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School of Engineering

FINAL REPORT

"REFINED TEST METHODS IN EVALUATING BRITTLE
MATERIAL FRACTURE STRENGTHS "

Contract No. N00019-79-C-0574

December 2, 1982

"Approved for Public Release--Distribution Unlimited"

William J. Craft
Principal Investigator
Professor
Mechanical Engineering
(919) 379-7549

George J. Filatovs
Co-Investigator
Professor
Mechanical Engineering
(919) 379-7620

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single variable, ϕ , the angle on the fracture surface from point of fracture to the $\hat{n}=(1,1,1)$ direction. (2) the creation of a simple test procedure and specimen design to find a variety of points on the fracture surface, and (3) the design of experiments to employ this testing system. In the first contract, both alumina and mullite specimens were tested in four-point loading and these results were reduced to a Weibull distribution by determination of the two-parameter coefficients.

In this contract, a torsion test method and apparatus were designed and implemented. Both alumina and mullite specimens, the same geometry as was used in the four-point bend test of the previous contract were loaded in torsion to failure at another angle on the fracture surface, or at $\phi=90^\circ$ to the $\hat{n}=(1,1,1)$ direction. The results are given for alumina and mullite tests.

Finally, a general test utilizing 'thick' disks of each material is described. The literature seems to lack a simple reliable disk test for which a simple stress distribution is readily available. The test produces an axially symmetric state of stress but one that embodies a variety of orientations to the $\hat{n}=(1,1,1)$ direction. The use of a hydraulic ram gives rise to an easily predictable stress field. The expected Weibullian scatter was detected in both alumina and mullite specimens which were tested in both as-cast and ground conditions. No problems attributable to lack of flatness in these low-strain-to-failure materials were detected in as-cast samples. The alumina samples tended not to show fracture propagation throughout the thickness while the mullite samples did demonstrate this behavior. Because it was sometimes difficult to determine precisely what pressure produced incipient cracking in alumina, a differential current detector employing a conductive brittle coating has been proposed--the coating brittleness being chosen to match the specimen strain-to-failure.



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Abstract

In this report entitled, "Refined Test Methods In Evaluating Brittle Material Fracture Strengths," Contract No. N00019-79-C-0574, is a follow-up of the work done under NAVAIR No. N00019-78-C-0520, entitled, "Fracture Prediction in Brittle Materials.".

In the former contract, an analytical system was devised to generate fracture data based on: (1) conversion of triaxial fracture data to a single variable, ϕ , the angle on the fracture surface from point of fracture to the $\hat{n}=(1,1,1)$ direction, (2) the creation of a simple test procedure and specimen design to find a variety of points on the fracture surface, and (3) the design of experiments to employ this testing system. In the first contract, both alumina and mullite specimens were tested in four-point loading and these results were reduced to a Weibull distribution by determination of the two-parameter coefficients.

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ACKNOWLEDGEMENT

This report represents the third in a series of fracture experiments on Mullite and Alumina tube specimens as supported by the Naval Air Systems Command. This set of investigations has led to refined bending and torsion tests and to a hydraulic disk test not previously noted. There are ongoing plans to present these findings in the literature. The authors gratefully acknowledge the support of Navair in making this research possible.

TABLE OF CONTENTS

ABSTRACT	Page 4
TABLE OF CONTENTS	7
TABLE OF GRAPHS	8
TABLE OF FIGURES	9
TABLE OF TABLES	11
<u>Chapters</u>	
I. THEORY OF THE TORSION TEST	12
II. DESCRIPTION OF THE TORSION TEST	16
III. RESULTS OF THE TORSION TEST	24
IV. DESCRIPTION OF THE HYDRAULIC TEST	39
V. RESULTS OF THE HYDRAULIC TEST	48
VI. CONCLUSIONS	60
APPENDIX I COMPUTER PROGRAMS	63
A. TORSIO (AUTO.FOR)	64
B. AGAR (AGARWA.FOR)	66
APPENDIX II DISTRIBUTION LIST	67

TABLE OF GRAPHS

GRAPHS	PAGE
3.1 Fracture Probability of Unit Volume of Alumina 998	29
3.2 Fracture Probability of Unit Volume of Mullite MV33	34

TABLE OF FIGURES

FIGURES	PAGE
1.1 Biaxial Stress Envelop for Circular Cross-Section Tube Subjected to Pure Torsion.	12
1.2 Mohrs Circle in Pure Torsion	12
1.3 Pure Torsion for a Circular Cross-Section Tube	13
2.1 Universal Testing Facility	17
2.2 Test Fixture Within Testing Machine	18
2.3 Torsion Testing Fixture	19
2.4 Torsion Testing Fixture-Dimensions	20
2.5 Detail of Torsion Specimen End Grips	21
3.1 Torsion Test Device Immediately Following Test	31
3.2 Failed Alumina Torsion Specimen AT60	32
3.3 Failed Alumina Torsion Specimen AT62	32
3.4 Mullite Failure in Torsion, Specimen MT87	36
3.5 Mullite Failure in Torsion, Specimen MT88	37
3.6 Mullite Failure in Torsion, Specimen MT89	37
3.7 Mullite Failure in Torsion, Specimen MT90	38
4.1 Alumina and Mullite Disk Specimens and Loading Conditions	40
4.2 View of Hydraulic Test Assembly	42
4.3 Specimen and Gasket-Both Sides	43
4.4 Specimen Ready to be Mounted in Assembly	43
4.5 Disk Specimen Fixture	44
4.6 Test Fixture for Disk Specimens	45
4.7 Schematic of Hydraulic System-Capacity 10,000 p.s.i.	46
4.8 Specimen Coating Scheme and Bridge Crack Detector.	47

TABLES OF FIGURES (cont.)

FIGURE	PAGE
5.1a Hydraulic Fracture of Specimen A11-Outer Surface (2.5X)	56
5.1b Hydraulic Fracture of Specimen A11P-Inner Surface (3X)	56
5.2a Hydraulic Fracture of Specimen A13-Outer Surface (2.5X)	57
5.2b Hydraulic Fracture of Specimen A13P-Inner Surface (3X)	57
5.3a Hydraulic Fracture of Specimen M108-Outer Surface (2.5X)	58
5.3b Hydraulic Fracture of Specimen M108P-Inner Surface (3X)	58
5.4a Hydraulic Fracture of Specimen M115-Outer Surface (2.5X)	59
5.4b Hydraulic Fracture of Specimen M115P-Inner Surface (3X)	59

TABLE OF TABLES

TABLE	Page
2.1 Torsion Specimen Dimension - Inches	23
3.1 Torsion Experimental Results - Alumina 998	26
3.2 Data on All other Alumina Tests	27
3.3 Assumed and Determined Probabilites of Fracture for Alumina 998 Original Test Data	28
3.4 Torsion Experimental Results - Mullite MV33	33
3.5 Assumed and Determined Probabalities of Fracture for Mullite MV33 Original Test Data	35
4.1 Fracture Surface Angles for Typical Types of Tests	39
4.2 Disk Specimen Dimensions - Means and Standard Deviations	40
5.1a Alumina Disk (As Cast) Pressure Tests	49
5.1b Alumina Disk (As Cast) Pressure Tests	49
5.2a Alumina Disk (Ground Finish) Pressure Tests	50
5.2b Alumina Disk (Ground Finish) Pressure Tests	50
5.3a Mullite Disk (As Cast) Pressure Tests	52
5.3b Mullite Disk (As Cast) Pressure Tests	52
5.4 Mullite Disk (Ground Finish) Pressure Tests	53

Chapter 1

THEORY OF THE TORSION TEST

A circular prism, rod, or tube can be subjected to a state of pure torsion along its gage length. Fig. 1.1.

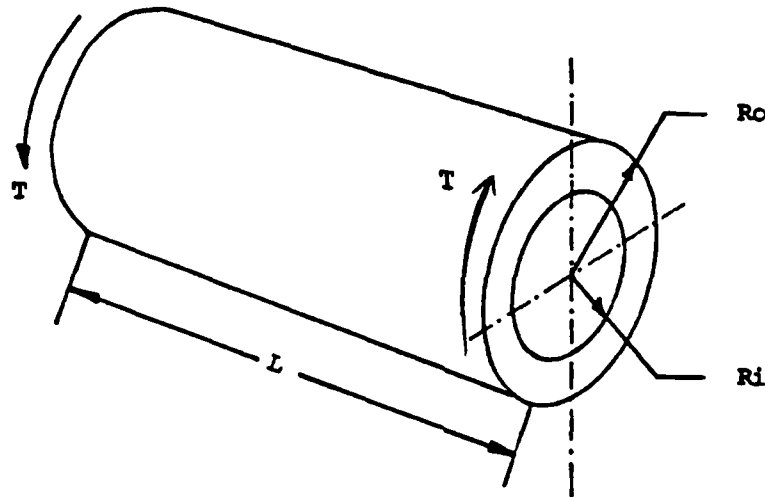


Figure 1.1 Biaxial Stress Envelope for Circular Cross-Section Tube Subjected to Pure Torsion

The Biaxial stress state depicted by Mohr's circle. Fig. 1.2. is applicable to every point of the specimen if edge effects are neglected. As demonstrated by the results in Chapter 3, the edge effects did not contribute to testing error in a significant way. However, almost some stress concentration is to be expected in any experiment and attempts should be devised to minimize it as with the special end caps and adhesives described in Chapter 2.

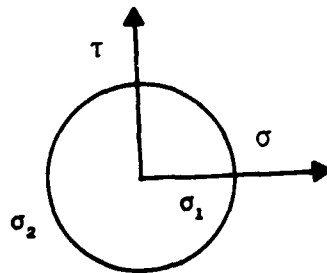


Figure 1.2 Mohr's Circle in Pure Torsion

The probability of failure is:

$$F(T) = 1 - \exp(-Bn)$$

1.1

The risk of fracture or rupture can be expressed easily in integral form since the radial volume element and stress level are simply related. Further, a scalar variable, τ , that is one possible contraction of the stress state torsion is used in the integral to obtain:

$$Bn = Lc \iint \left(\frac{T r}{J} - \sigma_u \right)^m r d\theta dr$$

where $c = \frac{1}{V_{un}(\sigma_o)^m}$, $\tau = \frac{Tr}{J}$ and $L =$ the gage length.

1.2

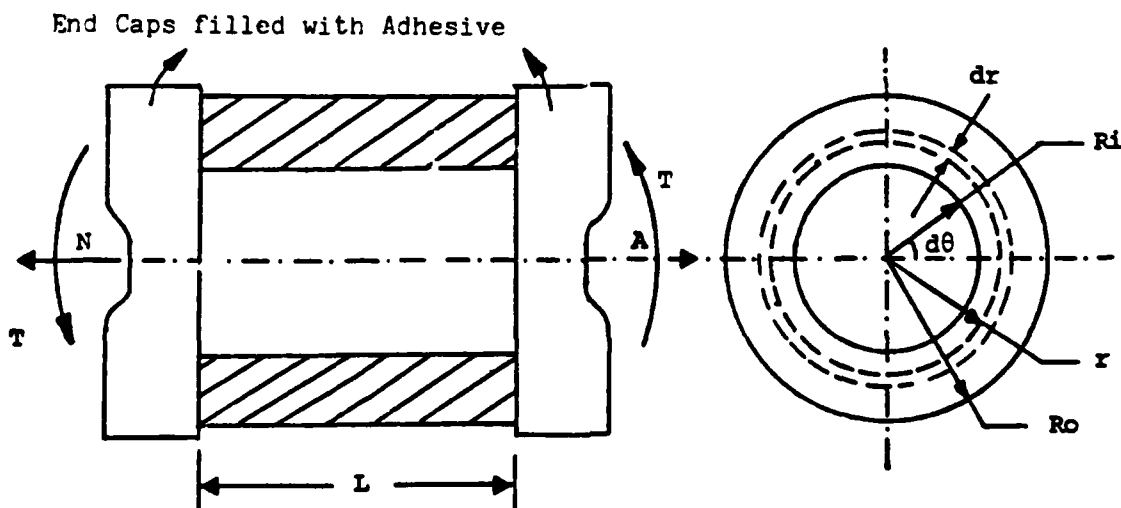


Fig. 1.3 Pure Torsion for a Circular Cross-Section Tube

Using the same approach as in [1] rods and tubes are separately considered:

- (1) Rod ($R_i = 0$) : Two cases occur;

(a) $0 \leq R_{th} < R_o$; where $R_{th} = \frac{\sigma_u J}{T}$, and B_n becomes

$$B_n = \frac{2\pi Lc}{(m+1)\left(\frac{T}{J}\right)} \left[r \left(\frac{T}{J} r - \sigma_u \right)^{m+1} - \frac{\left(\frac{T}{J} r - \sigma_u \right)^{m+2}}{\left(\frac{T}{J} \right) (m+2)} \right]_{R_{th}}^{R_o} \quad 1.3$$

(b) $R_o \leq R_{th}$; In this case;

$$B_n = 0. \quad 1.4$$

(2) Tube ($R_i \neq 0$): Three cases occur;

(a) $R_o > R_{th} > R_i$

B_n is expressed by Eq. (1.3)

(b) $0 \leq R_{th} \leq R_i$

B_n is expressed by Eq. (1.3) except the lower limit of integration is replaced by R_i .

(c) $R_{th} > R_o$; in this case;

$$B_n = 0.,$$

Equation (1.3) has the same form as the case of the uniaxial tension field and can be correlated with either the three-parameter Weibull distribution or the two-parameter distribution in an iterative programming scheme, similar to UNIAX.FOR. [1]

Any of the scalars, the shear stress, τ ; the effective tensile principal stress, $\sigma_1 = T r/J$; or the 'biaxial intensity', σ , can be used in Eq. (1.3) for the study. This intensity might be developed

in several ways including the correlation of test results with

1) the most tensile component of the stress tensor or 2) the strain energy of distortion [2].

In either case, each element is considered to be under a torsional stress, where $\tau = Tr/J$ is assumed, and the risk of rupture for the whole specimen is obtained by integrating the risk of rupture of each infinitesimal element over the gage volume of the specimen. Thus, 1.3 applies.

