WHITE SANDS TEST FACILITY

NASA Johnson Space Center
Las Cruces, NM

Proposed G114-06 Amendment

Standard Practices for

Evaluating the Age Resistance of Polymeric Materials Used in Oxygen Service

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J. M. Waller

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17 October 2006

ASTM Subcommittee G04.02 on Recommended Practices for Flammability and Sensitivity of Materials in Oxygen and Oxygen-Enriched Atmospheres, Silver Spring MD

G04.02 Subcommittee Balloting Results

05 Aug 2005 Close Date

33 Affirmatives   16 Abstentions   1 Negative

- **2 Affirmatives with Comment**
  - J. Cronk
    - reference G126 for certain terminology:
      - *aging*
      - *artificial aging*
      - *natural aging*
  - D. Baldwin
    - editorial

G04.02 Subcommittee Balloting Results

- **1 Abtension with Comment**
  - B. Werley

    "I am not sure I agree with the existing standard focused on polymers only, although there were only polymer test standards to cite as examples when this was first written in 1992. However, I would have seen no problem using it with metals. I, to the contrary, think the revision makes it more 'polymer-centric' and also swings it's spirit away from aging prior to fire testing and shifts it more to mechanical reliability which is something the committee used to avoid even though it is clearly important to the overall subject, and appear to be important very specifically to the CTFE issue. My own druthers would have been to incorporate these materials into a new standard dealing with mechanical testing, or even into a "Resources Adjunct," but clearly this is a committee decision."

G04.02 Subcommittee Balloting Results

- **Response To B. Werley's comment:**
  - Oxygen aging can cause a more severe drop in mechanical properties, than changes in either the AIT or ΔH_c[§]

§ Waller, J. M., Haas, J. P., Beeson, H. D., Polymer-Oxygen Compatibility Testing: Effect of Oxygen Aging on Ignition and Combustion Properties," *Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Ninth Volume, ASTM STP 1395*, T. A. Steinberg, H. D. Beeson, B. E. Newton, Eds., American Society for Testing and Materials, West Conshohocken, PA, 2000.

  - Severe mechanical property loss could lead catastrophic component failure, including secondary fire due to leakage, before primary ignition or combustion of the material occurs.

G04.02 Subcommittee Balloting Results

- **Response To B. Werley's comment:(cont.)**

  - Polymeric materials that are susceptible to oxidative degradation *generally* become less susceptible to ignition and combustion after aging (e.g., CR elastomers, nylon, polyolefins, hydrocarbon elastomers)
    - *Exception*: high surface area chars which can detonate.

Note 1— **Warning:** If integrity of the component has not been compromised by partial combustion of the polymeric part, and partial combustion leads to formation of a high surface area char, the component may pose a detonation hazard if left in oxygen service.

  - Polymeric materials that are immune to oxidative degradation are also *generally* immune to ignition and combustion property changes (e.g., PTFE, PCTFE, MVQ elastomers, FFKM elastomers, FEP, MFA, PFA)

Representative Oxygen Aging Effects

Table 1 — *Effect of Aging on Tensile Properties*

	as-received UTS	O_2-aged UTS		N_2-aged UTS[b]
Elastomers:	MPa	620 kPa-aged MPa (%)	6200 kPa-aged MPa (%)	6200 kPa-aged MPa (%)
Neoprene C873-70	16.1	8.7 (-46)	0 (-100 %)	16.4 (*negl.*)
Thermoplastics:				
Zytel 42	66.3	32.9 (-50)	12.5(-81)	74.9 (+8)
	as-received ε_{max}	O_2-aged ε_{max}		N_2-aged ε_{max}[b]
Elastomers:	%	620 kPa-aged % (%%)	6200 kPa-aged % (%%)	6200 kPa-aged % (%%)
Neoprene C873-70	580	63 (-89)	0 (-100 %)	512 (-11)
Thermoplastics:				
Zytel 42	444	10 (-98)	3 (-99)	430 (*negl.*)

