Fluid Sealing Association

STANDARD

FSA-G-604-07

Oxidation Test Standard for
Flexible Graphite Gasket Materials

994 Old Eagle School Road, Suite 1019
Wayne, PA 19087-1866
Phone: (610) 971-4850
Fax: (610) 971-4859
www.fluidsealing.com
Email: info@fluidsealing.com

For a complete list of FSA publications, please contact:

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Suite 1019
Wayne, PA 19087-1866
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Test Method "A" – Method of determining relative oxidation resistance between different types or styles of flexible graphite.

Test Method "B" – Method of determining oxidation resistance between different types or styles of flexible graphite under closely controlled environmental conditions for the purpose of material comparison.

Background:
Flexible graphite is often chosen as the facing material in several different styles of metal reinforced gasket materials for its high temperature stability. The maximum temperature in which a flexible graphite faced gasket can be used is limited by the oxidation rate of the flexible graphite. The flexible graphite weight loss due to oxidation over time will result in total gasket mass loss, flange load loss and eventually to leakage. The oxidation rate of the flexible graphite is highly dependent on the temperature, and the amount of oxygen in contact with the graphite. Other factors include the purity of the graphite, presence of oxidation accelerating contaminants, presence of oxidation inhibiting elements, whether the oxygen is flowing or stagnant around the graphite, and the surface area exposed. Determining the effect of all of these factors and therefore the life expectancy of a gasket can be a difficult challenge especially when the flexible graphite is in different gasket configurations, in a flange, and subject to the varying temperatures and chemicals of the process. As difficult as it is to predetermine a flexible graphite gasket's life in service, it is relatively easy to rank the performance of different flexible graphites by a short-term oxidation test. This test cannot be used to determine a specific gasket's life in a specific application, but will give a relative oxidation resistance ranking of different flexible graphites.

A good quality oxidation inhibited grade of flexible graphite should have an oxidation rate of less than 10% when tested by Method A of this procedure. Typical oxidation rates should be around 3%.

A good quality non-inhibited grade of flexible graphite will have an oxidation rate of less than 20% when tested by Method A of this procedure. Typical results will be in the 10-15% range.

Any flexible graphite with an oxidation rate of over 20% as tested by Method A of this procedure, has probably been contaminated with impurities that increase the normal rate of graphite oxidation and are not recommended for high temperature gasket applications.

Scope:

The following test procedure describes two methods for the evaluation of graphitic materials in high temperature environments. Both Method "A" and Method "B" are acceptable methods for

determining weight loss at elevated temperatures. Method A is a short term general screening method. It utilizes an open-air oven. Due to contaminants and residuals that can contaminate the oven from sample to sample and to temperature and gas flow variation within the oven, the user must understand that the method is intended for the determination of gross differences in material comparisons, and must accept a small degree of inaccuracy. The method is intended to act as a general screening method and a preliminary step towards Method "B". Method "B" is more accurate and repeatable compared to Method A in that the results can be duplicated using similar test apparatus, test parameters and sample sizes (other than variation in the flexible graphite itself). Method "B" requires the use of a Thermal Gravimetric Analysis (TGA) analyzer, which more accurately monitors the test temperature and environment and electronically graphs the effects of extreme temperature on graphitic materials. Some styles of TGA analyzers can test up to 19 samples at the same time so accurate side-by-side comparisons can be conducted.

Method A

1.0 PURPOSE:
This test is a general screening method utilizing an open-air oven to determine short term flexible graphite oxidation weight loss.

2.0 EQUIPMENT:
2.1 Muffle furnace or oven capable of holding 670^{o}C
2.2 Stainless steel screen with ½ " mesh.
2.3 2" x 6" steel rule die
2.4 Analytical Balance
2.5 Timer

3.0 TEST PROCEDURE:

3.1 Die cut and identify three samples of flexible graphite sheet 2" x 6".

3.2 Weigh and record the weight of each sample to the nearest 0.0001 grams. Identify this weight as the original weight.

3.3 Place each sample on a stainless steel screen in a muffle furnace at 670^{o}C+/-2^{o}C for 60 minutes +/- 1minute. The stainless steel support screen should have approximately ½" openings and be supported off of the floor of the muffle furnace to allow air to reach all sides of the samples.