Under this pure torsion, the stress field $\tau_{rz} = Tr/J$ results from which the principal direction stresses are:

$$\sigma_1 = \tau_{rz} ; \quad \sigma_2 = -\tau_{rz} \quad 1.5$$

Normally the compressive component, σ_2 , would be neglected and a uniaxial stress state, σ_1 , would be assumed. In this case $\sigma = \sigma_1 = Tr/J$. If distortion energy is employed to define σ_1 then under pure torsion:

$$U^1 = \frac{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2}{12G}$$

or

$$U^1 = \frac{\sigma_1^2}{2G} \quad 1.6$$

For this study, compression was ignored and $\sigma_1 = \tau = Tr/J$ led to 1.3.

Chapter 2

DESCRIPTION OF THE TORSION TEST

The torsion test method required the creation of a torsion test loading system designed to operate in conjunction with a standing universal testing machine, Figure 2.1. The actual torsion test fixture designed to accept a specimen between two universal joints, Figure 2.2, was also designed to eliminate all loads except pure torsion.

The pictorial view, Figure 2.3, demonstrates the current configuration in general while the component dimensioned drawings provided detail, Figure 2.4, 5. The experiment was first conceived as one that did not require a locking bar behind each collet. It was found, however, that the experiment was unsuccessful because the hardened collets or chucks slipped before fracture stress was obtained. Naturally, these early tests were conducted on alumina, the stronger of the two materials. The original sleeves, Figure 1.3, were thus machined to allow for a slot and the experiment repeated.

The next problem was one of adhesives. A rough calculation indicated that an elastomer adhesive would need to withstand up to 3000 p.s.i. in shear to sustain a load sufficiently high to fracture alumina. After consultations with 3M adhesive experts a film adhesive -- 3M AF-126-2 -- was selected. Surfaces of both the ceramic and metal sleeve were prepared carefully to permit good bonding. The adhesive was cured as specified and several specimens were tested.

The results of these tests were disappointing and all joints failed at no greater than 25% of the expected alumina fracture torque. Perhaps this problem was caused by (1) use of a 'bad' or degraded batch of adhesive