Table 2 — *Effect of Aging on Ignition and Combustion*

	as-received AIT	O_2-aged AIT		N_2-aged AIT[b]
Elastomers:	°C	620 kPa-aged °C (%)	6200 kPa-aged °C (%)	6200 kPa-aged °C (%)
Neoprene C873-70	262	328 (+25)	317 (+21)[c]	255 (-3)
Thermoplastics:				
Zytel 42	188	191 (*negl.*)	203 (+8)	185 (*negl.*)
	as-received ΔH_c	O_2-aged ΔH_c		N_2-aged ΔH_c[b]
Elastomers:	cal g^{-1}	620 kPa-aged cal g^{-1} (%)	6200 kPa-aged cal g^{-1} (%)	6200 kPa-aged cal g^{-1} (%)
Neoprene C873-70	6720	6300 (-6)	5900 (-12)[c]	6680 (*negl.*)
Thermoplastics:				
Zytel 42	8830	7480 (-15)	7380 (-16)	7550 (-14)

G04.02 Subcommittee Balloting Results

- **1 Negative**
  - B. Newton

    "I am voting negative on G114 only because I do not believe the current standard has been given adequate opportunity for review and debate. … the standard has changed substantially from the the previous version and has only been provided to the review task group ... recently. No discussion of the changes has been held to my knowledge a consensus from the group has not been derived. … I believe this ballot is premature."

Negative withdrawn at April meeting

G114-06 Amendments

1. Adding new 1.2

1. Scope

1.1 These practices describe several procedures that are used to determine the age resistance of plastic, thermosetting, and elastomeric materials exposed to oxygen-containing media.

1.2 While these practices focus on evaluating the age resistance of polymeric materials in oxygen–containing media prior to ignition and combustion testing, they can also be used to evaluate the age resistance of metals.

1.3 These practices address established procedures that have a foundation of experience for aging in air, but which have not been validated for aging in oxygen-enriched media containing greater than 25 mole % oxygen.

1.4 The results of these practices may not give exact correlation with service performance since service conditions vary widely and may involve multiple factors. This standard may be used to evaluate materials on a laboratory comparison basis.

1.5 Three procedures are described for evaluating the age resistance of polymeric materials depending on application and information sought.

1.5.1 *Procedure A: Natural Aging*—This procedure is used to simulate the effect(s) of one or more service stressors on a material's oxygen resistance, and is suitable for evaluating materials that experience continuous or intermittent exposure to elevated temperature during service.

1.5.2 *Procedure B: Accelerated Aging Comparative Oxygen Resistance*—This procedure is suitable for evaluating materials that are used in ambient temperature service, or at a temperature that is otherwise lower than the aging temperature, and is useful for developing oxygen compatibility rankings on a laboratory comparison basis.

1.5.3 *Procedure C: Accelerated Aging Lifetime Prediction*—This procedure is used to determine the relationship between aging temperature and predefined level property change, thereby allowing predictions to be made about the effect of prolonged service on oxidative degradation.

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G114-06 Amendments

2. Changes to 1.3

1.3 These practices address established procedures that have a foundation of experience for aging in air, but which have not been validated for aging in oxygen-enriched media containing greater than 25 mole % oxygen.

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1.5.3 *Procedure C: Accelerated Aging Lifetime Prediction*—This procedure is used to determine the relationship between aging temperature and predefined level property change, thereby allowing predictions to be made about the effect of prolonged service on oxidative degradation.

1.6 The values stated in SI units are to be regarded as the standard, however, all numerical values must also be cited in the systems in which they were actually measured.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 10.*

