INTERNATIONAL

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Standard Test Method for Rubber Deterioration—Crack Growth[1]

This standard is issued under the fixed designation D813; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of crack growth of vulcanized rubber when subjected to repeated bending strain or flexing. It is particularly applicable to tests of synthetic rubber compounds which resist the initiation of cracking due to flexing when tested by Method B of Test Methods D430. Cracking initiated in these materials by small cuts or tears in service, may rapidly increase in size and progress to complete failure even though the material is extremely resistant to the original flexing-fatigue cracking. Because of this characteristic of synthetic compounds, particularly those of the SBR type, this test method in which the specimens are first artificially punctured in the flex area should be used in evaluating the fatigue-cracking properties of this class of material.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D430 Test Methods for Rubber Deterioration—Dynamic Fatigue

D1349 Practice for Rubber—Standard Conditions for Testing

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3767 Practice for Rubber—Measurement of Dimensions

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 A molded test specimen with a pierced groove is repeatedly flexed on a DeMattia type machine with the flexing (bending) axis parallel to the groove. The cut length is measured at frequent intervals to determine the cut growth rate. The cut is initiated by a specially shaped piercing tool.

4. Significance and Use

4.1 The test gives an estimate of the ability of a rubber vulcanizate to resist crack growth of a pierced specimen when subjected to bending or flexing.

4.2 No exact correlation between these test results and service is implied due to the varied nature of service conditions.

5. Interference

5.1 Presence of significant concentrations of ozone will affect test results. Care should be taken that ambient ozone concentrations do not exceed 1 pphm.

6. Apparatus

6.1 *DeMattia Flexing Machine*—The essential features of the apparatus, one design of which is shown in Fig. 1, are as follows:

6.1.1 The machine has an adjustable stationary head or member provided with suitable grips for holding one end of the test specimens in a fixed position and a similar reciprocating member for holding the other end of each of the specimens.

6.1.2 The reciprocating member is so mounted that its motion is straight in the direction of and in the same plane as

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[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.15 on Degradation Tests.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