3.4 Remove the samples from the muffle furnace and cool in a desiccator until they reach room temperature.

3.5 Weigh each sample again and record the weight. Identify this weight as final weight.

3.6 Calculate the % oxidation = $\frac{\{(\text{original weight}) - (\text{final weight})\}}{(\text{original weight})} \times 100$

3.7 Average the results of the three samples and report as the % oxidation.

Variation in muffle furnace size, amount of air in the furnace, the amount of moisture in the samples, sample size variation, the temperature, and the temperature uniformity within the furnace can all affect the absolute value of the % oxidation. However, samples tested in the same lab set-up can consistently be ranked from high to low by their % oxidation. At elevated temperatures, the flexible graphite that has the least weight loss (lowest % oxidation) in this test would be expected to have the longest gasket life.

Method B

1.0 PURPOSE:
This TGA test method measures the weight changes of flexible graphite samples during heating under controlled atmospheres (capable of air, Oxygen, and Nitrogen). This TGA test method is used to compare the oxidation rate of different flexible graphite grades.

2.0 REFERENCES:
Thermo Gravimetric Analysis (TGA) tester Instruction Manual

3.0 DESCRIPTION:
Under the controlled environment, the TGA tester determines the weight change of samples of flexible graphite materials at the controlled temperature according to the selected analysis method.
The flexible graphite samples should have the same density and thickness (where possible). The sample size should be between 2.5 grams and 3.5 grams. The samples should be dried for 1 hour at 150^{o}C. The atmosphere should be air with a flow rate of 7.0 liters per minute. The temperature ramp up speed should be 10C^{o} per minute to the 150^{o}C drying temperature 1 hour long hold and then again 10^{o}C per minute to the final test temperature of 593^{o}C. This test temperature should be held for 24 hours and the total weight loss after drying should be recorded and compared. Three samples of each different flexible graphite grade should be tested at the same time.

4.0 EQUIPMENT:
4.1 Analytical Balance
4.2 Scissor
4.3 Crucibles
4.4 Weighing Dishes
4.5 Thermal Gravimetric Analysis (TGA) tester capable of meeting the sample size, air flow, heat up rates, hold times and temperatures as specified The LECO Corp Model TGA-601 or 701 has been shown capable of running this test method.

5.0 OPERATION PROCEDURE:

5.1 Sample Preparation – cut a rectangular sample approximately ¾" wide by the necessary length to obtain a sample weight of 2.5 grams to 3.5 grams. Wear rubber gloves while handling the flexible graphite samples to avoid any contamination. Samples should be folded (not torn or cut) to fit into crucibles. Maintain sample identification.

5.2 Follow TGA tester operating procedures.

5.3 Select gas supply : Air at a flow rate of 7.0 liters per minute

5.4 Select temperature rate of rise: 10 C^o per minute

5.5 Select Starting Temperature: 25^{o}C

5.6 Dry samples in the TGA tester for 1 hour at 150^{o}C. Use the dried sample weight for the weight loss percentage calculations.

5.7 Select Testing Hold Temperature: 593^{o}C

5.8 Select Hold Time: 24 hours

5.9 Load samples into TGA tester

5.10 Run tester program and record total weight loss after drying for each sample.

5.11 Average the results from the three replicate samples and report as % oxidation weight loss.

5.12 Report sample identification, % oxidation weight loss, test temperature, test temperature hold time, and any variation form the recommended test procedure.

There are a number of independent test labs and flexible graphite manufacturers' labs that have TGA testing equipment that can perform this test.

