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FRACTURE AND NITROGEN TETROXIDE/SUSTAINED LOAD FLAW GROWTH OF 6AI-4V TITANIUM

J. N. Masters, et al

October 1969

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FRACTURE AND NITROGEN TETROXIDE/SUSTAINED LOAD FLAW GROWTH OF 6Al-4V TITANIUM

Final Report

By

J. N. Masters, W. P. Haese and W. D. Bixler

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AEROSPACE SYSTEMS DIVISION

THE BOEING COMPANY

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ABSTRACT

The static fracture and sustained load flaw growth characteristics were evaluated experimentally for 6Al-4V titanium forgings and weldments. Fracture toughness tests were performed in air at room temperature and in liquid nitrogen at -320°F. The sustained load tests were conducted in nitrogen tetroxide at a temperature of 80 to 90°F. It was concluded based on these test results that the fracture toughness of 6Al-4V titanium is (1) moderately dependent on material heat, (2) lower with decreasing temperature and (3) a minimum in the weld heat affected zone. It was also concluded that the threshold stress intensity in nitrogen tetroxide at 80 to 90°F is above 28.0 ksi $\sqrt{in}$, independent of material heat or flaw location.

KEY WORDS

Fracture

Threshold stress intensity

Nitrogen tetroxide

FOREWARD

This report describes the work performed by the Aerospace Systems Division of The Boeing Company during the period from May 5, 1969 to September 3, 1969 under contract by the Jet Propulsion Laboratory, P.O. GR-488959 to determine the fracture characteristics in nitrogen tetroxide of the 6AL-4V titanium alloy to be used on Mariner Mars 1971 Propulsion Subsystem Propellant Tanks. No reportable items of new technology have been identified during the course of this program.

Boeing personnel who participated in this investigation include J. N. Masters, Program Leader, W. P. Haese, Technical Leader and W. D. Bixler, research engineer. Structural test support was provided by A. A. Ottlyk and G. E. Vermilion. Quality assurance was provided by A. F. Lawler.

The information contained in this report is also released as Boeing Document D2-121397-1.

SUMMARY

The objective of this program was to determine the plane strain fracture toughness and threshold stress intensity of 6Al-4V titanium forgings and weldments. Base metal and weld metal specimen thicknesses were selected to be representative of a Mariner 71 propellant pressure vessel. Two surplus surveyor material heats were used in the testing. The fracture toughnesses of the base metal and weld metal were determined in air at room temperature and in liquid nitrogen at -320°F. A summary of the average fracture toughness values (ksi $\sqrt{in}$) obtained is presented below:

TEMP	HEAT NUMBER	BASE METAL	WELD METAL	
			℄	HAZ
R.T.	G-804	45.1 [1]	46.7	48.5
	301275	44.9 [1] (55.0)	47.0	48.2
-320°F	G-804	55.8	46.0 [2]	38.0
	301275	46.3	38.8	32.1

[1] Not plane strain values; specimens failed as thru-the-thickness crack.

[2] Based on a specimen with an incomplete initial flaw.

The sustained load tests were conducted in a controlled nitrogen tetroxide environment containing a minimum of 0.60 percent nitric oxide. The nitrogen tetroxide was maintained between 80 to 90°F and at 315 psig. A summary showing the minimum threshold stress intensity is presented below:

MATERIAL	HEAT NUMBER	MINIMUM THRESHOLD STRESS INTENSITY (ksi $\sqrt{in}$)
Base Metal	G-804	30.0
	301275	28.0
Weld Metal	G-804	32.0
	301275	31.0

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1.0 INTRODUCTION

In general, unexpected failures in pressure vessels originate at flaws. Failures of pressure vessels have occurred during proof test and operational service. Proof test failures result where the size of a flaw is sufficiently large to become unstable before proof stress is attained or the flaw grows sufficiently large to become unstable at proof stress during a hold period. In the former case the critical stress intensity of the material is exceeded on loading. In the latter case the threshold stress intensity of the material/pressurizing fluid is exceeded resulting in subcritical flaw growth until the critical stress intensity of the material is reached. Operational failures have often occurred after a flaw grew sufficiently in size to become unstable at the operating stress. An estimate of the performance capability of pressure vessels requires knowledge of initial flaw sizes, critical flaw sizes, and subcritical flaw growth characteristics of the vessel material.