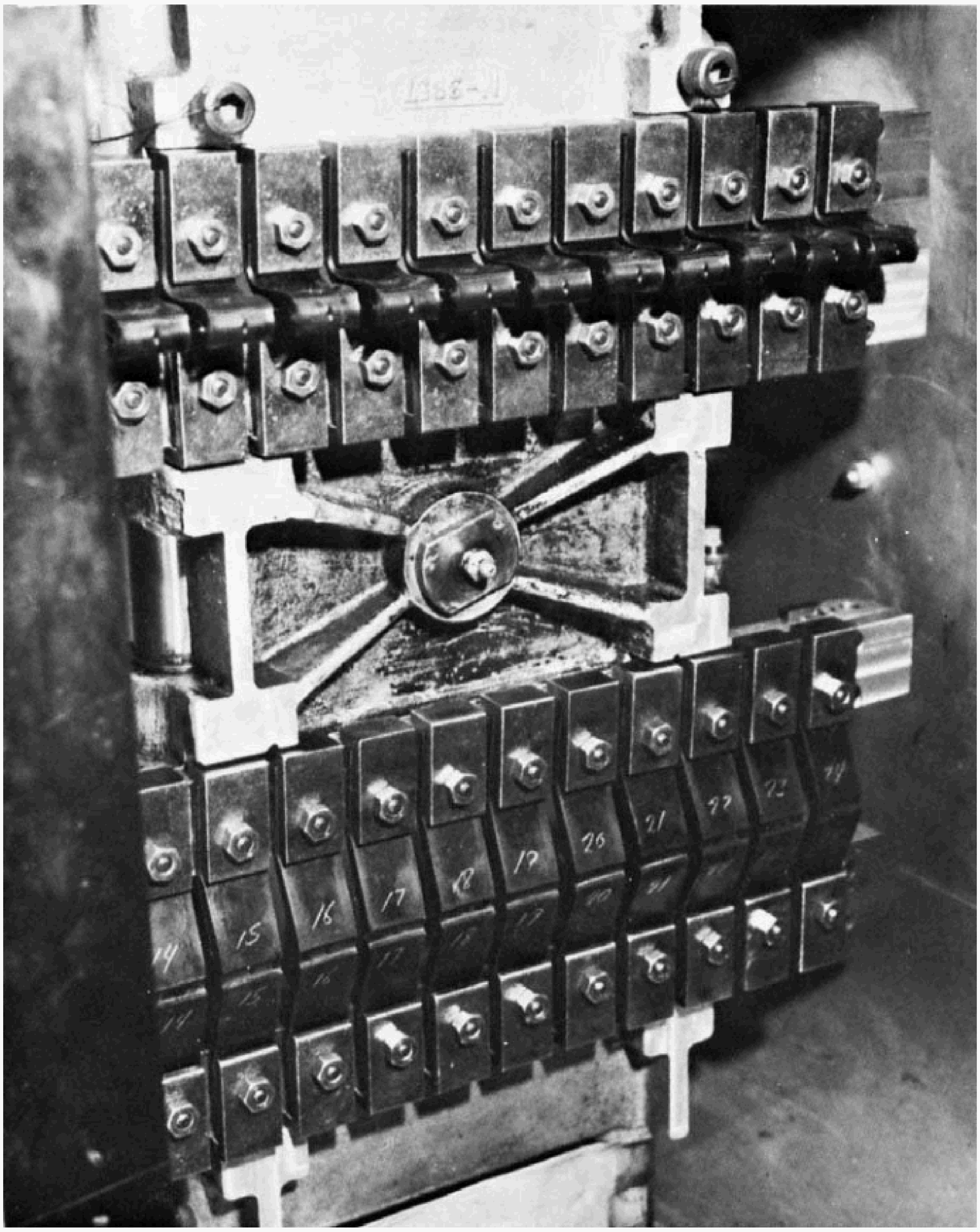


FIG. 1 DeMattia Tester with a Double Row of Specimens Mounted for Flex-Cracking Test

the center line between the grips. The travel of the moving member shall be adjustable and shall be obtained by means of a connecting rod and eccentric having a minimum length ratio of 10 to 1.

6.1.3 The eccentric shall be driven by a motor operating at constant speed under load and giving 5 ± 0.1 Hz (300 ± 10 cpm). Provision shall be made for a maximum travel of the moving grips of 100 mm (4 in.).

6.1.4 The machine may be designed so that all the specimens are mounted on a single bar and all are flexed at the same time. A double bar may also be used in which case those specimens mounted on one bar are being flexed while those on the other bar are being straightened. Such an arrangement improves the smoothness of operation.

6.2 *Mold*—A mold for curing individual test specimens is required, preferably of a multiple cavity design and having adequate overflow cavities. The cavity plate shall have a minimum thickness of about 20 mm (0.750 in.) and the cover plate a minimum thickness of 14.5 mm (0.575 in.). Refer to Practice D3182.

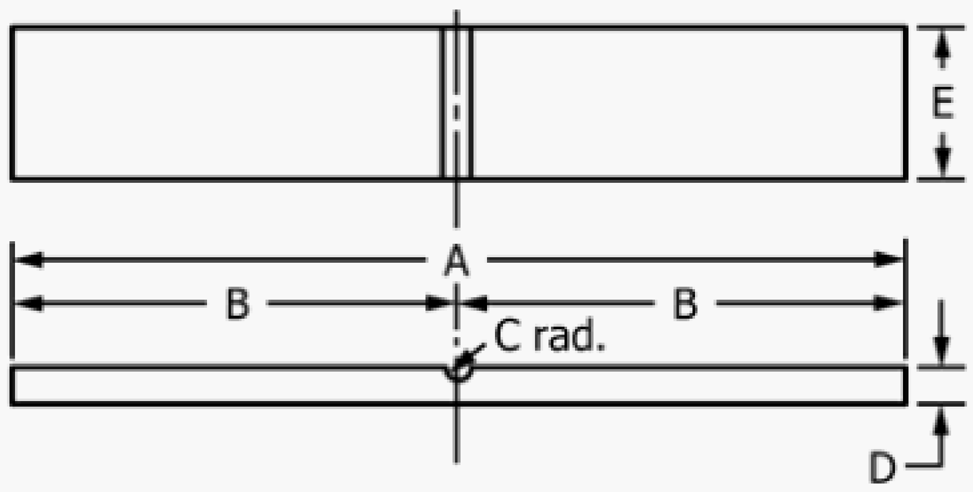
NOTE 1—The curing of wide samples from which specimens may be cut may be provided for by increasing the width of the cavity and maintaining all other dimensions.

6.3 *Measuring Scale* of suitable length, graduated in millimetres (or 0.01 in.) for measuring the length of cut growth.

6.4 *Micrometer,* to measure the thickness of the test specimen as specified in Practice D3767, Procedure A2.