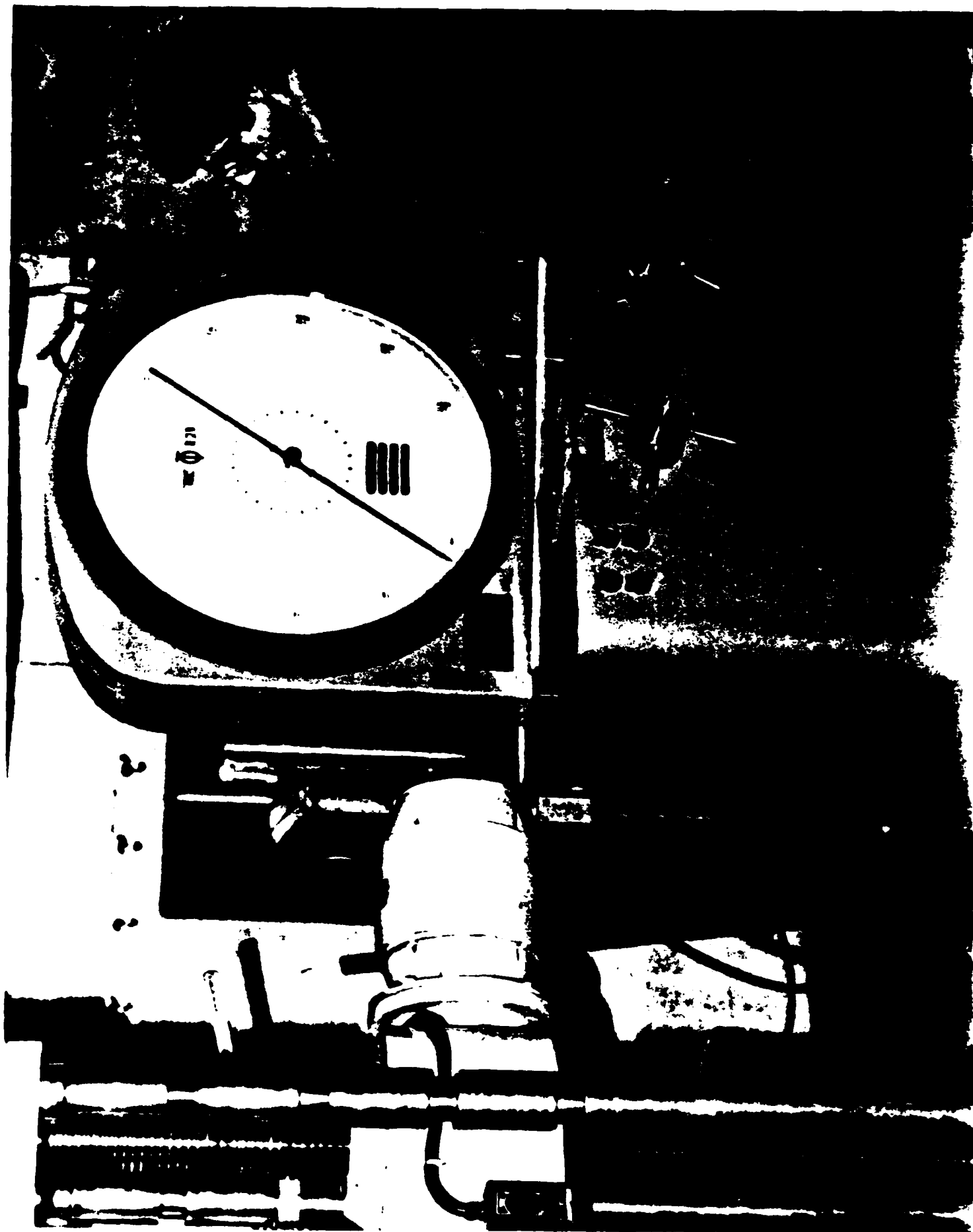


Figure 2.1. Universal Testing Facility

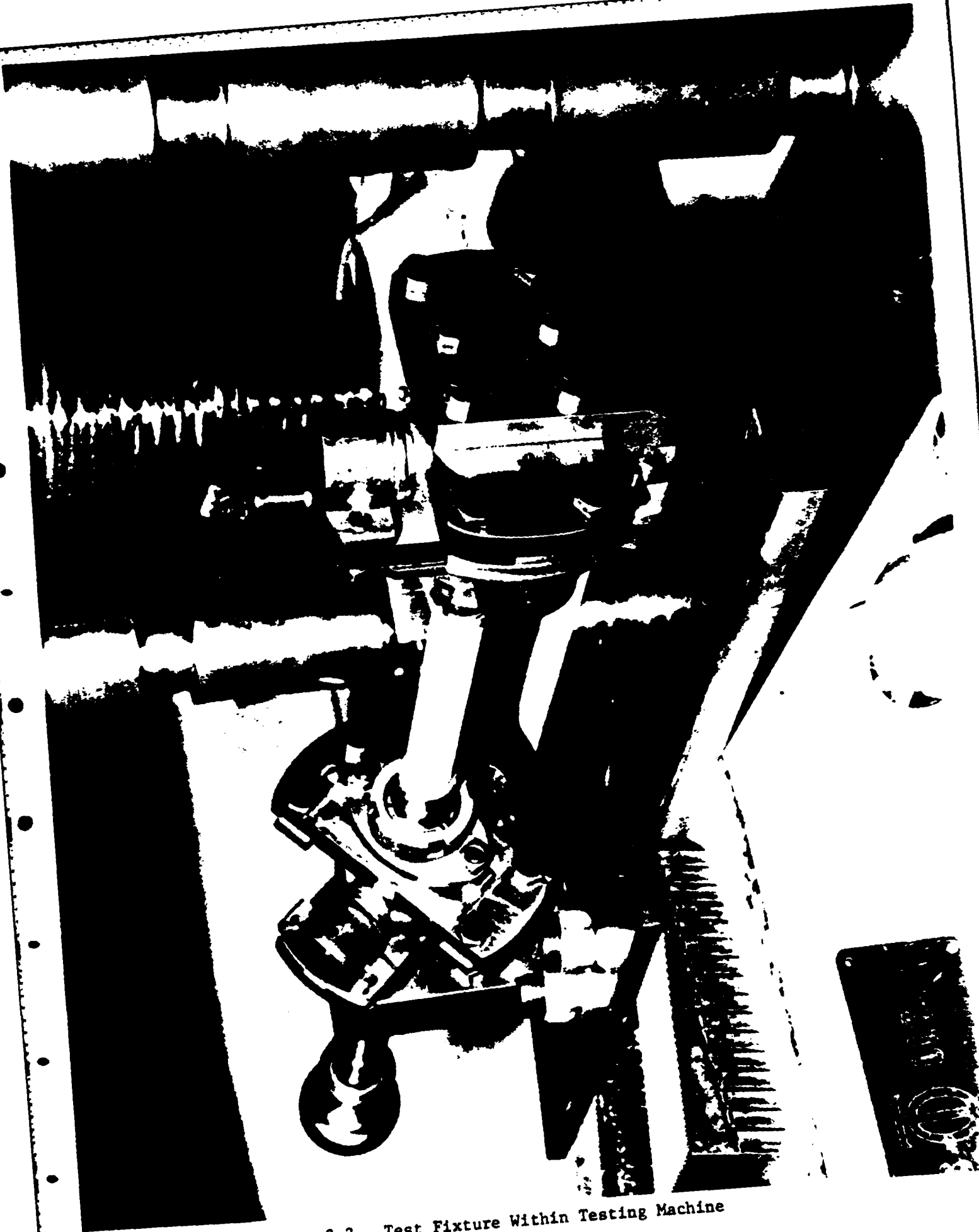


Figure 2-2. Test Fixture Within Testing Machine

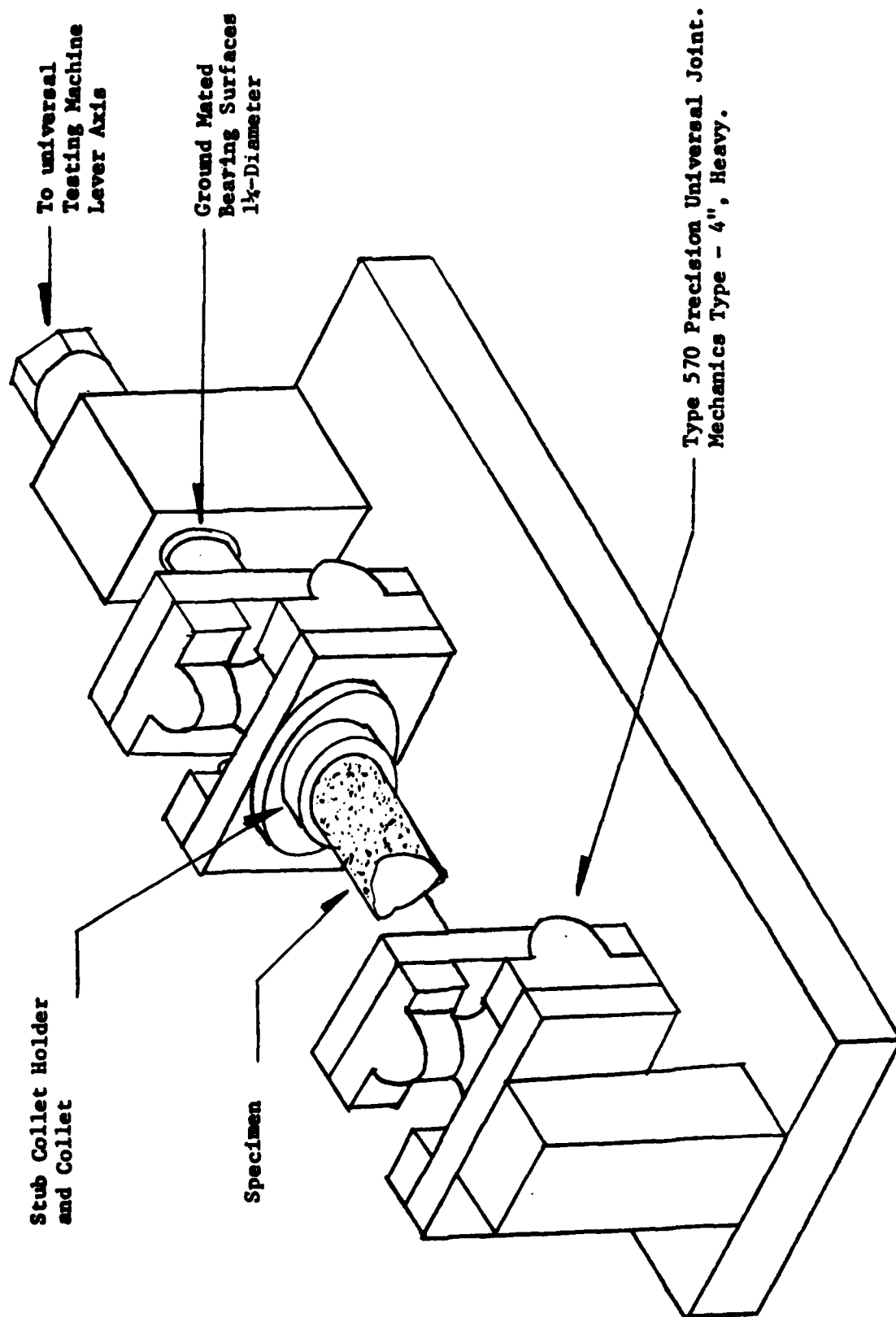


Figure 2.3 Torsion Testing Fixture

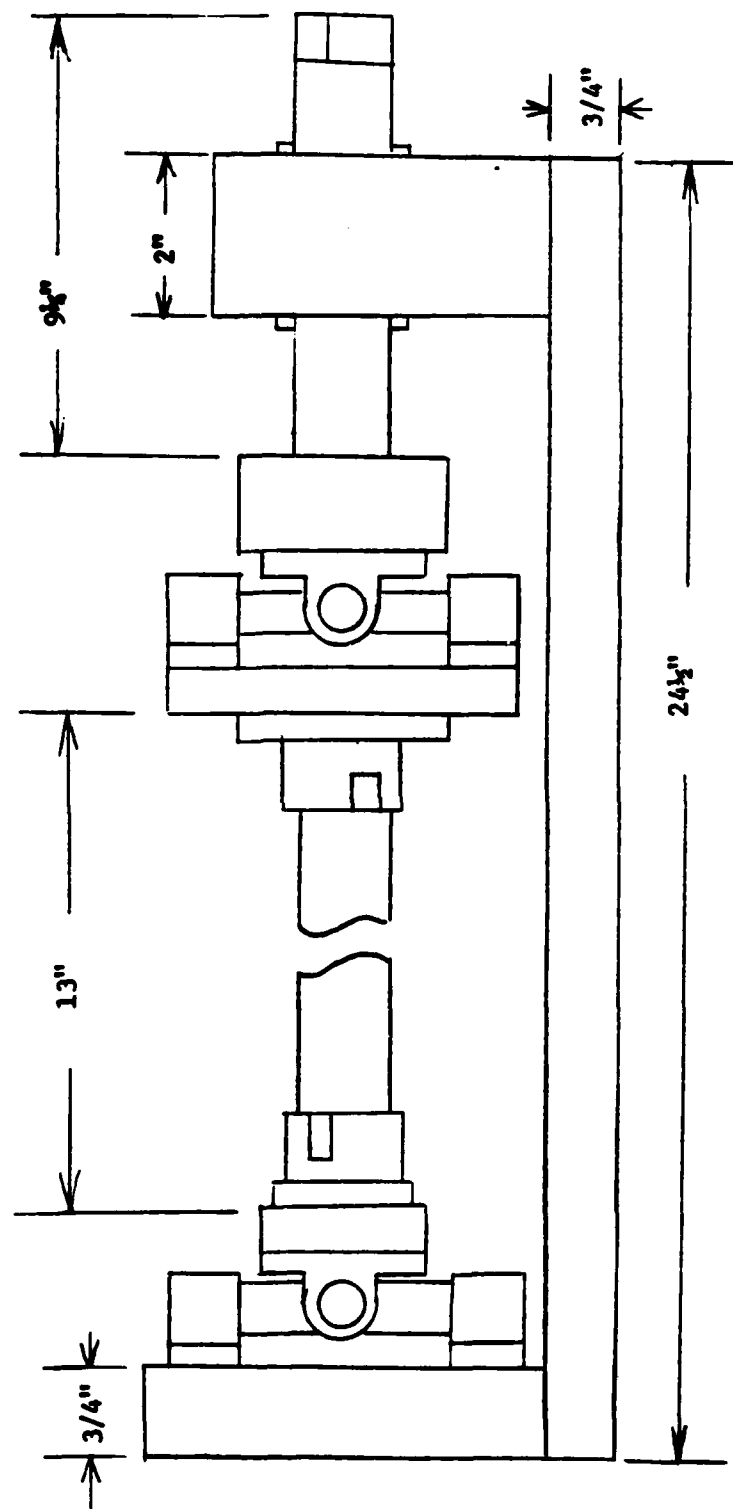


Figure 2.4 Torsion Testing Fixture-Dimensions

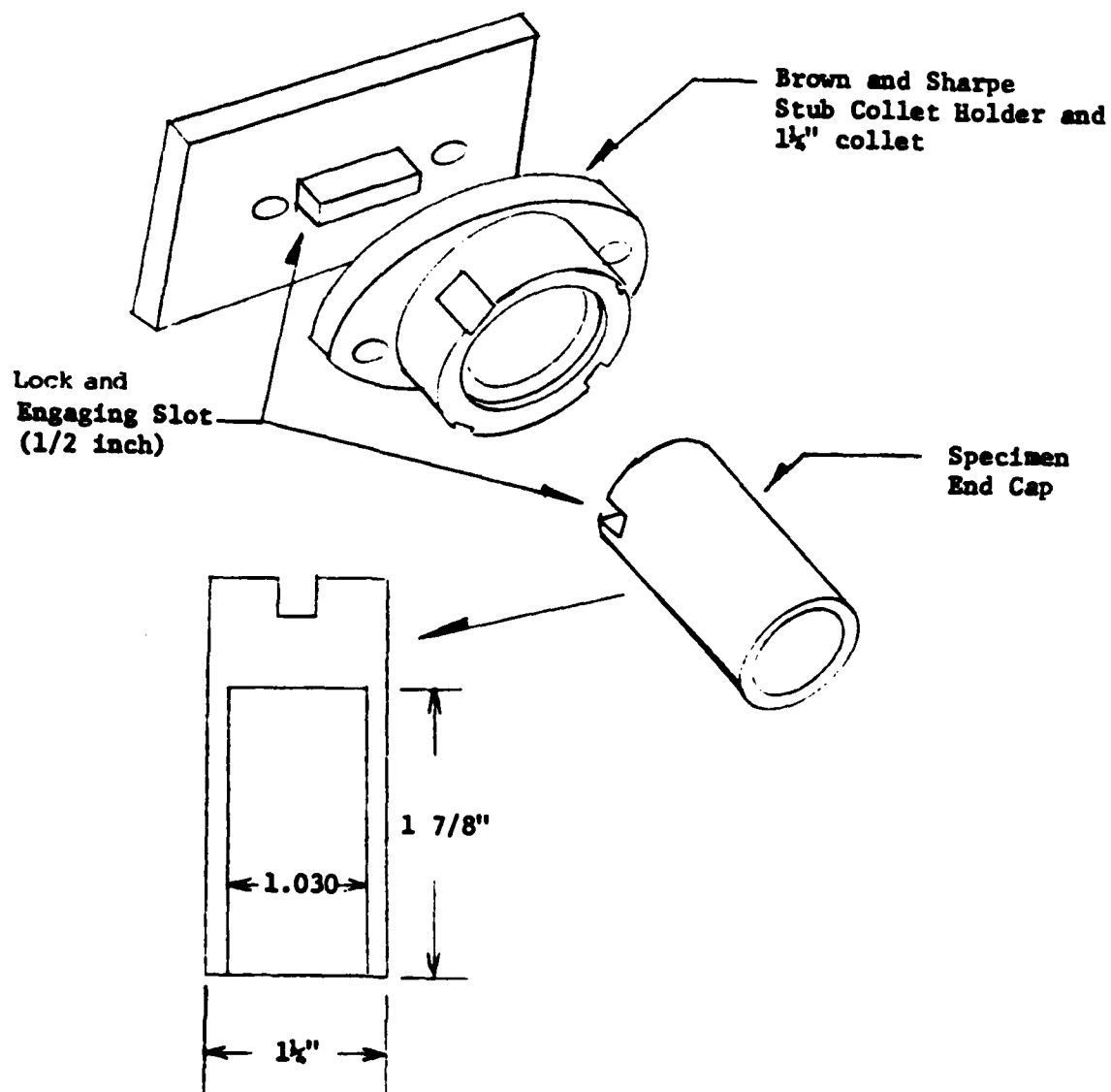


Figure 2.5 Detail of Torsion Specimen End Grips

as affected by transportation, or (2) by the lack of sufficient interference pressure during the curing process. The only available pressure during curing was obtained through the interference fit between the sleeve and alumina specimens largely due to thermal expansion of the adhesive.

The second adhesive tried was 3M Structural Epoxy, Scotch-Weld Brand, No. 2216 B/A. This gray two-part epoxy had less advertised strength, but it proved far more suitable even though a room temperature cure was used. Only in a very few cases did this adhesive fail. In all cases the epoxy was burned out after each test and a new specimen was bonded again. The burnout of end caps meant that fewer caps needed to be machined, thus, reducing expense considerably. The epoxy was also used to 'pot' the ceramic disks in place on copper gaskets in the disk tests as described in chapters 4 and 5.

The ceramic specimens used were made by McDanel Refractory of Beaver Falls, Pennsylvania. Their high density 988-Alumina and MV33-Mullite ceramic tubes were the specimens for both the bending tests of [1] and these torsion tests. The same materials and batches were also used for the disk tests of this report. For the disks, the method of casting was of necessity different. The tubes were extruded. Table 2.1 describes tube geometries.

	Alumina (998)	Mullite (MV33)
Mean and SD of Maximum OD	1.0010 ₊ .0021	1.0290 ₊ .0065
Mean and SD of Director <u>1</u> to Max. OD Direction	.9985 ₊ .0027	1.0236 ₊ .0082
Mean and SD of Minimum ID	.6156 ₊ .0203	.7276 ₊ .0087
Mean and SD of Direction <u>1</u> to Min. ID Direction	.6209 ₊ .0234	.7367 ₊ .0074
Estimated Outer Radius	.4999	.5132
Estimated Inner Radius	.3091	.3661
Test Length	7.3	7.3
Overall Length	11.0	11.0
Number of Samples Used for These Statistics	50	50

TABLE 2.1 TORSION SPECIMEN DIMENSIONS - INCHES

Chapter 3

RESULTS OF THE TORSION TESTS

Alumina 998 Results

Results for the torsion test of alumina are listed below in Table 3-1. It should be noted that most entries were considered successfully tested. Entries ranked 21 and 22 were used as 'good tests' even though they did not fracture; their epoxy bond failed first, but had a very high stress level. For that reason and because the high end of the curve might be more accurately depicted, it was decided to include them. Each of these other specimens actually fractured in torsion and their end caps were removed by heating the failed specimen end cap with an acetylene torch to burn out the adhesive. The cap was cleaned in a solvent, sandpapered, then reused. Thus, six sets of caps were required so that three were bonded to specimens being tested while three more were being cleaned on any work period. At two points, the mild, cold-rolled steel caps became deformed in the slot through plastic deformation so that new caps had to be manufactured, c.f., Fig. 2.5.

Table 3.2 lists all tests on alumina that were rejected. In the great majority of such tests the specimens slipped in the grips indicating bond failure. Two specimens that originally slipped at low levels were retested with new adhesive and were successfully tested to failure. Most tests in which bond failure occurred were never retested successfully. This is thought to be because those torsion specimens were found to be slightly undersized, thus, contributing to a thick bond of reduced sheer strength.

It was decided that any test which did not demonstrate failure through the specimen center would be rejected. This is because the most accurate representation of the Weibull Parameters was desired. Flaws near the attachments might propagate to failure by bond-induced stress concentrations, thus invalidation the results. No such examples occurred; however, it

should be pointed out that most specimens exhibited a spiral shaped crack through the grip attached to the torsion arm on the testing machine - the active grip. This phenomenon was thought to result from the release of strain energy in the heavy assembly through springback. All such specimens failed in the gage - length end active grip.

The data were analyzed as a two-parameter Weibull distribution through the use of an iterative programming code in Appendix I. AUTO.FOR and AGARWA.FOR. The results for the alumina samples are listed below:

Two-Parameter Family for Alumina 998

$$\begin{aligned} c &= 0.617E-28 \text{ (in.}^{2m} \text{ lbs.}^{-m}\text{)}: & 2.708E-52 \text{ (M}^{2m} \text{ N}^{-m}\text{)} \\ m &= 6.085 \text{ (dim less)}: & 6.085 \text{ (dim less)} \\ \sigma_u &= 0 \text{ (psi)}: & 0 \text{ (MPa)} \\ \sigma_o &= 4.325 E+4 \text{ (psi)}: & 298.2 \text{ (MPa)} \end{aligned}$$

Residual error from curve fitting, $Re = .3340E-01$

where $Re = \sum_{i=1}^N W_i * (P_i - \tilde{P}_i)^2$

N = Number of entries

P_i = Probability of Fracture used as input

$\tilde{P}_i$ = Derived probability of Fracture. Table 3.3

W_i = Weight of each Data Entry.

Because probabilities of fracture vs. load, Table 3.1, were generated by the rank method, the use of the three parameter distribution was not used.

Table 3 1. Torsion Experimental Results - Alumina 998
(Data from 24 good tests)

Specimen Number	Rank	Torque at Failure (Foot Pounds)	$F(\bar{T}) = 1/(N+1)$ (N=22)	Remarks
AT11	1	303.33	.0435	(Two specimens fractured at a load of 525)
AT62	2	373.75	.0870	
AT51	3	417.92	.1304	
AT20	4	429.74	.1739	
AT07	5	435.52	.2174	
AT21	6	460.83	.2609	
AT10	7	478.33	.3044	
AT06	8	517.92	.3478	
AT09	9	525.00	.3913	
AT12	(9)*	525.00	.3913	
AT25	10	530.83	.4348	Did not break -- adhesive failure Did not break -- adhesive failure
AT15	11	549.22	.4783	
AT61	12	552.50	.5217	
AT18	13	558.66	.5652	
AT60	14	575.00	.6087	
AT16	15	580.73	.6522	
AT64	16	585.00	.6957	
AT08	17	595.00	.7391	
AT56	18	600.00	.7826	
AT13	19	617.50	.8261	
AT14	20	659.17	.8696	
AT24	21	759.22**	.9130	
AT23	22	800.42**	.9565	

* Taken as same rank with twice normal weight

** These tests were included even though there was bond failure rather than fracture because the load at failure was considerably above the mean of other tests.