This experimental investigation was undertaken to determine the fracture toughness of 6Al-4V titanium forgings and weldments at room temperature and -320°F in liquid nitrogen, and the threshold stress intensity in nitrogen tetroxide. Two different heats of titanium remaining from the Surveyor program were tested with the weld metal specimens flawed in the weld centerline and heat affected zone. The nitrogen tetroxide used for the sustained load tests met MSC-PPD-2B and contained a minimum of 0.60 percent nitric oxide and was maintained between 80 and 90°F. The thresholds were established based on tests that were of 1000 hours duration.

2.0 BACKGROUND

Most prior experiments involving sustained load flaw growth behavior have dealt with relatively thick materials and the accompanying analytical procedures were based upon shallow flaw concepts. It appears reasonable that this type of data can be extrapolated to some extent to thinner materials by use of available approximate deep flaw solutions. However, it does not yet appear to be feasible to extend these procedures to the very thin vessels where surface flaws can extend nearly through the thickness. The major differences which are pertinent to the study reported herein are discussed in the following paragraphs.

Normally, in the testing of surface flawed specimens made of relatively brittle materials, stable slow crack extension occurring during the loading cycle to failure is so small that it appears permissible to neglect this factor in calculating results. K_{Ic} values are determined simply by measuring the maximum load and the initial flaw size. Similarly, analysis of sustained load tests of these same materials is simplified by this fact. For example, consider a precracked specimen rapidly loaded and then held at a given stress level for a period of time The specimen is then unloaded, fatigue "marked", then pulled to failure. Growth which occurred during the load cycle is then evidenced by a separation between the initial crack and the fatigue mark For brittle materials of sufficient thickness (i.e., where the critical flaw depth at moderate stress is small with respect to thickness) it is safe to assume that the amount of flaw growth during the loading cycle is negligible or even non-existent and, therefore, all flaw growth evidenced by the separation between the initial and final marks is time dependent growth. Certainly, if the growth (at low stress intensity levels, K) during loading was significant and measurable, then thresholds on the order of 80 to 90 percent of critical would not have been recorded. Recently, several cases of significant growth-on-loading have been observed [1,2,3] In each case, this has involved materials and thicknesses in which critical flaw depths were not small with respect to thickness. While firm evidence is not yet available, this behavior

might be related to the size of the plastic zone $(\rho)^*$ at the bottom of the flaw front in relation to the remaining unbroken ligament (t_n). It appears that if the flaw front is within one plastic zone size from the back side, then stable growth on loading can occur. In the extreme case the flaw may grow slowly completely through the thickness (without becoming critical). Additional increase in load merely results in failure of a through flaw. This was observed in the work of reference (1), and the test results fit remarkably well with predictions based upon K_c data.

The effect of this behavior on conventional sustained load experiments is to confound the analysis. At one extreme, the flaw might grow thru the thickness during loading (present test setups for testing in hazardous liquids are not equipped to detect this occurrence). In finite width specimens, this growth is accompanied with a significant increase in net stress. With additional time, the flaw can increase in length and a delayed failure results. On the other hand, depending on the stress intensity value at maximum load, the slow growth on loading can halt when the maximum load is reached. After this specimen is unloaded, marked, and failed, a sign of growth (separation of the marks) is seen, but it is virtually impossible to determine after the test just when this growth took place.

Once it is recognized that this type of behavior is taking place, it is a relatively simple task to instrument the specimen and thereby monitor the flaw growth continuously. An example of such an approach is disucssed in detail in reference (2).

* The plastic zone size can be approximately measured by $\rho = \frac{\pi}{16} \left(\frac{K_I}{\sigma_{ys}} \right)^2$

3.0 MATERIALS

3.1 Titanium Forgings & Weldments

Base metal specimens and panels for the welded specimens tested in this program were machined from eight 6Al-4V titanium forgings from the Surveyor program supplied in the solution-heat treated condition. Four of the forgings serial numbers 222, 229, 263 and 265 were from Reactive Metals heat number 301275. The other four forgings serial numbers 236, 238, 252 and 256 were from Titanium Metals Corporation of America (TMCA) heat number G-804. Chemical compositions of both heats are found in Table 1. Specimens and weld blanks oriented parallel to the tank axis are shown in the Figure 1. Weld panels were machined with the edge preparation found in Detail B of JPL propellant tank drawing 10028537, revision B. Welding of the panels was performed by the tank fabrication contractor in a rigid chamber using the tungsten inert gas process in accordance with the welding requirements stated in JPL's propellant tank specification ES 504496A.