7. Test Specimens

7.1 The test specimens shall consist of molded or cut strips, conforming to the shape and dimensions given in Fig. 2. They shall have a smooth surface and be free of surface irregularities and defects in the groove and adjacent area. The thickness shall



	A	B	C	D	E
mm	150	75	2.39 ± 0.03	6.35 ± 0.13	25
in.	6	3	0.094 ± 0.001	0.250 ± 0.005	1

FIG. 2 Test Specimen with Circular Groove

be measured in the area adjacent to the groove and results shall be compared only between specimens having a thickness of 6.4 ± 0.1 mm (0.250 ± 0.005 in.).

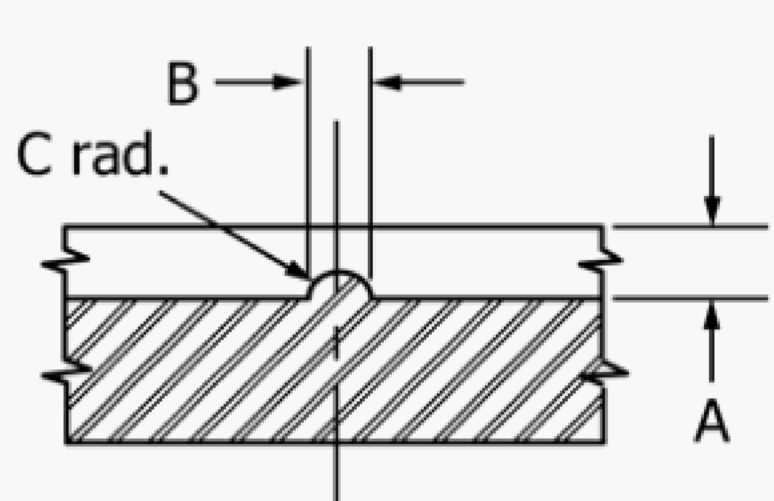
7.2 Test specimens shall be conditioned in accordance with Section 13.

NOTE 2—The thickness of a test specimen cured from a tread type compound consisting of 100 parts rubber and 50 parts of IRB Number 6 black, and the necessary curative materials and using the mold shown in Fig. 3, will conform to the thickness of 6.4 mm (0.250 in.) specified in Fig. 2. Preparation of the uncured specimens, on the basis of a constant volume plus an approximate overflow of 10 %, provides assurance of well-molded specimens of uniform thickness.

7.3 Compounds differing in stiffness may be expected to deviate from the thickness of 6.4 mm (0.250 in.) as obtained with the standard compound referred to above. Extreme cases resulting in thickness outside the tolerances may require additional study. It is recommended that a new average thickness be determined for such compounds, based on a minimum of ten specimens, and the allowable tolerance then be applied to this revised thickness measurement. The report of the data should then include such deviations from the control.

8. Number of Test Specimens

8.1 At least three specimens of each sample or compound shall be tested and the median value reported. It is desirable, when possible, to test a set of control specimens of known crack-growth resistance with each set of test specimens.