2. Referenced Documents

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G114-06 Amendments

3. Adding D2863 and G126 to Section 2 - BALLOT

G 114

- D 638 Test Method for Tensile Properties of Plastics
- D 1349 Practice for Rubber—Standard Temperatures for Testing
- D 1708 Test Method for Tensile Properties of Plastics By Use of Microtensile Specimens
- D 2240 Test Method for Rubber Property—Durometer Hardness
- D 2512 Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)
- D 2863 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)
- D 3045 Practice for Heat Aging of Plastics Without Load
- D 4809 Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method)
- D 5510 Practice for Heat Aging of Oxidatively Degradable Plastics
- G 63 Guide for Evaluating Nonmetallic Materials for Oxygen Service
- G 72 Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment
- G 74 Test Method for Ignition Sensitivity of Materials to Gaseous Fluid Impact
- G 86 Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Pressurized Oxygen Environments
- G 125 Test Method for Measuring Liquid and Solid Material Fire Limits in Gaseous Oxidants
- G 126 Terminology Relating to the Compatibility and Sensitivity of Materials in Oxygen Enriched Atmospheres

G114-06 Amendments

4. Terminology Section changes - BALLOT

DIN 53508 Accelerated ageing of rubber

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *aging*—see G126.

3.1.2 *accelerated aging*—a type of artificial aging whereby the effect of prolonged exposure during service is simulated by aging at elevated temperature.

3.1.3 *artificial aging*—see G126.

3.1.4 *oxidative degradation*—physical or mechanical property changes occurring as a result of exposure to oxygen–containing media.

3.1.5 *oxygen-containing media*—air media containing 21 mole % oxygen, and oxygen-enriched media containing greater than 25 mole % oxygen.

3.1.6 *oxygen resistance*—resistance of a material to ignite spontaneously, propagate by sustained combustion, or undergo oxidative degradation.

3.1.7 *oxygen service*—applications involving the production, storage, transportation, distribution, or use of oxygen–containing media.

3.1.8 *natural aging*—see G126.

3.1.9 *physical aging*—aging that occurs during normal storage which is a function of time after production.

4. Summary of Practice

4.1 This practice can be used to evaluate systematically the effect of natural aging (Procedure A) or accelerated aging (Procedures B and C) on oxygen resistance. To apply its principle, the user first characterizes the material, then subjects the

[10] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111.

Deleted: the exposure of a material to stressors including time, temperature, pressure, chemical exposure, humidity, abrasion, ionizing radiation, light, impact with gas or particles, mechanical load (static or dynamic), or any other stressor that may be present during service. These stressors may be present individually or in combination.

Deleted: aging which is a function of combined stressors lying outside the domain of normal storage and service domains; or alternatively, aging in which exposure to temperature is used to produce an effect that simulates that of time. The degree of artificiality may vary considerably. An example of mild artificiality might be exposure of a material to a greater pressure than it experiences during service. An example of extreme artificiality would be the use of sand paper to increase a material's surface roughness to simulate particle-impact abrasion that occurs during service. A high degree of artificiality affects the strength of conclusion that ...

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G114-06 Amendments

5. Add new 5.3 and Note 1 - BALLOT

5. Significance and Use

5.1 This practice allows the user to evaluate the effect of service or accelerating aging on the oxygen resistance of polymeric materials used in oxygen service.

5.2 The use of this practice presupposes that the properties used to evaluate the effect of aging can be shown to relate to the intended use of the material, and are also sensitive to the effect of aging.

5.3 Polymeric materials will, in general, be more susceptible than metals to aging effects as evidenced by irreversible property loss. Such property loss may lead catastrophic component failure, including a secondary fire, before primary ignition or combustion of the polymeric material occurs. These practices, therefore, are considered to be more conservative than ignition and combustion tests since failures associated with excessive property loss will occur at lower exposure thresholds than failures associated with primary ignition or combustion of the aged part.

NOTE 1— **Warning:** If integrity of the component is not been compromised by partial combustion of the polymeric part, and partial combustion leads to formation of a high surface area char, the component may pose a detonation hazard if left in oxygen service.

G114-06 Amendments

6. Adding new Note 9 (after 11.3)

11. Testing of Unaged Specimens

11.1 To minimize repeatability errors, it is recommended that properties of the unaged sample be determined within 96 h of the start of the aging interval. Results on specimens which are found to be imperfect shall be discarded and retests shall be made.

11.2 The material should be in the exact condition for use prior to aging. Any cleaning should be consistent with cleaning required for the application of interest.