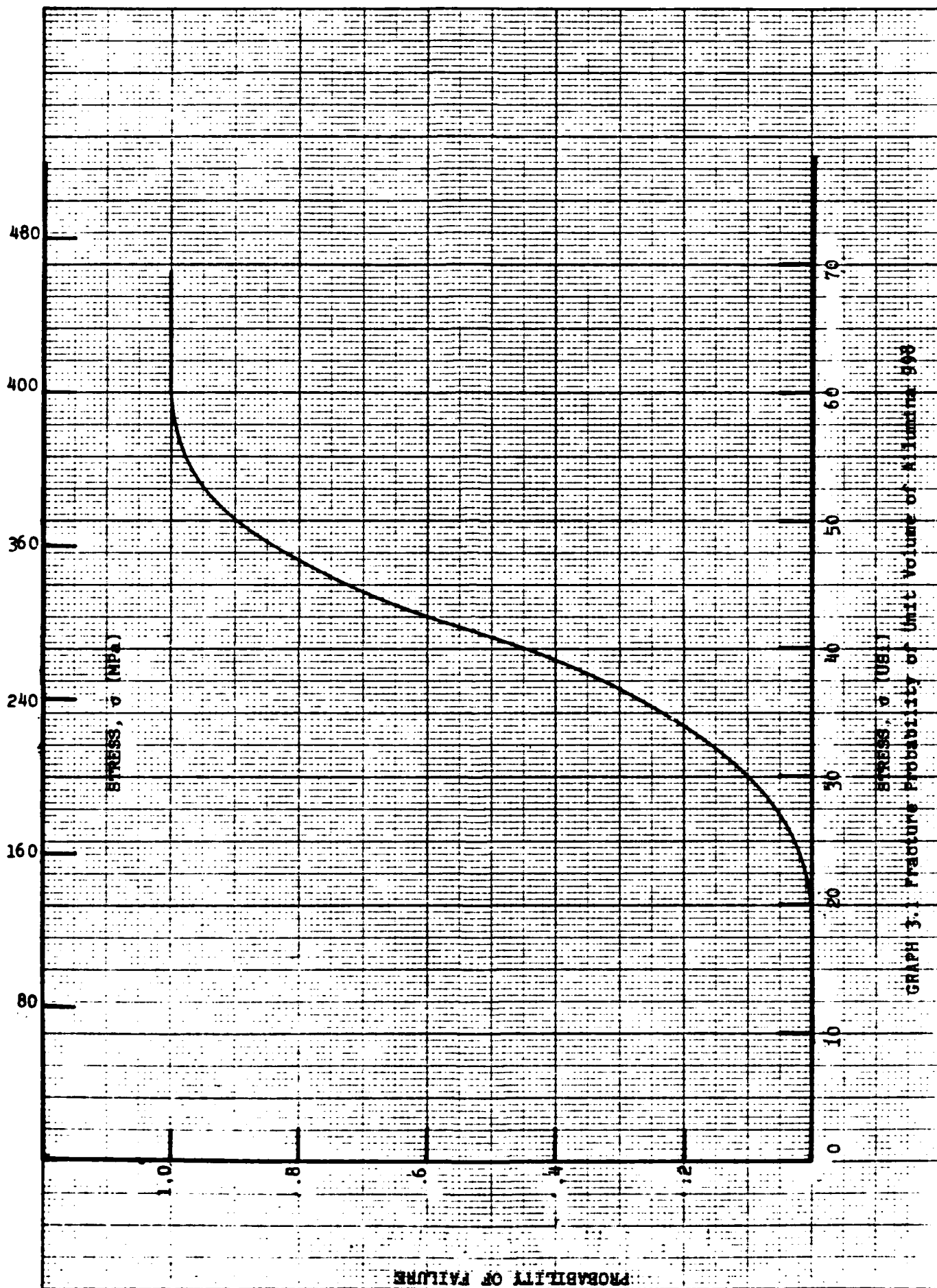
Table 3.2. Data On All Other Alumina Tests

Specimen Number	Torgue at End of Test (Foot Pounds)	Remarks
AT1	132.50	No end cap used -- initial set-up test.
AT2	100	Slipped in grip of initial design end slot.
AT3	100	Slipped in grip of initial design without end slot.
AT4	259.38	First modification of initial grip with no end slot but with roughened surface in contact with collet; it slipped.
AT5	259.38	First modification of initial grip with no end slot but with roughened surface in contact with collet; it slipped.
AT22 AT53 AT54 AT55 AT63	406.09 200 200 200 200	The following tests slipped in the bond with grip of latest design -- some were retested and still slipped. The specimens were discarded because they exhibited smaller than average outer diameters requiring a thick bond line; thus, making for a weakened test without machining undersized end caps.

The actual predicted fracture probability vs. load and input fracture probability vs. load, are given in Table 3.3 below. The probability of failure for a 1 inch³ specimen is given by Graph 3.1 for equation parameters 3.1.

<u>Specimen Rank</u>	<u>Probability of Fracture used as Input</u>	<u>Probability of Fracture from Equation 3.1</u>	<u>Weight</u>
1	.04348	.01997	1
2	.08696	.06428	1
3	.13044	.13205	1
4	.17391	.15447	1
5	.21739	.16639	1
6	.26090	.22631	1
7	.30435	.27519	1
8	.34783	.40665	1
9	.39130	.43272	2
10	.43478	.45462	1
11	.47826	.52563	1
12	.52174	.53850	1
13	.56522	.56273	1
14	.60870	.62682	1
15	.65217	.64900	1
16	.69565	.66534	1
17	.73913	.70284	1
18	.78261	.72108	1
19	.82609	.78145	1
20	.86956	.89587	1
21	.91304	.99549	1
22	.95652	.99959	1

TABLE 3.3 Assumed and Determined Probabilities of Fracture for Alumina 998 Original Test Data



GRAPH 3.1 Failure Probability of Unit Volume of Material

A photograph, Figure 3.1, demonstrates the fixture system immediately following one of the tests. Note the lack of crack propagation into the end-caps and the spiral nature of the fracture surface. Further examples of Alumina specimens just after failure depict samples AT60 and AT62. Attention again is drawn to the lack of any end cap fracture zone that was detectable. Again, a spiral fracture surface is evident that seems to initiate and terminate on a material bifurcation point or cusp presumably representing weakend material - Figures 3.2 and 3.3. Generally the Alumina tubes exhibited very high strengths in comparison to mullite. Some specimens did not fracture until surface tractions exceeded 50 ksi.

Mullite MV33 Results

Mullite Specimens selected randomly for the torsion test were labeled as depicted in Table 3.4. The Mullite (MV33) specimens exhibited somewhat less than $\frac{1}{2}$ the mean strength of the Alumina tubular specimens and were of a different mean dimension, Table 2.1. Because the Mullite was so much weaker, no end caps or adhesive bonds failed in these tests. For these specimens, the Weibull parameters were found to be:

$$\begin{aligned} c &= .958E-27 (\text{in}^{2m} \text{ lbs}^{-m}) & 1.848E-51 (\text{M}^{2m} \text{ N}^{-m}) \\ m &= 6.178 (\text{dim'less}) & 6.178 (\text{dim'less}) \\ \sigma_u &= 0 (\text{psi}) & 0 (\text{MPa}) \\ \sigma_o &= 2.3628E+4 (\sigma_o = c^{-1/m}) (\text{psi}): 162.9 (\text{MPa}) \end{aligned}$$

Residual Error from curve fitting = .3846E-01

As a measure of the fit of the derived distribution with the initial data, Table 3.5 provides a comparison. The graph 3.2 depicts the shape of the fracture curve for a 1 inch³ volume subjected to uniaxial tension.



Figure 3-1. Torsion Test Device Immediately Following Test



Figure 3.2. Failed Alumina Torsion Specimen AT60

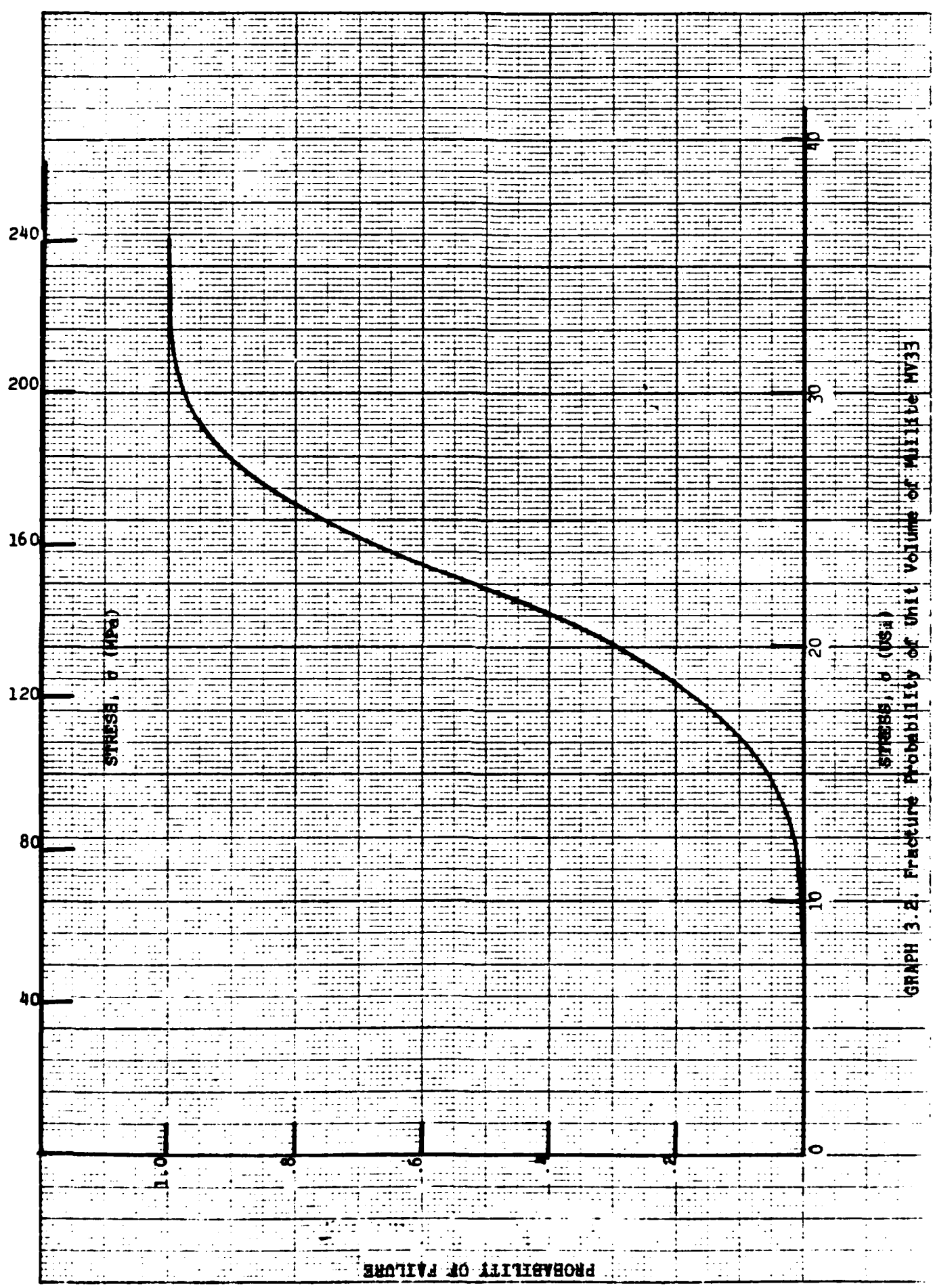


Figure 3.3. Failed Alumina Torsion Specimen AT62

Table 3.4. Torsion Experimental Results -- Mullite MV33
(Data from 24 good tests)

Specimen Number	Rank	Torque at Failure	$\bar{f}(\bar{T}) = i / (N+1)$	Remarks
MT80	1	155.63	.0454	All mullite tests were successful. None had to be duplicated because the fracture loads were significantly below those of the alumina. Four specimens were inadvertently subjected to an oven cure by a research assistant who thought the adhesive should be baked. Those specimens were MT75, 76, 77, and 78. The data was used because the failure loads appeared to be normal. Specimen 70 (MT70) was found to be chipped and was discarded without testing.
MT85	2	179.34	.0909	
MT82	3	192.50	.1364	
MT90	4	211.46	.1818	
MT84	5	247.71	.2273	
MT57	6*	250.00	.2727	
MT86	6*	250.00	.2727	
MT79	7	256.67	.3182	
MT83	8	258.33	.3636	
MT72	9*	266.67	.4091	
MT73	9*	266.67	.4091	
MT81	10	274.48	.4545	
MT71	11*	276.25	.5000	
MT75	11*	276.25	.5000	
MT87	12	285.00	.5455	
MT78	13	288.75	.5909	
MT77	14	292.50	.6364	
MT59	15	292.88	.6818	
MT74	16	300.00	.7273	
MT76	17	300.83	.7727	
MT52	18	328.23	.8636	
MT88	19	339.17	.8636	
MT58	20	345.00	.9091	
MT89	21	360.00	.9545	

* Taken as the same rank with twice normal weight.



GRAPH 3.2. Fracture Probability of Unit Volume of Millite HV35

<u>Specimen Rank</u>	<u>Probability of Fracture used as input</u>	<u>Probability of Fracture from Equation 3.2</u>	<u>Weight</u>
1	.04546	.01949	1
2	.09091	.04616	1
3	.13636	.07059	1
4	.18182	.12260	1
5	.22727	.29362	1
6	.27273	.30784	2
7	.31818	.35139	1
8	.36364	.36270	1
9	.40909	.42203	2
10	.45454	.40869	1
11	.50000	.49429	2
12	.54546	.56249	1
13	.59091	.59187	1
14	.63636	.62112	1
15	.68182	.62407	1
16	.72727	.67853	1
17	.77273	.68475	1
18	.81818	.86166	1
19	.86364	.91127	1
20	.90909	.93219	1
21	.95455	.96981	1

Table 3.5. Assumed and Determined Probabilities of Fracture for Mullite MV33 Original test Data

The computer programs used to derive these data are interactive in nature which may seem inconsistent with the existence of a closed form for the Weibull volume integral (1.3) and simple Least Square solution available [1,3] for circular torsion specimens. This approach, however, allows other minimization parameters to be used as well as a 3-parameter model as appropriate.

It is interesting that the fracture of the mullite specimens differ substantially from the fracture of the alumina specimens. The difference, one of number and size of the fracture fragments, is similar to that observed in the bend tests although on a more dramatic scale. Figures 3.4, 3.5, 3.6, and 3.7 show that the fracture of mullite produces a great many more fragments than the alumina; the uniformity of the fracture patterns with generally a spiral geometry is unmistakable. This uniformity is thought to be a result of (1) the nearly constant strain energy density achieved in this type of test, and (2) the apparent lack of an appreciable stress concentration at the caps. Some of the specimens in the figures have failure well into the caps, Figure 3.4, through 3.7, and some do not, Figure 3.8.



Figure 3.4. Mullite Failure in Torsion. Specimen MT87



Figure 3.5. Mullite Failure in Torsion, Specimen MT88



Figure 3.6. Mullite Failure in Torsion, Specimen MT89



Figure 3.1. Mullite Failure in Torsion. Specimen MT90

The existence of specific bifurcation crack initiation points again is in some evidence in the Mullite specimens. This material demonstrates greater brittleness than Alumina, a finding also to be noted in the pressure tests and in the 4-point loading tests of [1].