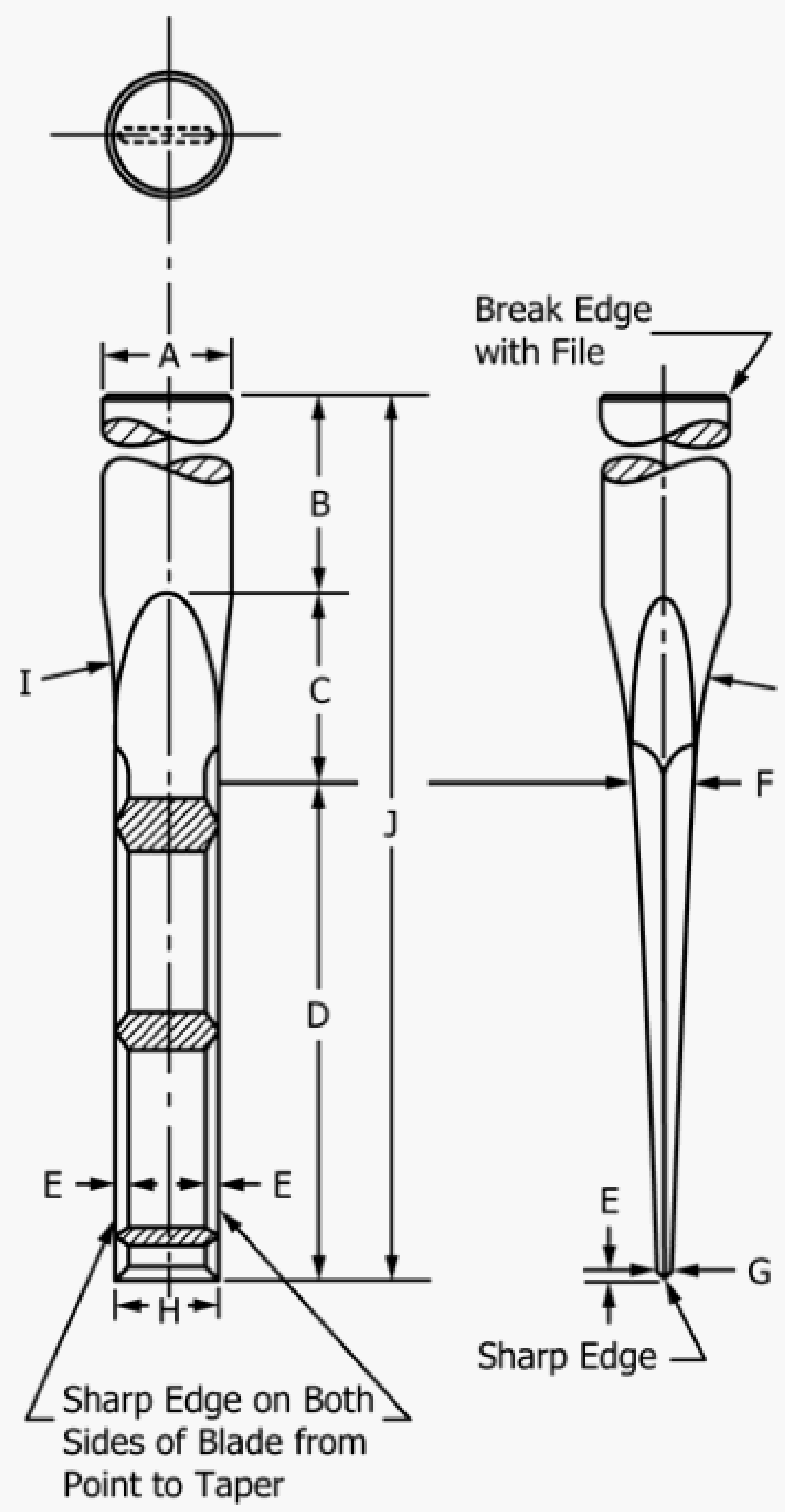


	A	B	C
mm	6.45 ± 0.03	4.78 ± 0.051	2.39 ± 0.03
in.	0.254 ± 0.001	0.188 ± 0.002	0.094 ± 0.001

FIG. 3 Test Specimen Mold

9. Preparation of Specimens

9.1 Each test specimen shall be prepared by piercing the bottom of the groove at a point equidistant from the sides. The tool shall be maintained perpendicular to both the traverse and longitudinal axes and the cut accomplished by a single insertion and withdrawal of the tool. The dimension H (of Fig. 4) of the tool shall be parallel and centered with the longitudinal axis of the groove. The leading cutting edge of the tool shall completely penetrate the specimen and project through the specimen a minimum of 3.2 mm (0.125 in.). Lubrication with water containing a suitable wetting agent may be used. The piercing tool shall be a spear-type instrument conforming to the dimensions given in Fig. 4. It is imperative that the tool is sharp and maintained to the correct dimensions, or test results will be affected. It is recommended that the piercing tool be held in a



	mm	(in.)
A	2.62	(0.103)
B	44.5 ± 04	(1.75 ± 0.015)
C	3.8 ± 0.4	(0.150 ± 0.015)
D	10.3 ± 0.1	(0.400 ± 0.005)
E	0.38 + 0.05 – 0.00	(0.015 + 0.002 – 0.000)
F	1.00 + 0.05 – 0.00	(0.041 + 0.002 – 0.000)
G	0.3 + 0.05 – 0.00	(0.015 + 0.002 – 0.000)
H	2.03 + 0.05 – 0.00	(0.080 + 0.002 – 0.000)
I	25 rad	(1.0 rad)
J	58.42	(2.300)

FIG. 4 Piercing Tool

suitable apparatus[3] that will maintain the tip in a perpendicular plane to the specimen and allow it to be pierced in one continuous motion.

10. Clamping Specimens in Machine

10.1 One end of the specimen shall be clamped in the stationary grip and the other in the movable grip, care being taken to see that the longitudinal axis of the specimen is parallel to the direction of motion.

