/1.3547590>.
16. K. T. Gillen, "New Method for Predicting Lifetime of Seals from Compression-Stress Relaxation Experiments", Die Angewandte Makromol. Chemie, 261/262, 4619, pp. 83-92, 1998.
17. K. T. Gillen, "Predicting and Confirming the Lifetime of O-rings", Polymer Degradation and Stability, 87, pp.257-270, 2005.
18. M. Celina, "Accelerated Aging and Lifetime Prediction: Review of Non-Arrhenius Behavior Due to Two Competing Processes", Polymer Degradation and Stability, Vol. 90, pp. 395-404, 2005.
19. MIL-HDBK-695, Rubber Products: Recommended Shelf Life, Dept. of Defense.

2.2 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

AMS2810	Identification and Packaging Elastomeric Products
AMS2817	Packaging and Identification of Molded Elastomeric Seals and Sealing Components
ARP5316	Storage of Elastomer Seals and Seal Assemblies Which Include an Elastomer Element Prior to Hardware Assembly

2.3 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM D2990	Tensile, Compressive, and Flexural Creep and Creep Rupture of Plastics
ASTM D3045	Heat Aging of Plastics Without Load
ASTM D6147	Vulcanized Rubber and Thermoplastic Elastomer – Determination of Force Decay (Stress Relaxation) in Compression
ASTM E1641	Standard Test Method for Decomposition Kinetics by Thermogravimetry Using the Ozawa/Flynn/Wall Method

2.4 ISO Publications

Copies of these documents are available online at <http://webstore.ansi.org/>.

ISO 3384	Rubber, vulcanized – Determination of stress relaxation in compression at ambient and at elevated temperatures
ISO 6056	Rubber, vulcanized or thermoplastic – Determination of compression stress relaxation (rings)
ISO 27996	Aerospace Fluid Systems – Elastomer Seals – Storage and Shelf Life

2.5 UL Publications

Available from UL, 333 Pfingsten Road, Northbrook, IL 60062-2096, Tel: 847-272-8800, www.ul.com.

UL 746B	Polymeric Materials: Long Term Property Evaluations
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3. GUIDELINES

The following minimal guidelines are presented to aid the designer in the selection of long life elastomeric parts:

- a. The elastomer compound should be resistant to oxidative attack.
- b. The elastomer compound should be resistant to ozone cracking.
- c. The elastomer compound should be resistant to the service fluid media involved.
- d. The elastomer compound should suitably resist permanent set, creep, and/or stress relaxation.
- e. The elastomer compound should possess a sufficient safety factor in mechanical properties to allow for known degradation.
- f. The elastomer compound should possess sufficient resistance to cut, tear, and abrasion to give the required life.
- g. The elastomer compound should resist special environments such as temperature, water, humidity, radiation, fungus, hard vacuum, corrosion, cleaning and processing media, and the like when required.
- h. Functional hardware should be artificially aged as part of normal qualification testing.
- i. The quality and product control systems should be explicit and be enforced to make certain the specified elastomer is received and is properly packaged and stored.

It must be recognized that it is not necessary that an elastomer meet all of the above guidelines in all cases. If, for example, ozone resistance is not a major item of consideration, some polymers such as nitrile-butadiene rubber (NBR) with poor ozone resistance could perform satisfactorily.

4. DISCUSSION

Discussions covering each of the guidelines are as follows:

4.1 Oxidative Attack

Most elastomers are subject to oxidative attack, but antioxidants have been developed to mitigate against this mode of degradation. Certain elastomers, such as fluorocarbons, silicones or ethylene propylenes, have a greater resistance to oxidation and may require only a lower level of antioxidants, or possibly, even none at all. The control of oxidation is a complex empirical problem. Hence, specifications do not specify the chemical type(s) of antioxidants or the amount to be used. Rather, they define the effect using accelerated heat oven tests to set limits to the extent of degradation. The designer must insure that a suitable test of this type is included in the specification invoked. The time and temperature of an accelerated test such as defined in ASTM D3045 can provide an indication as to how long this material may be used safely in a design at a rated service temperature. However, the accelerated heat aging temperature must not be so high as to change the degradation mechanism and thereby give misleading results (see references 8 to 8 above). Antioxidant technology has been developing for almost 75 years and extensive tests have shown that most quality antioxidant protected elastomer compounds will resist gross degradation due to oxidation for periods greater than ten years at ambient temperature.