3.2 Test Fluids

The sustained load tests were conducted using nitrogen tetroxide that had been analyzed per MSC-PPD-2B. The results of the assay, taken prior to test initiation, are shown in Table II. The initial nitric oxide content of 0.64 percent was within the specified amount of 0.80 ± 0.20 percent, and was selected to give worst case results within the specification since the threshold decreases with decreasing content(Reference 4). Samples of the test fluid were extracted after completion of the 1000 hour tests and analyses were performed to determine if the chemical components were still within specification limits. Only the nitric oxide content of the samples did not meet the specification as indicated in Table II. In two of the three samples extracted, the nitric oxide content was below the minimum specification limit. The low nitric oxide in these samples can possibly be explained by the fact that in the early phase of the test, the nitrogen tetroxide level dropped in these test machines, independent of each other. As a result, additional oxidizer had to be added to each test setup. During this process of leak and refill any introduction of air, water or material that would reduce its quantity by converting to nitrogen dioxide. Likewise, the nitric oxide can combine with chloride to form nitrosylchloride which did show a slight increase over the test period.

4.0 PROCEDURES

4.1 Base Metal Specimen Fabrication

The specimen configuration used for evaluation of base metal forging properties is shown in Figure 2. Specimen thickness was selected as 0.031 inches, the basic membrane thickness of the Mariner 71 propellant tank. The fabrication sequence was planned to simulate, as closely as possible, the steps used to fabricate the tank. The sequence was as follows:

1. The solution-heat treated specimens were rough machined. The rough machining thickness was selected to assure a minimum of 0.02 inches of metal removal during final machining.
2. The specimens were stress relieved in air at 1050 ± 10°F for four hours.
3. The specimens were final machined except for final width of sustain loaded specimens.
4. Aged at 1050 ± 10°F for four hours in air.
5. Ti-Brite cleaned to remove oxide film.
6. Electric discharge machined (EDM) semi-elliptical surface flaws.
7. Final machined width of test section for sustain load specimens.
8. Fatigue cracked specimens using maximum fatigue stress of 30 ksi and a stress ratio Min/Max = 0.06.
9. Specimens were final cleaned as follows:
   a. Turco Vitro-Klene 8-10 oz. per gallon of water at 200°F for 10 to 15 minutes. Air agitated constantly.
   b. Rinsed with distilled water.
   c. Water break tested.
   d. Dried with gaseous nitrogen.
   e. Wipe tested.

4.2 Weld Metal Specimen Fabrication

The specimen configuration used to evaluate weld metal properties is shown in Figure 2. The specimen thickness was selected as 0.062 inches, the thickness of the tank weld. Flaws were placed in both the weld centerline and weld heat affected zone (HAZ) as determined from etching the edge of the weld. The fabrication sequence used for welded specimens was as follows:

1. Weld half panels were rough machined per Figure 3.
2. Panels were stress relieved in air at 1050 ± 10°F for four hours.
3. Panels were finish machined per Figure 3.
4. Half panels were welded together by the Mariner Mars 1971 Propellant Tank supplier using the tungsten inert gas process in accordance with JPL Specification ES 504496A.
5. Weld panels were radiographed and penetrant inspected per ES 504496A.
6. The static specimens were machined to width and length, holes were drilled and weld bead removed. The same procedure was followed for sustain load specimens except excess of 0.10 inch was left on specimen width.
7. Aged at 1050 ± 10°F for four hours in air.
8. Ti-Brite cleaned to remove oxide film.
9. EDM semi-elliptical surface flaw.
10. Final machined width of test section for sustain load specimens.
11. Specimens were fatigue cracked using maximum fatigue stress of 30 ksi and a stress ratio Min/Max = .06.
12. Specimens were final cleaned as follows:
    a. Turco Vitro-Klene 8-10 oz. per gallon of water at 200°F for 10 to 15 minutes. Air agitated constantly.
    b. Rinsed with distilled water.
    c. Water break tested.
    d. Dried with gaseous nitrogen.
    e. Wipe tested.