Chapter 4

Discription of the Hydraulic Test

The experimental designs and methods employed for the tube specimen tests are characterized by nearly uniform stress states in the test section except for the bending test where a constant principal stress ratio was established. If there is symmetry of Weibull parameters as a function of the direction, ϕ the angle between that point on the fracture surface and the $\hat{n} = (1,1,1) \frac{1}{\sqrt{3}}$ direction, then the identification of these parameters at a sufficient number of values of ϕ defines fracture. The table below describes values of ϕ obtained by common tests.

ϕ Degrees	Test Type
54.7	Uniaxial tension or pure bending
39.2	Internal Pressure
90.0	Torsion
125.3	Compression

Table 4.1. Fracture Surface Angles for Typical Types of Tests

While neither compression nor internal pressure tests were conducted, they could be employed on the same tube specimens. The test developed and described in this chapter supplies a variety of stress ratios by hydraulically loading disk specimens simply supported along their edges, Figure 4.1. This test is necessary to complement fracture theory data gleaned from tube specimens employing a single ϕ . Independence of the action of each principal direction stress, a commonly applied assumption, can be evaluated in this way [4]. In addition this hydraulic test may be an alternate to disk bend tests that roughly approximate this method [5,6].

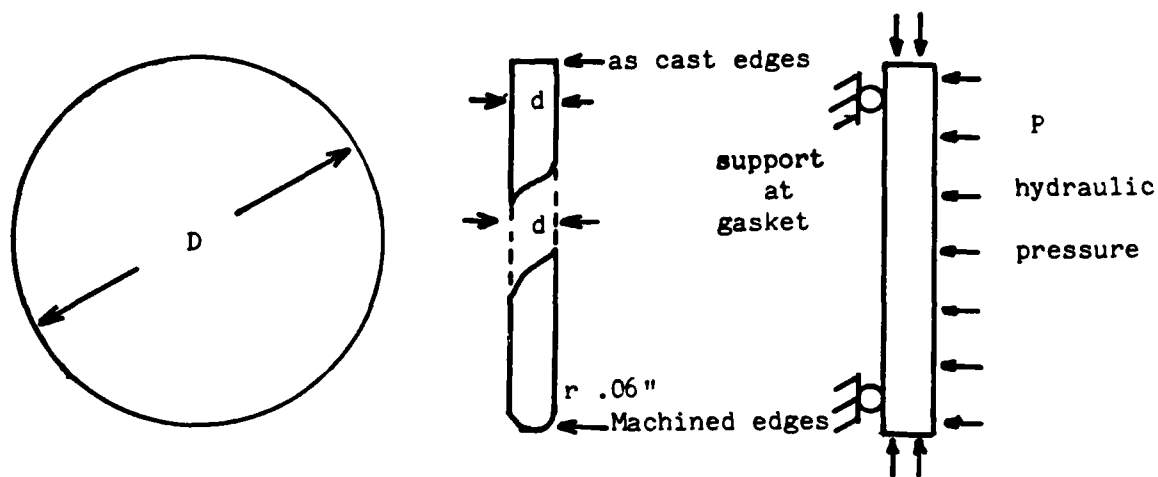


Figure 4.1. Alumina and Mullite Disk Specimens and Loading Conditions

This also provides an additional geometry for application of Weibull theory. Both 'as-cast' samples and disks machined to ensure flat parallel surfaces were used as specimens in both Alumina and Mullite from the same batches as those of the tube specimens. D , d refer to the radius and thickness of 'as-cast' disks and D , d refer to those measurements of machined disks. The machined specimens were ground for flatness and for the removal of sharp edges, Fig 4-1. resulting in a radius of about .125". There was concern that 'as-cast' disks would be pre-disposed to warpage thereby causing considerable parasitic stresses during testing. In order to examine this potential problem, 'as-cast' and ground specimens in both alumina and mullite were all tested to failure. Their dimensions and deviations are given in Table 4.2 in reference to the geometries of Figure 4-1.

Sample Type	D	d	d
	Diameter (in)	Thickness at Center (in)	Thickness .25" From Edge (in)
Alumina - 'as-cast'	2.0210±.0140	.2522±.0038	.2519±.0034
Alumina - ground	2.0263±.0161	.2730±.0060	.2736±.0063
Mullite - 'as-cast'	2.0721±.0172	.2507±.0012	.2508±.0014
Mullite - ground	2.0533±.0196	.2488±.0058	.2502±.0048

Table 4.2 Disk Specimen Dimensions - means and standard deviations

In reference to Table 4.2, it appears that the supplier selected thicker alumina samples for grinding and that grinding did not improve the overall thickness deviations.

While there have been numerous proposed disk specimens for brittle materials, the majority of such tests in checking Weibull parameters are of little value because either (1) they produce inordinately complex stress fields, (2) the mode of testing imparts high stress over a small volume, or (3) the small strain to failure requires the use of expensive high-precision samples and fixtures.

The test fixture of this report consists of a small hand-operated hydraulic pump capable of 10 ksi pressures and a pressure gage and tubing attached to the specimen testing assembly, Figure 4.2. In order to provide for simple support around the side of the specimen and to discourage leakage of hydraulic fluid, a soft copper gasket, cut to fit the assembly bolt pattern is bonded with 3M structural epoxy to the edge of the specimen, Figure 4.3.

The specimen with copper gasket is sandwiched between heavy steel members and held together by stiff preloaded bolts. One side of the specimen is in contact with pressurized hydraulic fluid while the other side is vented to the atmosphere through a 1-1/2 inch hole, Figure 4.4. The difference in a diameter of the specimen and vent provides a lip of 1/4 inch allowing contact with the soft copper seal to achieve a simply-supported condition. More detail is provided by Figures 4.5 through 4.7.

With each test the apparatus is disassembled through the removal of eight bolts and reassembled by the insertion of a new seal and bonded specimen. While some of the seals leaked perhaps due to plastic flow of the copper during the test, it is felt that a better seal design might alleviate this minor problem.

Before a new test is begun, the assembly is bled of air through a small valve; it is then placed in a safety cage. While this test does take longer to complete than the torsion or bending experiments of [1], it can be done in about five minutes by an experienced operator.

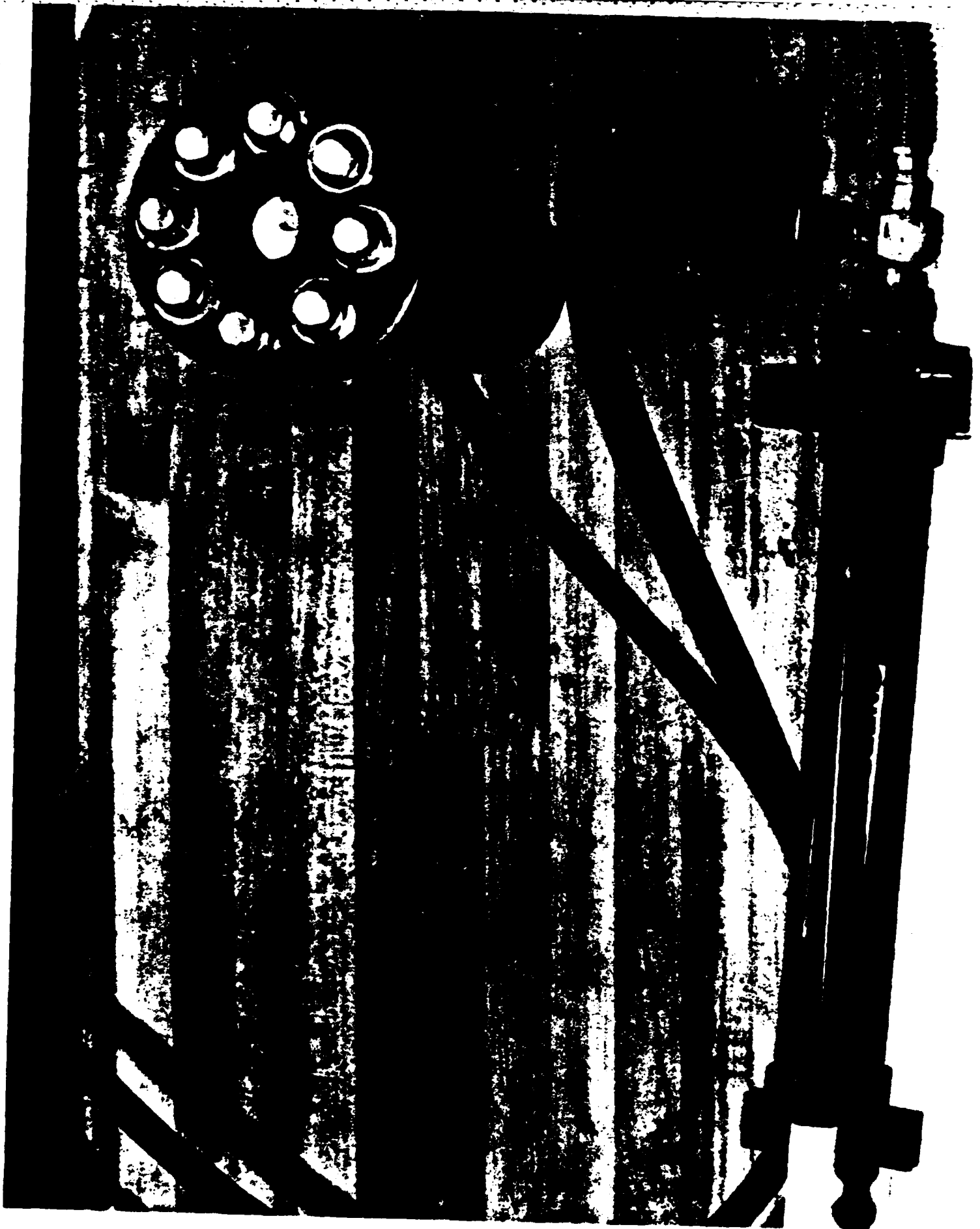


Figure 4-2. View of Hydraulic Test Assembly

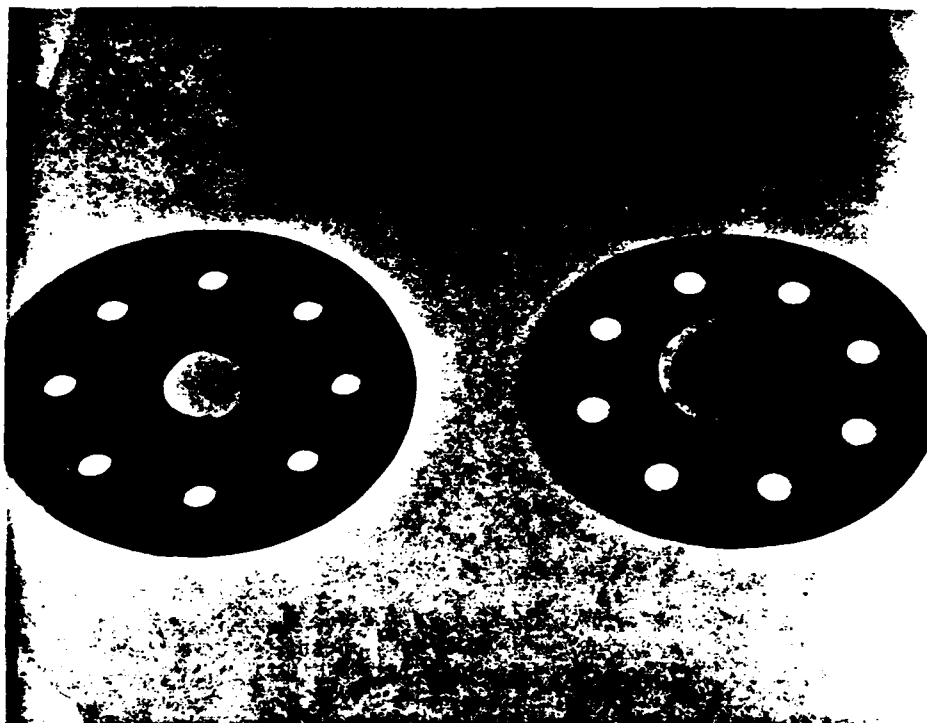


Figure 4.3. Specimen and Gasket - both sides

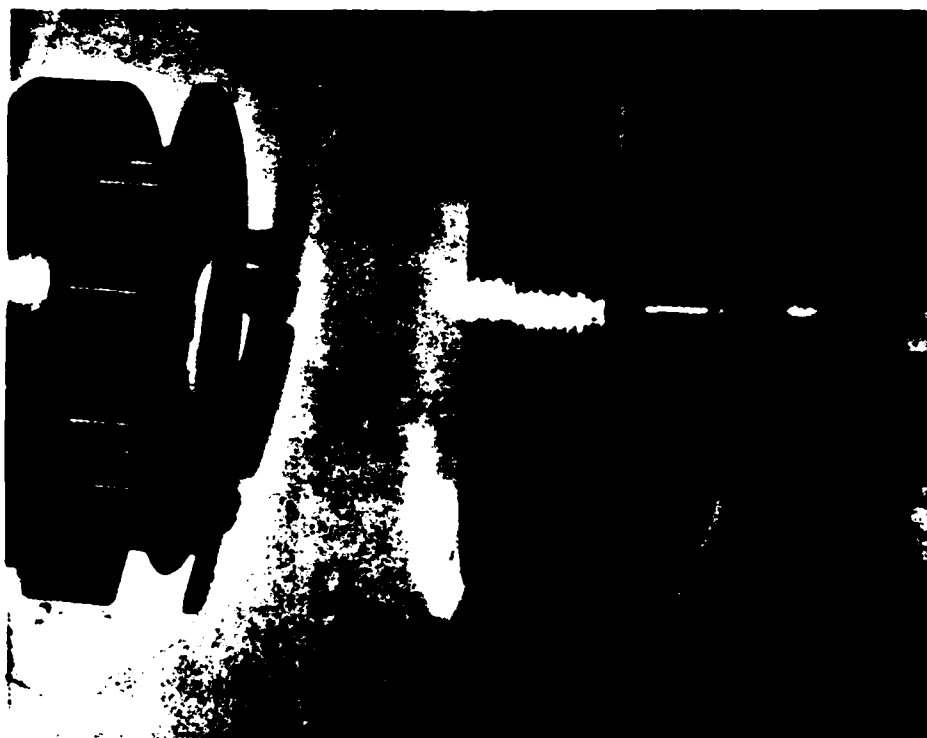


Figure 4.4. Specimen Ready to be Mounted in Assembly - Hydraulic Line and Vent to remove trapped air shown at right.