Appendix 1

FSA Round Robin Oxidation Test Method A

Samples are measured for weight loss after 1 hr exposure at 670ºC in a muffle furnace using Method A.
Sample size 2" x 6" x nominal thickness
Test Labs involved:
GrafTech
Teadit
Thermoseal
Flexitallic

Sample information:

			Grade	Density
Sample A	Nominal thk =	.010" avg	Inhibited 98%C	70#
Sample B	Nominal thk =	.010" avg	Not inhibited 99.5%C	40#
Sample C	Nominal thk =	.010" avg	Not inhibited 99.5%C	70#
Sample D	Nominal thk =	.015" avg	Not inhibited 95%C	70#
Sample E	Nominal thk =	.030" avg	Inhibited 98%C	70#

Sample F Nominal thk = .030" avg Chinese 70#

Sample data:

Initial wt. as cut	Sample A	Sample B	Sample C	Sample D	Sample E	Sample F
avg	2.25	1.29	2.30	3.32	6.63	6.45
std dev	0.047	0.059	0.028	0.051	0.116	0.116
max	2.36	1.37	2.35	3.4	6.83	6.67
min	2.17	1.16	2.25	3.22	6.29	6.11
AWU (3std dev/avg)	6.2%	13.7%	3.7%	4.6%	5.3%	5.4%

	Klinger/Thermosea	Teadit	GrafTech	Flexitallic UK
Sample A	3.11	5.72	8.61	12.72
	3.14	6.94	8.25	8.24
	3.64	11.76	8.22	7.83
	3.68	7.37	8.15	9.15
				11.41
Sample A avg	3.4	7.97	8.31	9.87
Sample A stdev	0.268	2.283	0.178	2.111
Sample B	27.06	52.09	39.61	69.2
	18.55	34.66	39.7	48.17
	24.78	36.03	39.88	59.31
	21.79	54.52	39.98	33.17
				48.72
Sample B avg	23.19	44.41	39.79	51.714
Sample B stdev	3.693	10.432	0.168	13.499
Sample C	7.45	21.98	17.14	21.73
	12.13	12.77	16.38	19.21
	10.52	24.38	16.61	20.33
	12.29	12.77	16.72	17.87
				22.4
Sample C avg	10.55	17.98	16.71	20.308
Sample C stdev	2.245	6.090	0.318	1.840
Sample D	27.36	34.17	32.11	46.74
	24.25	15.11	31.04	28.14
	20.79	36.43	30.16	41.79
	26.02	47.57	32.01	36.76
				43.07
Sample D avg	24.62	33.29	31.33	39.3
Sample D stdev	2.844	13.479	0.917	7.190
Sample E	1.59	2.02	2.3	3.49
	1.29	2.32	2.33	2.23
	1.36	3.08	2.4	3.1

	1.2	1.9	2.37	3.19
				2.34
Sample E avg	1.36	2.33	2.35	2.87
Sample E stdev	0.167	0.530	0.044	0.555
Sample F	28.81	39.63	34.25	46.99
	26.81	26.75	33.14	46.43
	33.15	42.32	29.17	40.17
	26.63	27.42	31.27	51.93
				47.85
Sample F avg	28.86	34.04	31.95	46.674
Sample F stdev	3.032	8.099	2.228	4.227

	Klinger	Teadit	Graftech	Flexitallic	avg
sample A	3.4	7.97	8.31	9.87	7.3875
sample B	23.19	44.41	39.79	51.71	39.775
sample C	10.55	17.98	16.71	20.31	16.3875
sample D	24.62	33.29	31.33	39.3	32.135
sample E	1.36	2.33	2.35	2.87	2.2275
sample F	28.86	34.04	31.95	46.67	35.38

Ranking	E,A,C,B,D,F	E,A,C,D,F,B	E,A,C,D,F,B	E,A,C,D,F,B	E,A,C,D,F,B

Conclusions:

A. All labs except one had the same ranking from high to low oxidation.

B. Klinger was consistently the low reading and Flexitallic the highest.

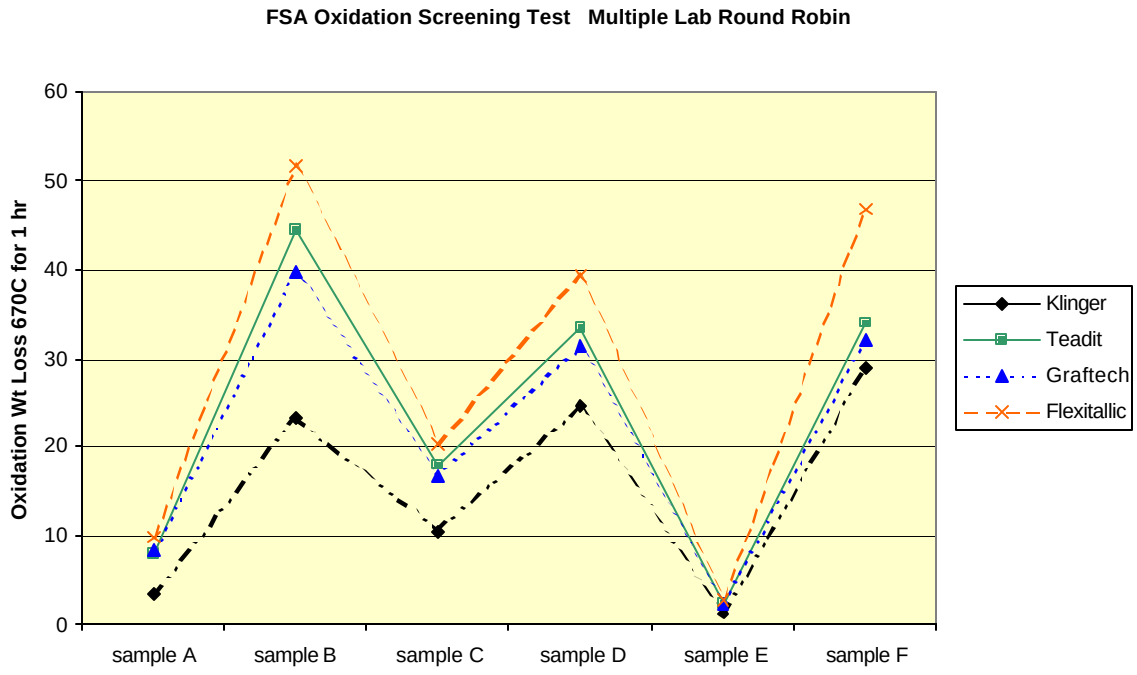
C. Graftech had the lowest within sample variation.

D. Teadit had the highest within sample variation

E. Thicker samples had less wt loss than same density and grade thinner samples

F. Lower density samples had higher wt loss than higher density for the same thk and grade

G. Graftech low within sample variation may have been because all pieces of one sample were run together.



Appendix 2

ASTM E691 Precision for Method A

Requirements for Determining Precision of Test Method: wt loss
Carefully examine the data for cases having no data for a particular material within a lab. Unbalanced studies cannot be properly calculated by this program. (All labs not having the same number of materials)

The number of laboratories, materials, and determinations in this study DOES NOT meet the minimum requirements for determining precision prescribed in ASTM Practice E691:

	THIS STUDY	ASTM E691 MINIMUM
Laboratories:	4	6
Materials	6	4
Determinations:	4	2

Precision Statement for Test Method: wt loss
Precision, characterized by repeatability, Sr, r, and reproducibility, SR, R has been determined for the materials to be:

Materials	Average	Sr	SR	r	R
Sample A	7.28	1.74	3.07	4.86	8.59
Sample B	39.15	9.39	14.44	26.29	40.42
Sample C	16.27	3.35	4.93	9.38	13.80
Sample D	31.90	7.96	8.94	22.30	25.04
Sample E	2.26	0.39	0.76	1.09	2.12
Sample F	35.30	5.07	8.85	14.21	24.79

This precision statement is provisional. Within five years, additional data will be obtained and processed which does meet the requirements of E691.