10.2 The circular groove of the clamped specimens shall be located symmetrically, midway between the clamps. The specimens may be mounted on the machine most conveniently by holding them properly spaced in parallel positions in a special rack. The distance between the outer edges of the side bars of the rack shall be equal to the space between the jaws of the testing machine when positioned for holding the specimens without tension. The specimens can be mounted on the testing machine by bringing the jaws into contact with the mounting rack and tightening the clamps on the projecting ends of the specimens.

10.3 The specimens shall not be closer than 3 mm (0.125 in.) when mounted in the machine. The free length (not in tension) of the specimen between the clamps shall be 75.9 + 0.3 or – 0.0 mm (2.99 + 0.01 or – 0.00 in.).

10.4 The circular groove shall be so restrained that it will become the outer surface of the bent specimen.

11. Adjustment of Machine

11.1 The positions of the stationary and movable grips relative to each other and the length of the eccentric arm and connecting rod shall be adjusted so that during each stroke of the machine the grips approach each other to a distance of 19.0 ± 0.1 mm (0.750 ± 0.005 in.) and separate to a distance of 75.9 + 0.3 or – 0.0 mm (2.99 + 0.01 or – 0.00 in.).

11.2 Parallelism of the grips must be maintained at all times and can only be assured by periodic checks.

11.3 Machines operating within enclosures may be subject to conditions resulting in different rates of cracking for different positions in the grips. Correlation between all positions should be determined for each machine, using a standard control compound, and corrections made when necessary.

12. Procedure

12.1 After adjustments of the apparatus and specimens have been completed, start the machine and record the time. At the end of any period of operation, calculate the number of flexing cycles by multiplying the observed time in minutes by the machine rate of 5 Hz (300 cpm). This shall also be checked by means of a counter on the machine.

12.2 Since the rate of crack growth is important, take frequent readings early in the test. Stop the machine after 1000, 3000, and 5000 cycle periods, observe the specimens, and measure the length of the developed crack to the nearest 0.3 mm (0.01 in.) with an accurate scale, preferably metric.

12.3 For improved precision, make the observation with the aid of a low-powered reticulated magnifying glass while the grips are separated 65.0 mm (2.56 in.).

12.4 Continue the test with readings at regular intervals until a crack at least 12.5 mm (0.5 in.) in length is developed. Continuation to break may be desirable when testing aged specimens or when operating at elevated temperatures. The initial cut produced by the puncturing spear shown in Fig. 4 is 2 ± 0.1 mm (0.08 + 0.002 – 0.000 in.) in width.

13. Temperature of Test

13.1 The standard temperature for testing shall be 23 ± 2°C (73.4 ± 3.6°F). Specimens shall be conditioned for at least 12 h prior to testing. Controlled temperatures outside the standard range are acceptable and often desirable. Special note of such temperatures shall appear in the report.

NOTE 3—The standard test temperature herein specified is that prescribed for the Standard Laboratory Atmosphere in Practice D1349. Any changes or revisions hereafter in Practice D1349 relating to the standard test temperature shall be considered effective at once for this procedure.

14. Report

14.1 The molded dimensions A, C, D, and E (as specified in Fig. 2) of the test specimen shall be reported.

14.1.1 The dimensions shall be obtained in accordance with Practice D3767 except that the dimensions shall be obtained using a 2 mm spherical radius contact foot.

14.2 The crack growth data may be reported in any one of three ways, as follows:

14.2.1 As the number of cycles required to reach a specified crack length; for example, from 2 mm to 20 mm (0.080 to 0.80 in.);

14.2.2 As the average rate of crack growth over the entire test period;

14.2.3 As the rate of cracking in millimetres per kilocycle during a portion of the test, for example:

(*a*) 2 to 4 mm,
(*b*) 4 to 8 mm, or
(*c*) 8 to 12 mm.

15. Precision and Bias[4]

15.1 This precision and bias section has been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other statistical calculation details.

15.2 Two precision evaluation programs have been conducted for this test method.

15.2.1 Program 1 was conducted in 1985 with five different laboratories testing four different compounds on two separate days. The measured parameter is the flexlife in kilocycles to a crack length of 12.5 mm. A test result is defined as the mean (average) of the flexlife for two different test specimens.

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[3] The sole source of supply of the piercing tool and apparatus known to the committee at this time is CCSI, 221 Beaver St., Akron, OH 44304. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1041.