Fixture Made From SAE 4340-Ground Finish

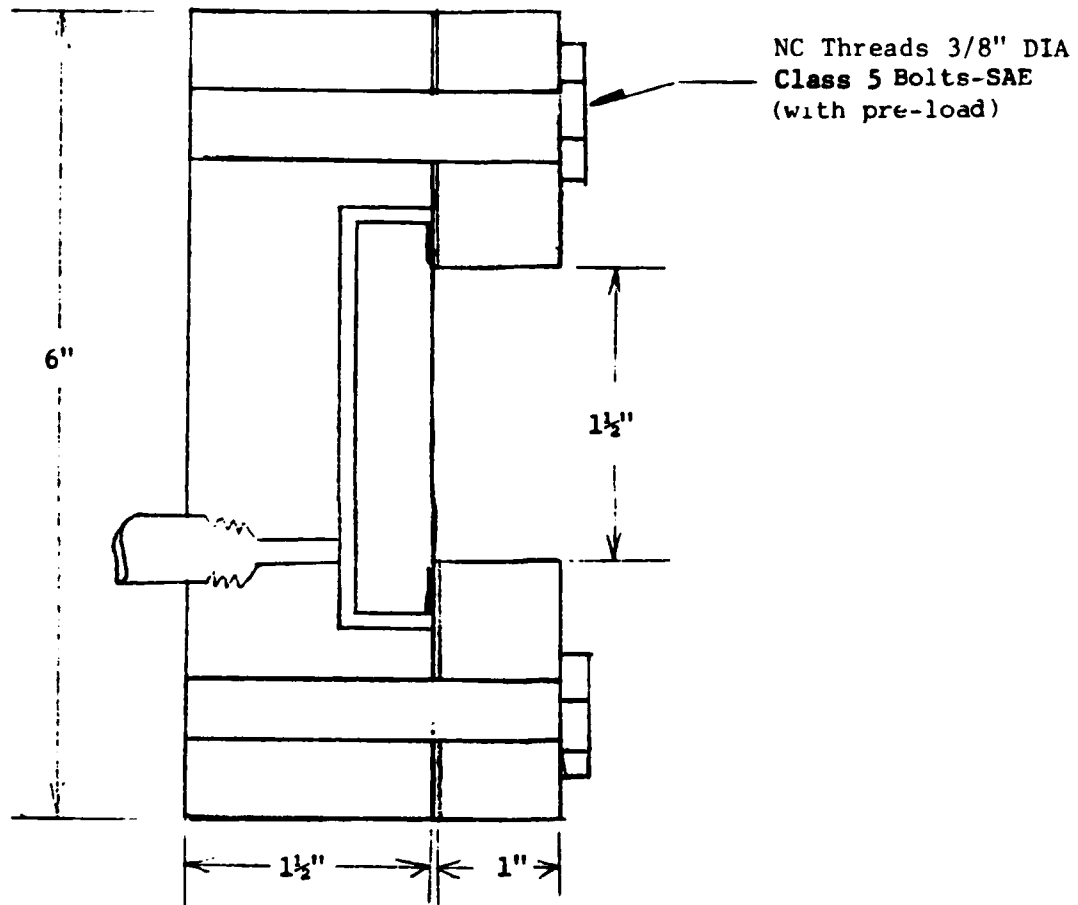


Figure 4.5. Disk Specimen Fixture

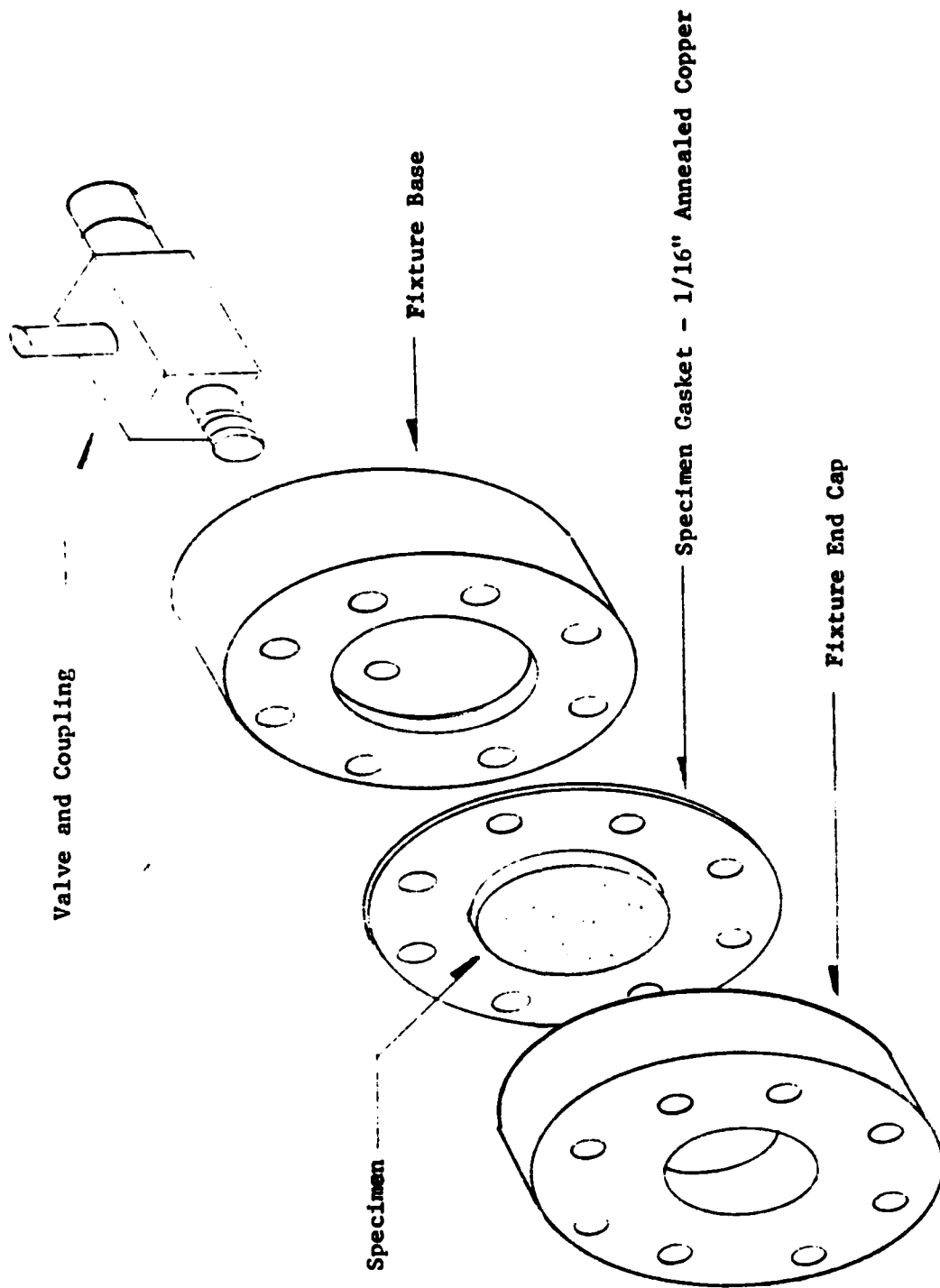


Figure 4.6. Test Fixture for Disk Specimens

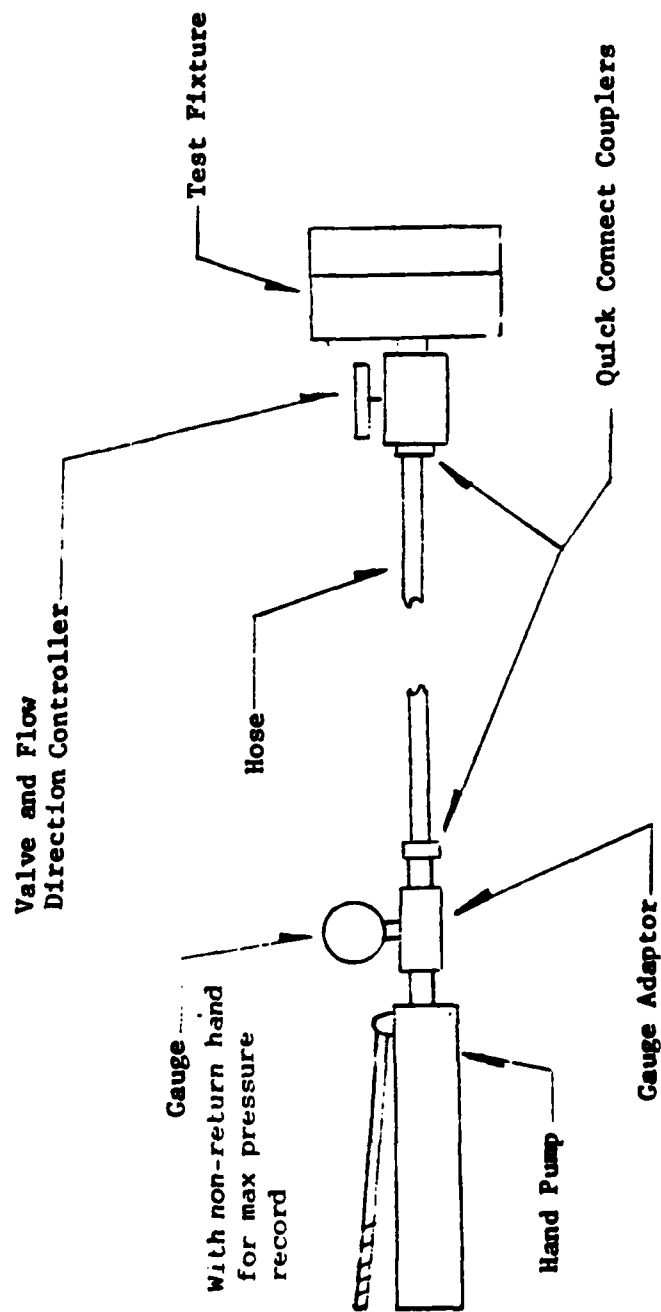


Figure 4 7. Schematic of Hydraulic System Capacity 10,000 p.s.i.

Following the Alumina tests, it was found that the apparatus could benefit from a device to help measure incipient specimen cracking. Particularly with alumina, the pressure at failure was not necessarily the pressure at which a crack first appeared. The alumina specimens seemed to arrest cracks growth until very high pressures were achieved. A conducting brittle coating over the outer side of the specimen with a balance bridge recorder might be of considerable benefit in detecting these initial cracks. Figure 4.8 depicts such devices.

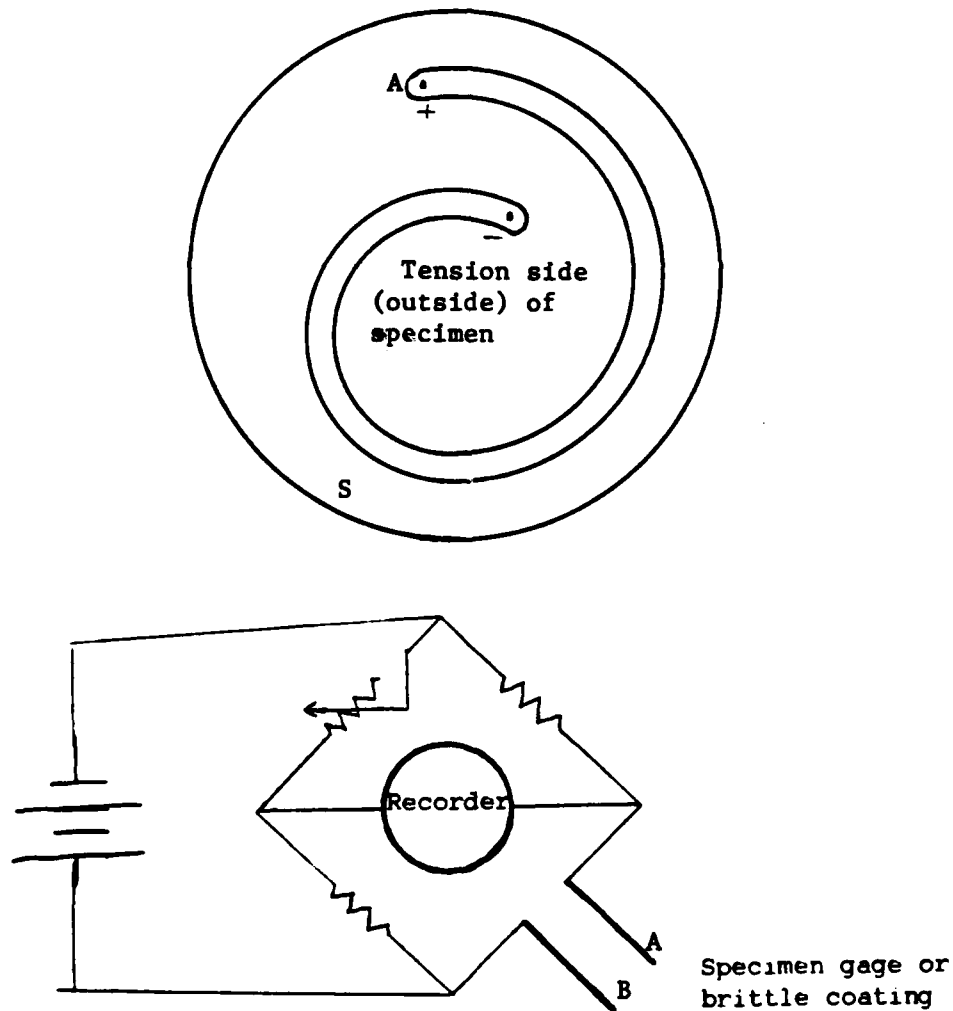


Figure 4.8. Specimens Coating Scheme and Bridge Crack Detector

Chapter 5

Results of the Hydraulic Tests

Both Alumina (998) and Mullite (MV33) disks made from the same batches as the tubular specimens of [1] were tested to failure through hydraulic pressure applied to one surface. The experiment was designed to approximate simple support at the outside where the soft copper seal was compressed between the specimen and pressure retainer.