4.3 Static and Flaw Growth Test Setups

The tensile and static fracture specimens were tested in 12,000 and 120,000 lb Baldwin universal testing machines. The specimens tested in a liquid nitrogen environment were enclosed by a cryostat as depicted in Figure 4. All static tests were performed in an environmentally controlled laboratory at the Boeing Space Center.

The sustained load test specimens were loaded in 10,000 lb. dead load creep machines as shown in Figure 5. In each of the six testing machines used, four specimens were mounted in series inside a thermally controlled enclosure. Small cups of the variety shown in Figure 6 were clamped onto each specimen. Under prior work [5], specimens were instrumented with strain gages to determine if the cup clamping force reduced the specimen stress. This reduction was found to be less than three percent. Flex lines were used to interconnect each of the small cups as indicated in Figure 7. The system was checked for leaks using helium gas. Repeated pressurization and venting with helium was performed to purge the system and then a vacuum was pulled on the system for about one-half hour, after which time nitrogen tetroxide, supplied at 30 psig, entered and filled the system. The temperature of the enclosure was brought up to 80 to 90°F by a coil heater and the load was applied to the specimens. The oxidizer was pressurized to 315 psig. These tests involving hazardous propellants were performed at Boeing's rmote Tulalip Test Site.

4.4 Experimental Approach for Static and Sustained Load Tests

Mechanical property specimens were tested at a strain rate of 0.004 inch/inch/min. while the static fracture specimens were tested at a load rate of 10,000 lb/min. For the static fracture specimens tested in liquid nitrogen, a thermocouple was used with the first specimen tested to determine the length of time required to reduce the temperature of the specimen to -318°F. Approximately four minutes were required to accomplish this and therefore a 10 minute soak period was estabd ished for the remaining specimens prior to actual testing.

The following approach was used in defining the threshold stress intensity of the base metal and weld metal specimens. After determining the apparent fracture toughness of the base metal specimens at room temperature, the first group of four specimens were sustained loaded to about 80 percent of the apparent toughness. Failure of one specimen occurred in approximately five minutes after reaching load. Two additional specimens, BI-1 and AS-10, were loaded to about 70 percent of the toughness. Specimen BI-1 failed in the grips (due to a steel stamp mark) immediately upon reaching load. The specimen was inspected after removal from the testing machine and the precracked surface flaw had grown through the thickness. Specimen AS-10 was reloaded to the same stress level it had been previously subjected to. After 22.5 hours, the specimen was removed and analyzed. Growth was also observed on this specimen. Based on this information, an additional group of four base metal specimens were loaded to approximately 65 percent of the apparent toughness. No failures occurred after 24 hours so two groups of four base metal specimens each were loaded to 65, 60 and 55 percent. All 24 specimens, six groups of four specimens each, remained intact under load for 1000 hours. After completion of the sustained load testing, all specimens were low stress cycled in air to mark the flaw front, and then pulled to failure. Evidence of sustained load growth was then observed by a separation between the initial fatigue crack extension and that of the final marking.

4.5 Fractographic Technique

The ease by which one determines whether or not sustained load flaw growth has taken place in a fracture specimen is greatly enhanced by use of fractographs of the fracture surface. The fractographic technique takes advantage of the difference in surface texture between fatigue growth zones and sustained load growth zones. The zones are made up of relative amounts of flat and obliquely oriented surfaces. Polarized light, directed normal to the fracture face, is reflected at different angles depending upon the surface orientation. Photographic film is exposed to the polarized light reflected normal to the fracture face capturing the contrast differences in the fracture surfaces. In some cases, the polarized light reflected to the camera lens is again polarized using a polarizing filter and then recorded on film. The fractographic technique is illustrated in Figure 8.