15.2.2 Program 2 was conducted in 1993 using a revised procedure as stipulated in the latest edition of this test method. In this program five different laboratories tested three compounds on two separate days. Two measured parameters were evaluated for this program:

(*1*) the flexlife in kilocycles to 300 % crack growth, and

(*2*) the flexlife (in kc) to 600 % crack growth.

A test result for either parameter is the mean (average) of three specimens.

15.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory programs as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

15.4 The results of the precision evaluation are given in Table 1 for Program 1 and in Table 2 for Program 2.

15.5 The precision may be expressed in the format of the following statements that use an appropriate value of *r*, *R*, (*r*), and (*R*) to be used in the decisions about the test results. The appropriate value is that value of *r* or *R*, associated with a mean level in Table 1 or Table 2, closest to the mean level under consideration at any given time for any test result for a material in routine testing operations. The general statements for repeatability and reproducibility apply to the parameters of both programs.

15.5.1 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 1 or Table 2. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

15.5.2 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 1 or Table 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

15.5.3 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results (in absolute units).

15.6 The precision results of both programs indicate that the value of *r* and *R* increase as the flexlife increases. Plots of *r* and *R* versus flexlife for both programs, in general, follow the same regression line. As commonly found in other precision evaluations, the dependence (slope) of *R* on flexlife is much greater than the same dependence for *r*. The values of (*r*) and (*R*), however, show a decreasing trend as flexlife increases.

15.7 Both of these precision evaluation programs had an inadequate number of laboratories for a satisfactory evaluation of the testing precision. One laboratory had to be eliminated from the Part B precision evaluation of Table 2 because of excessive deviation from the other four. This further reduces the confidence of the final results for the evaluated precision.

15.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias cannot therefore be determined.

16. Keywords

16.1 crack growth; DeMattia (De Mattia) flexing machine; dynamic fatigue; flex fatigue; flexing; flexing fatigue; rubber products

TABLE 1 Type 1 Precision Results (Program 1)[A]

NOTE 1—Flexlife in kilocycles to 12.5 mm crack length.

Material	Average[B]	Within Laboratories			Between Laboratories		
		S_r	*r*	(*r*)	S_R	*R*	(*R*)
Material 1	3.78	0.355	1.00	26.6	2.45	6.93	183.4
Material 2	19.3	1.35	3.84	19.8	5.63	15.9	82.2
Material 3	45.8	2.41	6.83	14.9	11.8	33.6	73.2
Material 4	48.7	1.78	4.99	10.2	14.8	42.0	86.3
Pooled Values[C]	29.4	1.65	4.67	15.8	9.99	28.2	96.1

[A] S_r = repeatability, standard deviation,
r = repeatability (2.83 × the square root of the repeatability variance),
(*r*) = repeatability (as percentage of material average),
S_R = reproducibility, standard deviation,
R = reproducibility (2.83 × the square root of the reproducibility variance), and
(*R*) = reproducibility (as percentage of material average).
[B] Values expressed in kilocycles.
[C] No values omitted.

TABLE 2 Type 1 Precision Results (Program 2)[A]

Part A—Flexlife in Kilocycles to 300 % Crack Growth:[B]							
Material	Mean[C]	Within Laboratories			Between Laboratories		
		S_r	r	(r)	S_R	R	(R)
CPD A	6.7	1.14	3.19	47.6	2.14	6.01	89.7
CPD B	9.0	1.41	3.96	44.0	4.56	12.80	142.0
CPD C	19.1	1.38	3.86	20.2	6.41	18.00	94.0

Part B—Flexlife in Kilocycles to 600 % Crack Growth:[D]							
Material	Mean[C]	Within Laboratories			Between Laboratories		
		Sr	r	(r)	S_R	R	(R)
CPD A	12.6	0.94	2.62	20.8	2.48	6.95	55.2
CPD B	17.0	1.50	4.20	24.7	6.88	19.30	114.0
CPD C	33.5	3.81	10.70	31.9	7.72	21.60	64.5

[A] S_r = repeatability, standard deviation, in measurement units,
r = repeatability = 2.83 × repeatability standard deviation,
(r) = repeatability, as percentage of material mean (average) value,
S_R = reproducibility standard deviation, in measurement units,
R = reproducibility 2.83 × reproducibility standard deviation, and
(R) = reproducibility, as percentage of material mean (average) value.
[B] $p = 5$, $q = 3$, and $n = 2$.
[C] Mean in Kilocycles.
[D] $p = 4$, $q = 3$, and $n = 2$.