4.2 Ozone Cracking

Chemically unsaturated elastomers are subject to ozone cracking. This is familiar to most persons who have seen ozone cracking on the sidewalls of their automotive tires. In former times, this was incorrectly attributed to sunlight. Ozone is an extremely active form of oxygen. Its attack is not to be confused with oxidative attack as discussed above. Chemically saturated elastomers can be used with little or no concern for ozone cracking. These include among others: polyacrylates, ethylene propylene, fluorocarbons, silicones, butyl rubber, and polysulfides.

4.2.1 The more common chemically unsaturated elastomers which may be attacked by ozone include: neoprene, nitrile-butadiene rubber (NBR), polybutadiene, natural, isoprene and styrene-butadiene rubber (SBR). These should be used only where protected from atmospheric or other source of ozone, or provided by addition of antiozonants or by physical separation from exposure to ozone. As with antioxidants, the requirement is implemented by tests for resistance to ozone cracking, rather than by tests for antiozonant content.

4.3 Fluid Media

Elastomers can be dissolved, swollen, softened, or degraded by fluids that have chemical structures or solubilities similar to their own. There is no such thing as an elastomer that resists all fluid media. The rubber must be compatible with any operational fluid media (or nonoperational fluid such as lubricants or cleaning fluids) with which it comes in contact at any time. True compatibility will depend on the concentration of the fluid, temperature, duration of exposure, and the state of cure of the elastomer.

4.3.1 Elastomer specifications often have fluid immersion tests in various standard fluids under heat-accelerated conditions. Volume change is one measurement. These tests may be in fluids never seen in application since the intent is to verify appropriate state of cure of the rubber specimen. Negative volume change (shrinkage) is almost always considered unacceptable, but positive changes up to 25% or even higher with some designs may be acceptable depending upon the application. Unless carefully structured it has been found that heat-accelerated volume change tests may not always predict what will happen at room temperature, i.e., positive volume changes were found in the heat-accelerated tests, but shrinkage was experienced in long-term usage at room temperature.

Pneumatic sealing conditions may vary from those typically experienced with fluids contained in hydraulic systems and engine components. In some applications, more squeeze may be required, perhaps double the squeeze required in hydraulic systems. The use of nonstandard O-rings and/or modification of groove dimensions may be advisable. In others, less squeeze may be required, for example, in dynamic pneumatic cylinders where high sealing forces tend to produce binding.

4.3.2 Changes in physical properties are also tested under heat-accelerated conditions. A truly meaningful immersion test uses the actual exposure fluid in the actual environments (except for temperature) and for extended periods. However, the accelerated heat aging temperature must not be so high as to change the degradation mechanism and thereby give misleading results (see references 8 to 18 above). Parts or material so exposed must be tested for change of volume, ultimate tensile strength, tensile stress at a specified elongation (called "modulus"), elongation, and hardness as a minimum.

4.4 Creep, Stress Relaxation, and Permanent Set

When elastomers are used as sealing devices, they are compressed or strained to some degree. When that strain is removed after a period of time, the material will not fully recover its original shape. Creep, stress relaxation and compression set are undesirable related phenomena, which occur in all elastomeric articles and reflect the inherent viscoelastic nature of the elastomer and the limited stability of crosslinks of its vulcanizate.

Creep is a time dependent increase in deformation under conditions of constant stress. Continuous stress relaxation is decay in stress, as a function of time, under conditions of constant strain. It is of great importance to sealing devices such as O-rings and gaskets. Both creep and stress relaxation are significant because they often play a role in the failure of rubber components. Set, often referred to as "permanent set", or irrecoverable creep, is the permanent deformation which remains when a material is released from the strain imposed and is measured in tension, or more commonly in compression.

Both stress relaxation and creep are the result of physical and chemical relaxation processes. The physical process is due to the viscoelastic nature of rubber and usually decreases linearly with the logarithm of time. It is associated with reorientation of the elastomer molecular network under strain, with disengagement and rearrangement of chain entanglements. The chemical process is caused by chain scission or isomerization of crosslinks and usually occurs linearly as a function of linear time. Both processes occur simultaneously. Physical relaxation predominates at short times, while chemical effects are more significant at longer duration. Chemical effects are more obvious under conditions that favor oxidation, such as high temperatures and absence of chemical antioxidants in rubber compounds.