4.6 Stress Intensity Solution

Initial and critical stress intensity values for all specimens were calculated using the expression:

$$K_I = 1.1\,\sigma \left(\frac{\pi\ a}{Q}\right)^{1/2} M_K$$

where K_I = applied stress intensity

σ = gross stress

a = flaw depth

Q = shape parameter

M_K = Kobayashi's deep flaw magnification factor

Values for Q and M_K are shown in Figures 9 and 10. Specimen width was maintained at approximately ten times the flaw length.

5.0 TEST RESULTS

5.1 Mechanical Properties

Mechanical property tests were conducted at room temperature for both heats of the base metal and weld metal. The values obtained from each specimen are shown in Table III. A summary presenting the average 0.2 percent yield strengths (ksi) of the axially oriented specimen is given below:

MATERIAL	HEAT G-804	HEAT 301275
Base Metal	149.0	156.5
Weld Metal	133.3	137.9

For yield strength at -320°F, values of 148 percent of the room temperature values were used based on the data presented in Reference 6.

5.2 Fracture Tests

Static fracture tests were performed on the base metal and weld metal specimens at room temperature in air and at -320°F in liquid nitrogen. All base metal and weld metal calculated specimen toughnesses are presented in Tables IV and V. A summary of the fracture toughness averages (ksi $\sqrt{in}$) is presented below:

TEMPERATURE	HEAT NUMBER	BASE METAL	WELD METAL	
			℄	HAZ
R.T.	G-804	45.1 [1]	46.7	48.5
	301275	44.9 [1] (55.0)	47.0	48.2
-320°F	G-804	55.8	46.0 [2]	38.0
	301275	46.3	38.8	32.1

[1] Not plane strain values; specimens failed as thru-the-thickness cracks.

[2] Based on a specimen with an incomplete initial flaw.

Growth of the flaw during loading was observed with the thin (representative of the tank wall - 0.033 inches thick) base metal specimens tested at room temperature. The flaw front broke through the back side of the specimen prior to failure. The stress levels at flaw break through and failure were recorded and apparent fracture toughnesses were calculated, realizing that these values were affected by the flaw extension. The calculated thru-the-thickness fracture toughness was only a few percent less than the calculated failure fracture toughness. Both heats demonstrated about the same average calculated failure fracture toughness of 45 ksi $\sqrt{in}$. In order to obtain a true plane strain fracture toughness for the base metal, two thick (0.153 inches thick) specimens from heat 301275 were tested. An average value of 55.0 ksi $\sqrt{in}$ was obtained.

The liquid nitrogen base metal tests yielded average fracture toughnesses of 55.8 and 46.3 ksi $\sqrt{in}$ for heat G-804 and 301275, respectively. These values are thought to be of plane strain in nature since the plastic zone size at -320°F is less than the remaining ligament between the crack tip and back side of the specimen.

The weld metal specimens were representatives of the tank welds (0.062 inches thick) and yielded plane strain fracture toughness values. Both heats tested, with either flaws located on the weld centerline or in the weld HAZ demonstrated virtually the same fracture toughnesses at room temperature, ranging from 46.0 to 51.1 ksi $\sqrt{in}$. An average value of 47.6 ksi $\sqrt{in}$ was obtained.

A greater amount of scatter was observed with the liquid nitrogen tests than the room temperature tests. Heat G-804 demonstrated fracture toughnesses of 46.0 and 38.0 ksi $\sqrt{in}$ in the weld centerline and HAZ, respectively. The value obtained for the weld centerline is slightly elevated due to incomplete flaw extension during initial fatigue pre-cracking. Heat 301275 demonstrated fracture toughness of 38 8 and 32.1 ksi $\sqrt{in}$ in the weld centerline and HAZ, respectively.

5.3 Sustained Load Flaw Growth

The results of the sustained load tests for two different heats of base metal and weld metal exposed to nitrogen tetroxide are presented in Figures 11 through 14. A summary of the sustained load thresholds is tabulated below:

MATERIAL	HEAT NUMBER	LOWER BOUND THRESHOLD STRESS INTENSITY(KSI $\sqrt{IN}$)
Base Metal	G-804	30.0
	301275	28.0
Weld Metal	G-804	32.0
	301275	31.0

These thresholds represent a lower limit at which the threshold could exist while in actuality the threshold might be higher. As indicated by Figures 11 through 14, no specimen failures were observed for specimens loaded for 1000 hours. There were some base metal specimens on which growth was observed, but it is believed that this growth is due to loading as discussed in paragraph 5.4. A total of 30 specimens were used in establishing these lower bound thresholds.