Actual test results performed on alumina are ranked for (as-cast) disks, Table 5.1a, and for ground-finish disks, Table 5.2a. Four of these specimens in each group, Table 5.1b and 5.2b, could not be fractured due to leaky seals.

The mean strength for ground specimens over 'as-cast' specimens (alumina) showed about a 16.2% increase based on a total of 21 successful as-cast, and 17 successful ground-finish disks.

The leaky seals encountered in both sets of alumina specimen tests all occurred at nearly no pressure reading. Thus, they were not the result of normal gasket failure in the copper but rather a result of potting material failure in the epoxy which was employed in a highly unusual fashion. Because equal numbers of as-cast and ground finish samples could not be tested due to leaks, this phenomenon is was thought not to be related to specimen warpage.

Of further concern in establishing table 5.1a and 5.2a is the fact that the actual pressure level at which fracture occurred in the alumina specimens was difficult to determine with precision. Typically specimens failed without an apparent noise or an abrupt pressure drop. Rather, the alumina samples began to ooze hydraulic fluid as the operator attempted to sustain or increase pressure levels. The figures cited in Tables 5.1a and

Table 5.1a. Alumina Disk (as cast) Pressure Tests
(Mean Pressure at Failure = 2860 p.s.i.)

Specimen Number	Applied Pressure at Failure (p.s.i.)	Remarks
A24P	1400	All tests were normal
A25P	1500	
A22P	2000	
A10P	2175	
A13P	2225	
A14P	2350	
A12P	2400	
A1P	2550	
A17P	2750	
A16P	2925	
A23P	2970	
A26P	3000	
A29P	3050	
A0P	3200	
A1P	3200	
A11P	3350	
A28P	3400	
A15P	2500	
A27P	3670	
A18P	3950	
A30P	4500	

Table 5.1b. Alumina Disk (as cast) Pressure Tests
The following specimens were rejected.

Specimen Number	Maximum Applied Pressure	Remarks
A3P	Not recorded	Leakage at seal
A4P	Not recorded	Leakage at seal
A5P	Not recorded	Leakage at seal
A6P	Not recorded	Leakage at seal

Table 5.2a. Alumina Disk (ground finish) Pressure Tests
(Mean Pressure at Failure = 3365 p.s.i.)

Specimen Number	Applied Pressure at Failure	Remarks
A31P	2360	All test were normal
A41P	2635	
A45P	2750	
A35P	2790	
A8P	2800	
A7P	2900	
A9P	2955	
A42P	3000	
A20P	3150	
A38P	3150	
A33P	3190	
A19P	3650	
A39P	3695	
A32P	3985	
A40P	4500	
A44P	4600	
A21P	5100	

Table 5.2b. Alumina Disk (ground finish) Pressure Tests

The following specimens were rejected.

Specimen Number	Maximum Applied Pressure	Remarks
A34P	Not recorded	Leakage at seal
A36P	Not recorded	Leakage at seal
A37P	Not recorded	Leakage at seal
A43P	3400 p.s.i.	Produced fracture to one side

5.2a essentially represent a pressure at which significant sustained leakage occurred through an identifiable crack. It generally could not be exceeded through the use of a hand operated ram.

The test results for Mullite are given for as-cast samples in Table 5.3a and for ground samples in Table 5.4. The average strength was considerable below that of alumina which may have accounted for fewer failures due to leakage. The as-cast seal failures are given in Table 5.3b. There were no failures in the mullite ground finish specimens. With the mullite disk test there is a 10% difference between average strengths of as-cast tests over ground test, a trend that is opposite that of the alumina disks. A further variance of the mullite results is that three samples in the Table 5.3b failed via a single diagonal crack. These are not included in Table 5.3a due to the concern that wrapage of the as-cast specimens may have produced these anomalies.

A further difference in the testing of Alumina and Mullite disks is that when mullite disks fail, there is an instantaneous pressure drop and an unmistakable crack in the specimen. In addition to the tabulated results of the pressure test, there is additional information to be gleaned from an observation of the fractured specimens. In the photo reproductions that follow, it is important to note that each specimen is labeled with or without a trailing "P" in the specimen number. All disk specimens in Tables 5.1 - 5.3 have a "P" suffix to avoid confusion with tube specimens. This "P" indicates the specimen side exposed to hydraulic fluid pressure and the specimen number with no "P" indicates the external or atmospheric side. Thus, for hydraulic specimen, Al3P, a photograph of its sides will be labeled Al3 (outside) and Al3P (inside). The outside surface photos can also be detected by the adhesive that protrudes at the edge.

Table 5.3a. Mullite Disk (as Cast) Pressure Tests
(Mean Pressure at Failure = 1814 p.s.i.)

Specimen Number	Applied Pressure at Failure	Remarks
M120P	1000	
M114P	1100	
M119P	1100	
M117P	1200	
M121P	1210	
M122P	1290	
M123P	1300	
M126P	1300	
M109P	1450	
M125P	1450	
M101P	1500	
M115P	1500	
M110P	1750	
M108P	1850	
M103P	1900	
M116P	2100	
M113P	2400	
M124P	2450	
M99P	2650	
M100P	3700	
M102P	3900	

Table 5.3b. Mullite Disk (as Cast) Pressure Tests
The following specimens were rejected.

Specimen Number	Maximum Applied Pressure	Remarks
M104P	950	Single, large, diagonal break
M105P	900	Single, large, diagonal break
M106P	800	Single, large, diagonal break
M107P	Not Recorded	Leak at seal
M111P	Not Recorded	Leak at seal
M112P	Not Recorded	Leak at seal
M118P	Not Recorded	Leak at seal

Table 5.4. Mullite Disk (Ground Finish) Pressure Tests
(Mean Pressure at Failure = 1638 p.s.i.)

Specimen Number	Applied Pressure at Failure	Remarks
M202P	600	
M201P	1280	
M200P	1410	
M205P	1430	
M207P	1525	
M208P	1750	
M203P	1900	
M204P	2200	
M206P	2650	

A series of two alumina photos are listed for both specimens A11P and A13P. Figure 5.1a shows the outer side and 5.1b shows the inside. The inside of this specimen and of others are shown in greater magnification to provide enhanced evidence of cracks. It is interesting that this specimen gives no indication of a crack on its compression or inner surface although there is a radial or 'crow foot' pattern in the outer surface, Figure 5.1a. This type of crack was typical of almost all alumina specimens such as Figure 5.2a (outside) and 5.2b (inside). While the outside surface exhibited a series of hairline width cracks, no visual crack could be detected on the inside. Indeed there was little change in pumping pressure as detected by the test operator. In fact, with the alumina, pressures could be re-elevated provided the pumping rate exceeded the small leakage rate through the specimen. There was an attempt to detect incipient cracking by listening while observing the specimens by closed circuit TV. In the case of alumina, this revealed nothing.

Several of the alumina specimens were later cleaned; they appeared still to be intact except for the hairline cracks made visible by rubbing the surface with graphite mixture. Again, future tests could benefit from an addition of a conducting brittle strip to aid in crack detection. These specimens appeared to have cracks, Figures 5.1 and 5.2, that propagated to depth of about 50 mm to 70 mm under the outer surface where they appeared to arrest. The exact cause of the hydraulic leak in alumina seems to have been due to seepage through internal cracks that became sealed after the test and not by leakage at the seal.

The corresponding tests on mullite showed a great deal of difference in both the strength of the specimen and in the fact that again fracture appeared much more definite and catastrophic. In specimen M108, representative of mullite samples, fracture is through the entire thickness of

the specimen, Figure 5.3a (outside surface) and Figure 5.3b (inside surface). While the fracture is through the entire thickness, the pattern on the two surfaces is greatly different indicating that the crack has not propagated uniformly throughout the mullite. There is considerable evidence of spalling on the pressure (compression) side of this specimen while a lesser amount of material has been lost at the outer side.

Specimen M115 shown in Figures 5.4a and 5.4b provides a more complex fracture pattern although no spalling can be noted. Again, it is seen that the surface crack patterns are greatly different on the inside and outside. In about all cases the radial ray or 'crow foot' fracture pattern was noted. This was observed in both alumina and mullite failed samples and is thought to be the result of a similar stress field with radial symmetry with failure occurring along rays where the material is statistically weak.



Figure 5.1a. Hydraulic Fracture Specimen A11 - Outer Surface (2.5X)



Figure 5.1b. Hydraulic Fracture Specimen A11P - Inner Surface (3X)

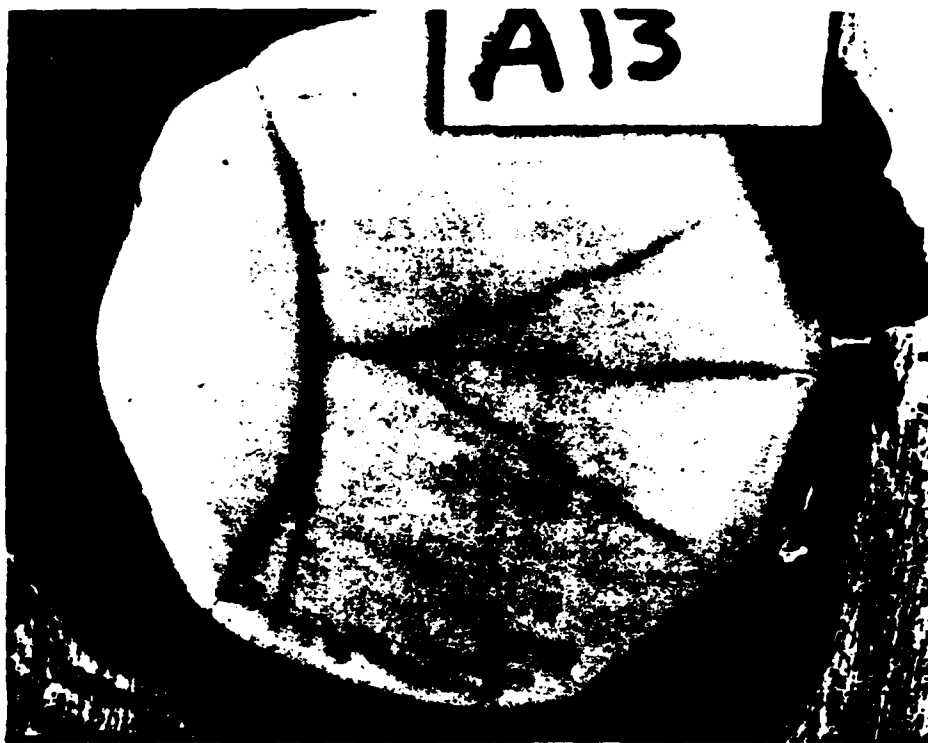


Figure 5.2a. Hydraulic Fracture of Specimens A13 -
Outer Surface (2.5X)



Figure 5.2b. Hydraulic Fracture of Specimens A13P -
Inner Surface (3X)

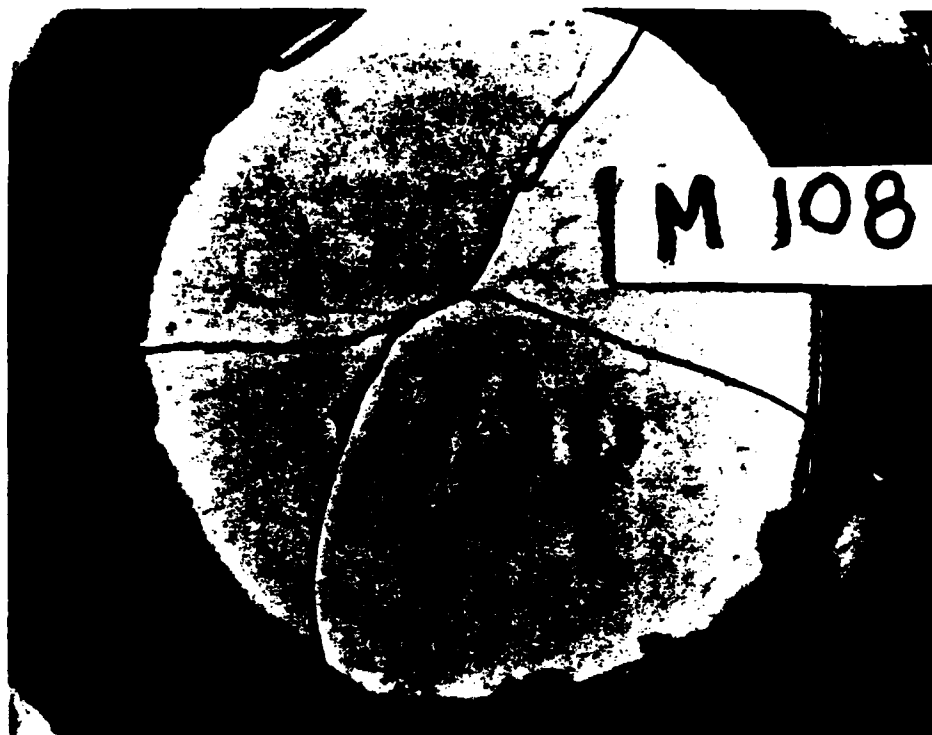


Figure 5.3a. Hydraulic Fracture of Specimen M108 -
Outer Surface (2.5X)