4.4.1 Creep

When an elastomeric component is subjected to a static load, the load will cause a progressive increase in deformation as a function of time. Bi-axial stress relaxation on compression in rubber consists of both physical creep and chemical creep (due to molecular chain breaking).

4.4.2 Stress Relaxation

When a constant strain is imposed on an elastomer, the force necessary to maintain that strain is not constant but decays with a non-linear dependence on time from the initial maximum to an eventual equilibrium state. This phenomenon of force decay is called stress relaxation and is of great importance in rubber sealing devices such as O-rings, packings and gaskets. Stress relaxation can be the dominant factor that limits the effective life of the sealing device. ASTM D6147, ISO 3384, and ISO 6056 reference a test method for determining stress relaxation in rubber.

4.4.3 Permanent Set

Permanent Set, or unrecoverable strain (or unrecovery from % compression), is the failure of an elastomeric part to completely recover from prolonged deformation in a finite time, usually 30 minutes for test purposes. In most sealing configurations, the seals are deformed to a defined level of strain and not subjected to a constant load. Permanent set may occur in tension, shear, torsion, or compression. Compression set resistance is the property most often specified in seal specifications. As a guideline, for compression seals, the permanent set taken with time should not allow the squeeze to fall below 10%.

Sealing force, an elastomer seal's resistance to a given deflection, is a better indicator of sealing ability, but one more difficult to measure than compression set. When performed with the appropriate equipment, test configuration and test method, compressive stress relaxation (CSR) results can be faster, easier and less expensive to test, and provide results that are much higher in resolution and more useful and relevant to the performance of seals in static sealing applications. It is important to note that even though this document notes the values of measuring sealing force or CSR, there are no aerospace guidelines or specifications that currently use this test methodology.

Compression set and sealing force are related in the sense that a material with high compression set will generally exhibit poor sealing force retention. However, in some instances, an elastomeric material with reasonable compression set may provide poor sealing force retention with subsequent leakage. Conversely, a material with high compression set and poor sealing force retention may not always leak since the retained absolute sealing force may be sufficient to maintain a seal. Therefore, whenever possible, sealing force measurements should be used in design of critical long life sealing components.

4.5 The compression set resistance and sealing force retention of a compound depend on its state of cure. However, for economic reasons, production cure cycles are kept as short as possible. The designer must insist on a properly cured seal, controlled by invoking a specific accelerated compression set test and by running lot acceptance tests on actual production parts. Utilizing test parameters appropriate to the generic polymer base for the compound, the compression set for critical parts as a general guide should preferably be between 20 to 55%, otherwise the ability to discriminate effectively between properly and improperly cured (undercured or overcured) rubber specimens is diminished. With a few specific types of elastomers, such as the fluorocarbons, room temperature compression set tests must be run in addition to accelerated temperature tests. Accelerated tests using Arrhenius aging, may be run using ASTM D3045 or ASTM D2990, or alternately, UL 746B as guidelines. However, the accelerated heat aging temperature must not be so high as to change the degradation mechanism and thereby give misleading results (see references 8 to 18). In addition, ISO 3384 and ISO 6056 reference stress relaxation in compression.

4.6 High Initial Tensile and Elongation Properties

The higher the dilution of the elastomer with fillers and the poorer the strengthening action of the specific class of reinforcing fillers, the lower the strength and elongation of the resulting compound. Similarly, high dilution with plasticizers will reduce mechanical properties. Hence, tensile strength and elongation are primarily an index of quality rather than properties for use in stress analysis. Low strength (low quality) elastomer compounds generally age poorly. Further, high quality (high strength) compounds have better abrasion and tear resistance and can degrade to a greater extent and for much longer periods of time and still remain functional. Extensive studies have shown that with many elastomers the tensile stress at 100% elongation will approximately double in about 3 years of aging at room temperature and will remain fairly constant from 3 to 10 years and beyond. Hardness does not measure this effect. Elongation drops continuously with aging time. Many concepts consider 100% elongation as the end point because the material becomes too rigid and inelastic to be useful in most elastomeric applications.