Tables VI and VII present the sustained load test results for each individual specimen tested.

5.4 Flaw Growth During Loading

Tests were conducted on four specimens to determine if any flaw growth occurred during loading. These specimens were loaded at a rate of about 10,000 lb/min. and upon reaching a predetermined stress level the load was dropped immediately to zero. The specimens were then fatique marked and then were pulled to failure. The results of these tests are shown in Table VIII. The thickness of these specimens were selected such that ρ/t_n values were comparable to that of the base metal specimens tested during sustained loading. Flaw growth on the order of magnitude as that observed in some of the base metal sustained load tests was obtained. These specimens were from the same material that had shown no growth on loading when tested in thicker sections, see Table VII. Fractographs showing the fracture face of these four specimens are shown in Figure 15.

6.0 DATA ANALYSIS

6.1 Static Fracture Behavior

The influence of material heat, temperature and flaw location on fracture toughness was determined. The flaw locations included base metal and weld metal centerline and HAZ.

For the liquid nitrogen tests of base metal and weld metal, the G-804 heat demonstrated a higher fracture toughness than the 301275 heat. This increase amounted to approximately 20 percent. The base metal fracture toughness was observed to decrease with decreasing temperature. In the case of heat 301275, a 17 percent reduction occurred when tested at -320°F as compared to room temperature tests. No definitive statement can be made concerning heat G-804 since the plane strain fracture toughness was not determined for this heat at room temperature. The trend was also to decrease the fracture toughness of the weld metal with decreasing temperature. The toughness values obtained for centerline and HAZ flawed specimens showed a reduction of between 20 and 33 percent, depending upon the material heat.

The room temperature plane strain fracture toughness was about 15 percent higher for the base metal specimens of heat 301275 as compared to the weld metal specimens with either centerline or HAZ located flaws. At the liquid nitrogen temperature of -320°F, the base metal again exhibited higher toughness than the weld metal. The increase ranged from 22 to 74 percent, depending upon the material heat and weld metal flaw location. The HAZ appears to have the lowest fracture toughness.

6.2 Threshold Stress Intensity Behavior

The results of the 1000 hour sustained load tests in nitrogen tetroxide indicate that the threshold of the base metal and weld metal was above 28.0 ksi $\sqrt{in}$, regardless of material heat. Selected sustained load specimen fractographs are presented in Figures 16 and 17.

Use of the data generated in this program can best be illustrated as in Figure 18. The figure shows critical and reference threshold curves for 0.033 inch base metal of heat G-804. For simplicity, the curves are constructed in terms of gross stress versus flaw depth, a. (Note that this is valid for a given a/2c value; in this case, a value identical to that tested, is used). Most direct use of the data shown in figure can be made without consideration of actual K values. That is, by observation of the -320°F static data points, it is seen that flaws deeper than about 0.018 inches would be screened by the contemplated proof test at 200 ksi membrane stress. Secondly, observe the six sustain loaded data points. These also had initial flaw depths on the order of 0.018 inches, and did not fail during the 1000 hour tests. The maximum stress tested in this group of specimens was about 104 ksi. After a cryogenic proof at 200 ksi stress, an operating stress approaching 104 ksi, then, is the maximum allowable stress proven by this set of data.

7.0 CONCLUSIONS

1. Fracture Toughness

   a) Heat G-804 has a higher fracture toughness than heat 301275 for base metal and weld metal tested at room temperature and -320°F in liquid nitrogen.

   b) The fracture toughness is higher for base metal and weld metal specimens of both heats tested at room temperature than at -320°F in liquid nitrogen.

   c) The fracture toughness of the base metal is higher than the weld metal specimens when tested at room temperature and -320°F, regardless of material heat. The weld HAZ exhibited the lowest fracture toughness.