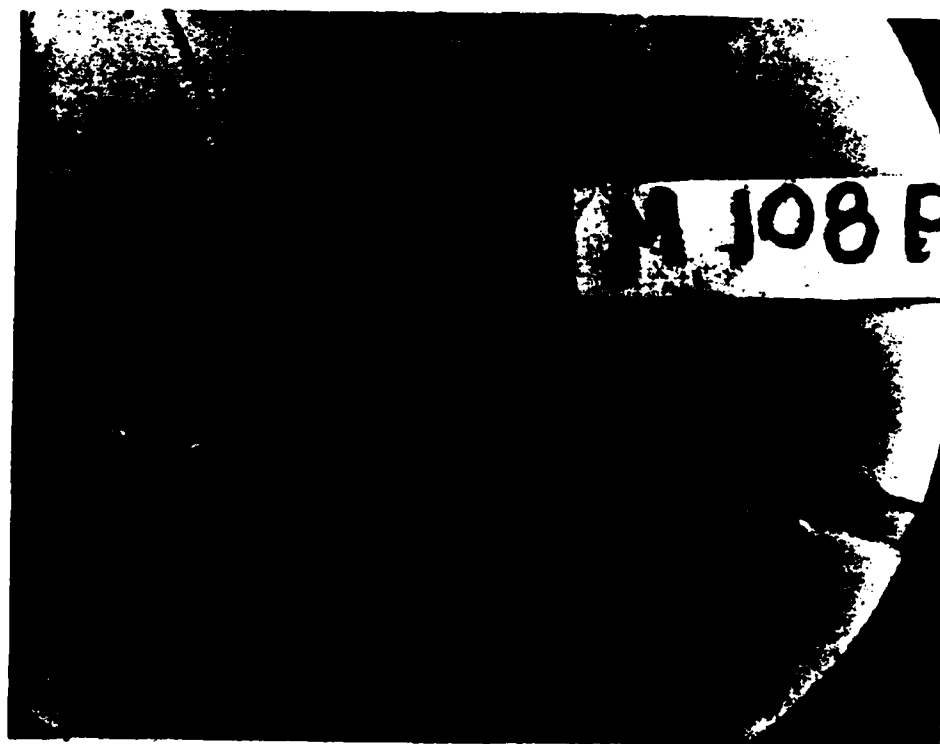


Figure 5.3b. Hydraulic Fracture of Specimen M108P -
Inner Surface (3X)

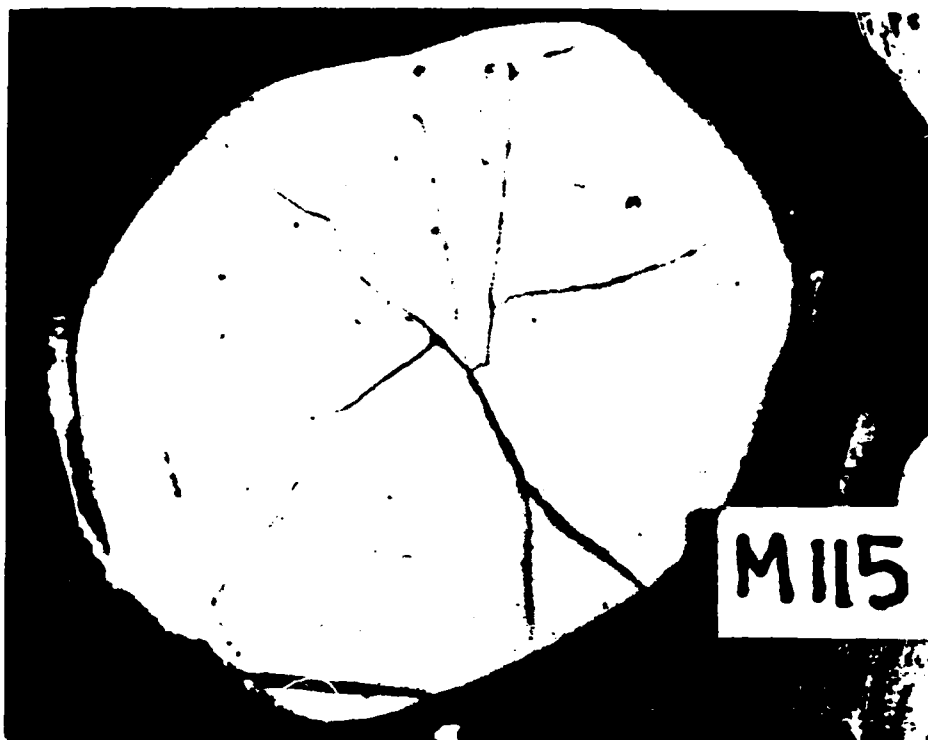


Figure 5.4a. Hydraulic Fracture of Specimen M115 -
Outer Surface (2.5X)



Figure 5.4b. Hydraulic Fracture of Specimen M115P -
Inner Surface (3X)

Chapter 6

Conclusion

The experimental investigations embodied by Navair Reports N00019-78-C-0520 and N00019-79-C-0574 form a set of experiments that provide Weibull data on bending and torsion for tube specimens and for a uniformly loaded circular plate. General conclusions are:

1) The feasibility of torsion and bending tests on one specimen geometry of a class of ceramic material has been demonstrated. Evidence of erratic results produced by stress concentrations near points of load application is not significant.

2) The use of inexpensive tube geometries may reduce the cost of ceramic specimen tests when complemented by the experiments, theory and programming techniques of this report.

3) The use of hydraulic pressure in providing a suitable loading mechanism has been demonstrated. Improvements to this technique may however be:

- a) the use of a simple retaining bladder to restrict hydraulic fluid excursion and seepage into potentially porous specimens.
- b) the inclusion of a fracture detecting brittle conductive coating or other techniques to provide a more precise measurement of incipient fracture in materials that have some fracture arresting capability such as the alumina tested.
- c) the modification of current copper seals and specimen bonding materials or techniques to reduce reject tests.

4) A thorough analysis is yet to be made of the disks to test whether or not raw Weibull data can accurately predict failure distributions in materials to which a complex stress field is applied. Such a study is ongoing and will be the subject of a forthcoming submitted publication.

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- [3] Weil, N. W., and Daniel, I. M., "Analysis of Fracture Probabilities in Non-Uniformly Stressed Brittle Materials," American Ceramic Society Journal, Volume 47, No. 6, June 1964.
- [4] Barnett, R. C., et al., "Utilization of Refractory Non-Metallic Materials in Future Aerospace Vehicles," Flight Dynamic Laboratory, Air Force Systems Command, Contract No. AF33-(615)-1494, Sept., 1965.
- [5] Shetty, D. K., Rosenfield, A. R., Bansal, G. K., and Duckworth, W. H., "Biaxial Fracture Studies of a Glass-Ceramic," Journal of the American Ceramic Society, Volume 64, No. 1, January 1981.
- [6] Marshall, David B., "An Improved Biaxial Flexure Test for Ceramics," Ceramic Bulletin, Volume 59, No. 5, 1980.

Appendix I Computer Programs -- 64

- A) Torsio (AUTO.FOR)
- B) AGAR (AGARWA.FOR)

Appendix II Distribution List -- 67

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      PROGRAM TORSION
C THIS PROGRAM GIVES WEIBULL PARAMETERS FOR TORSION
      REAL I
      DIMENSION EPF(40),W(40),AA(3), P(40)
      DIMENSION VAR(3,3),RES(3,3)
      COMMON/PAK/K,L,C,T(40),EJ,SIGU,EM,RO,RI,TPF(40)
      WRITE(5,40)
40    FORMAT(2X,'ENTER DATA FILE NUMBER AND ITER FRPOP ROUND')
C N=NO OF DATA POINTS
C T=TORQUE AT RUPTURE
      READ(5,*) ND,FRP
      READ(ND,*) K
      DO 400 I=1,K
      READ(ND,*) T(I),W(I)
      T(I)=12.*T(I)
C THIS IS TO CONVERT FROM FT.LB. TO IN.LB.
C THE FILE'S UNITS ARE FT.LB.
400  CONTINUE
      X=FLOAT(K+1)
C NOW RANK THE DISTRIBUTION
      DO 1 I=1,K
      EPF(I)=FLOAT(I)/X
      READ(ND,*) L,RO,RI
      EJE=3.1416/2.*(RO**4-RI**4)
C LEAGAGE LENGTH OF SPECIMEN
C NO=OUTER RADIUS OF SPECIMEN
C KI=INNER RADIUS OF SPECIMEN
C LJ=POLAR MOMENT OF SPECIMEN
      WRITE(5,59) L,RO,RI,EJ
59    FORMAT(/,2X,'L=',1X,F8.4,' RO=',1X,F8.4,' RI=',1X,F8.4,
1    ' EJ=',1X,E10.5)
      GO TO 11
111  WRITE(5,112) C,FM,SIGU,RES(1,3)
112  FORMAT(1X,'C,FM,SIGU,RES=',4E15.6)
11  WRITE(5,42)
42  FORMAT(1X,'INPUT FM,SIGU, AND ICODE FOR C ITERATION')
      READ(5,*) EM,SIGU,ICODE
      IF (ICODE.EQ.1) GO TO 90
      SIGMAX=T(K)*RO/EJ
      DEN1=(SIGMAX-SIGU)**EM
      ENC=ALOG(2,)/DEN1
      VAR(1,3)=ALOG(2,)*FLOAT(K)/DEN1
      CN=0
C NOW REGIN C ITERATION TO MINIMISE RES
      WRITE(5,69) VAR(1,3),ENC
69  FORMAT(1X,'INPUT INITIAL C AND DELTA C.OLD C,D=',2E10.4)
C 10  READ(5,*) VAR(1,3),ENC
C 12  COMCN
C 14  ICT=0
      VAR(1,1)=VAR(1,2)
      VAR(1,2)=VAR(1,3)
      RES(1,1)=RES(1,2)
      RES(1,2)=RES(1,3)
      VAR(1,3)=VAR(1,3)+ENC
      ICT=ICT+1
      IF (ICT.GT.1000) GO TO 98
      C=VAR(1,3)
      CALL AGAR
C NOW COMPUTE THE RESIDUALS
      RES(1,3)=0.
      DO 5 I=1,K
      WN=(EPF(I)-TPF(I))
      RES(1,3)=RES(1,3)+W(I)*WN*WN
5    CONTINUE

```

```

C      WRITE(5,78) (VAR(1,J1),J1=1,3),(RES(1,J2),J2=1,3)
/8      FORMAT(1X,'VAR,RES 1 TO 3 ARE:'6E10.4)
C IF LESS THAN 3 VALUES JUST CONTINUE.
      IF (ICT.LT.4) GO TO 14
C IF RES(1,2) IS LESS THAN RES(1,1) AND RES(1,3) THEN WE HAVE A MIN.
C SO CALCULATE WHERE MIN OCCURS, DROP ENC BY 10 AND CONTINUE.
      IF ((RES(1,2).LT.RES(1,1)).AND.(RES(1,2).LT.RES(1,3)))
1GO TO 13
C IF RES(1,3).LE.RES(1,2) CONTINUE ELSE REVERSE ENC.
      IF (RES(1,3).LE.RES(1,2)) GO TO 14
      ENC=ENC/2.
      GO TO 12
13      CALL MINI(1,VAR,RES,CN)
      WRITE(5,89) CN,RES(1,3)
89      FORMAT(1X,'CN AND RES(1,3) FOR THIS MIN ARE:',2E15.6)
      IF (ABS((CO-CN)/CN).LT.ERR) GO TO 111
      ENC=ENC/10.
      VAR(1,3)=CN
      GO TO 10
90      DO 81 I=1,K
      WRITE(5,79) (I,EPF(I),TPF(I),W(I))
79      FORMAT(1X,'I,EPF,TPF,W=',I2,3(1X,F13.6))
81      CONTINUE
      WRITE(5,310)
310      FORMAT(1X,'NOW INPUT SIG1,DELSIG1,NO OF DIVISIONS')
      READ(5,*) ST1,DST1,NOD
      DO 312 I=1,NOD+1
      F=0
      IF (ST1.LE.SIGH) GO TO 313
      Z=C*(ST1-SIGH)*EM
      F=1.-EXP(-Z)
      WRITE(5,314) ST1,F
314      FORMAT(1X,'STRESS=',F10.5,' PROR=',F7.5)
      ST1=ST1+DST1
312      CONTINUE
88      WRITE(5,88) ICT
88      FORMAT(1X,'IMAX EXCEEDED, IMAX=',I6)
      GO TO 99
99      STOP
      END
      SUBROUTINE MINI(I,VAR,RES,XXX)
      DIMENSION VAR(3,3),RES(3,3)
      P1=RES(I,1)/((VAR(I,1)-VAR(I,2))*(VAR(I,1)-VAR(I,3)))
      P2=RES(I,2)/((VAR(I,2)-VAR(I,1))*(VAR(I,2)-VAR(I,3)))
      P3=RES(I,3)/((VAR(I,3)-VAR(I,1))*(VAR(I,3)-VAR(I,2)))
      AAA=P1+P2+P3
      RRR=P1*(VAR(I,2)+VAR(I,3))+P2*(VAR(I,1)+VAR(I,3))
1  +P3*(VAR(I,1)+VAR(I,2))
      XXX=.5*RRR/AAA
      IF (VAR(I,1).GT.VAR(I,3)) GO TO 2
      IF ((XXX.GT.VAR(I,3)).OR.(XXX.LT.VAR(I,1)))
1 XXX=(VAR(I,1)+VAR(I,3))/2.
      RETURN
2      IF ((XXX.GT.VAR(I,1)).OR.(XXX.LT.VAR(I,3)))
1 XXX=(VAR(I,1)+VAR(I,3))/2.
      RETURN
      END

```

```

      SUBROUTINE AGAR
      REAL L
      COMMON/PAR/K,I,C,T(40),FJ,SIGU,FM,RO,RI,TPF(40)
C   COMPUTE DETERMINED PROBABILITIES AT EACH TORQUE,
C   STORE IN TPF(1 TO K).
C   FIRST COMPUTE NECESSARY CONSTANTS,
      R=3.1416*2.*L*C/(FM+1.)
      DO 1 I=1,K
      TPF(I)=0.
      TIL=T(I)/FJ
C   NOW COMPUTE R AT WHICH THE THRESHOLD STRESS OCCURS.
      RLOW=RI
      RTH=SIGU/TIL
      IF (RTH.GE.RO) GO TO 1
      IF (RTH.GT.RI) RLOW=RTH
C   NOW COMPUTE BN.
      C1=RO*TIL
      C2=(C1-SIGU)**(FM+1.)
      C3=C2*(C1-SIGU)
      D1=RLOW*TIL
      D2=(D1-SIGU)**(FM+1.)
      D3=D2*(D1-SIGU)
      RN=R*((RO*C2-C3/(TIL*(FM+2.))))-
1      (RLOW*D2-D3/(TIL*(FM+2.)))/TIL
      TPF(I)=1.-EXP(-RN)
      CONTINUE
      RETURN
      END

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Richard W. Perkins, Jr.
Professor and Chairman,
Department of Mechanical
and Aerospace Engineering
139 E. A. Link Hall
Syracuse, N. Y. 13210

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