11.3 Test the material as specified in the test method(s) chosen: Test Methods D395 (compression set), D412 (tension – rubbers), D638 (tension – plastics), D1708 (microtension – plastics), D2240 (Durometer hardness), D2512 (liquid oxygen impact), D4809 (heat of combustion), G72 (AIT), G74 (gaseous oxygen impact), G86 (mechanical impact), G125 (fire limit), or other method as described in the Note below. If time is suspected to be a key aging parameter, retain some of the material in its original condition for later testing in concert with the aged material.

NOTE 9—Other property indicators that can be used to determine the age resistance of plastic, thermosetting, and elastomeric materials to oxygen–containing media include exothermicity testing using an Accelerated Rate Calorimeter, friction/rubbing testing, particle impact, promoted and hot wire ignition, electric arc testing, resonance, or internal flexing.

G114-06 Amendments

7. Adding Durometer before hardness in 13.1.2

13. Calculation

13.1 *Procedure A: Natural Aging, and Procedure B: Accelerated Aging Comparative Oxygen Resistance*

13.1.1 For properties such as tensile strength and elongation, the aging results shall be expressed as a percentage change for the given property:

$$P = [(A - O) / O] \times 100 \quad (3)$$

where:

P = percentage change in property,

O = original value, and

A = value after aging.

13.1.2 For properties like Durometer hardness, the aging results shall be expressed as an absolute change:

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G114-06 Amendments

8. Adding a citation to D2863 in 12.2.3 and 14.2.4 - BALLOT

12.2.3 *Temperature*—For materials used in elevated temperature service, aging at the same elevated temperature will simulate natural aging. In this case, the effect of temperature is determined directly. For materials used in ambient temperature service, exposure to elevated temperatures will simulate accelerated aging. In this case, an Arrhenius method is used to convert the effect of temperature to that of time, thereby allowing predictions to be made about the effect of time (prolonged service) on a given property. Aging at elevated temperature often leads to an increased AIT as determined by Test Method G 72. Oxidation caused by chemisorption of oxygen may cause a decrease in the heat of combustion as determined by Test Method D 4809, or may increase the oxygen index, see Test Method G 125 or D2863. Aging may lead to a cracking, loss of resiliency, and other physical and mechanical property loss, see Test Methods D 395, D 412, D 638, D 1708, D 2240. Aging may also lead to an increase in specific surface area, that can produce easily ignitable edges, hence ambient temperature mechanical impact ignition tests per D 2512 or G 86, or pneumatic impact ignition tests per G 74 may be worthwhile. If specific information about the effect of temperature up to 280°C (540°F) on impact ignition properties is desired, heated gaseous oxygen mechanical impact ignition tests per G 74, or heated gaseous oxygen pneumatic impact ignition tests per G 86 may be worthwhile.¶

14.2.4 For use of Test Method G 125 or D 2863, the change in oxygen index should be reported and a decrease should be called a degradation, and an increase should be called an enhancement.¶

G114-06 Amendments

9. Adding new 14.2.5 - BALLOT

14.2.1 For use of Test Method G72, the change in AIT should be reported, and a decrease in AIT shall be called a degradation, an increase is called an enhancement.

14.2.2 For use of Test Method G74, the change in reactive pressure should be reported, and a decrease in reactive pressure should be called a degradation, an increase an enhancement.

14.2.3 For use of Test Method G86, the change in reactive threshold energy should be reported and a decrease in threshold should be called a degradation, and an increase should be called an enhancement.

14.2.4 For use of Test Method G125 or D 2863, the change in oxygen index should be reported and a decrease should be called a degradation, and an increase should be called an enhancement.

14.2.5 For use of Test Method D2512, the change in reactive threshold energy should be reported and a decrease in threshold should be called a degradation, and an increase should be called an enhancement.

14.2.6 For use of Test Method D4809, the change in heat of combustion should be reported and an increase should be called a degradation and a decrease should be called an enhancement.

Proposed Action

- Issue a concurrent ballot to accept above revisions