2. Threshold

   The threshold of the base metal and weld metal, when exposed to a 80-90°F, 315 psig nitrogen tetroxide (0.60 min. nitric oxide content) environment, is above 28.0 ksi $\sqrt{in}$. Significant amounts of growth were observed at stress intensity levels above 28.0 ksi $\sqrt{in}$, but it is not believed that this growth was environmentally assisted.

   Normally, the threshold of a material, environment combined is presented as a percentage of the fracture toughness rather than as an absolute stress intensity value as presented in this report. The use of thin material, simulating the actual pressure vessel thickness, prevented determining the forging plane strain fracture toughness at room temperature and therefore an absolute stress intensity is presented.

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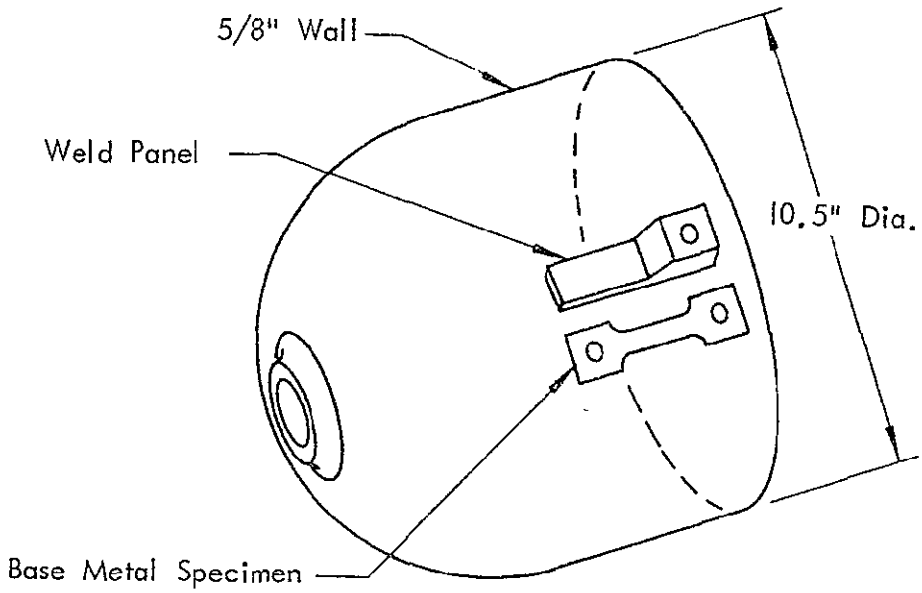
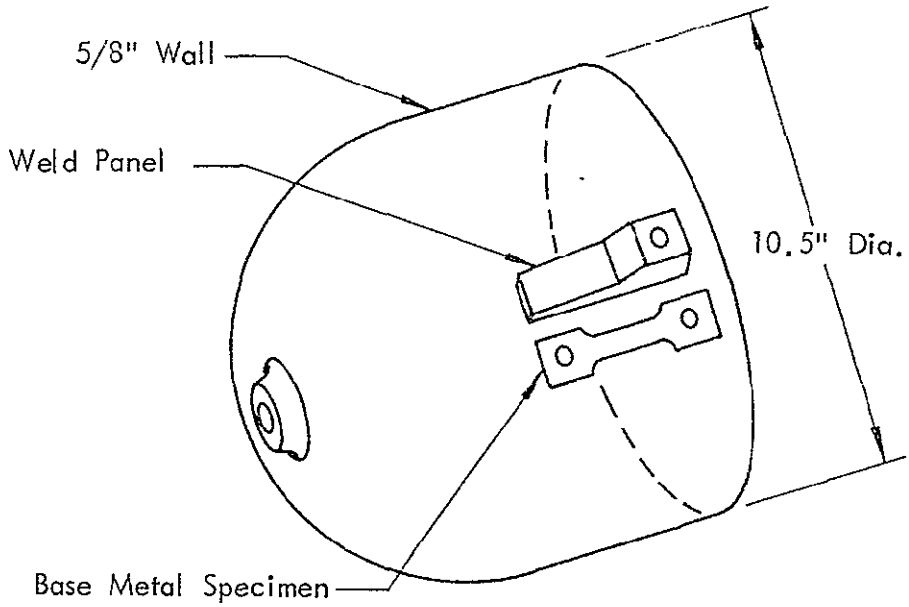
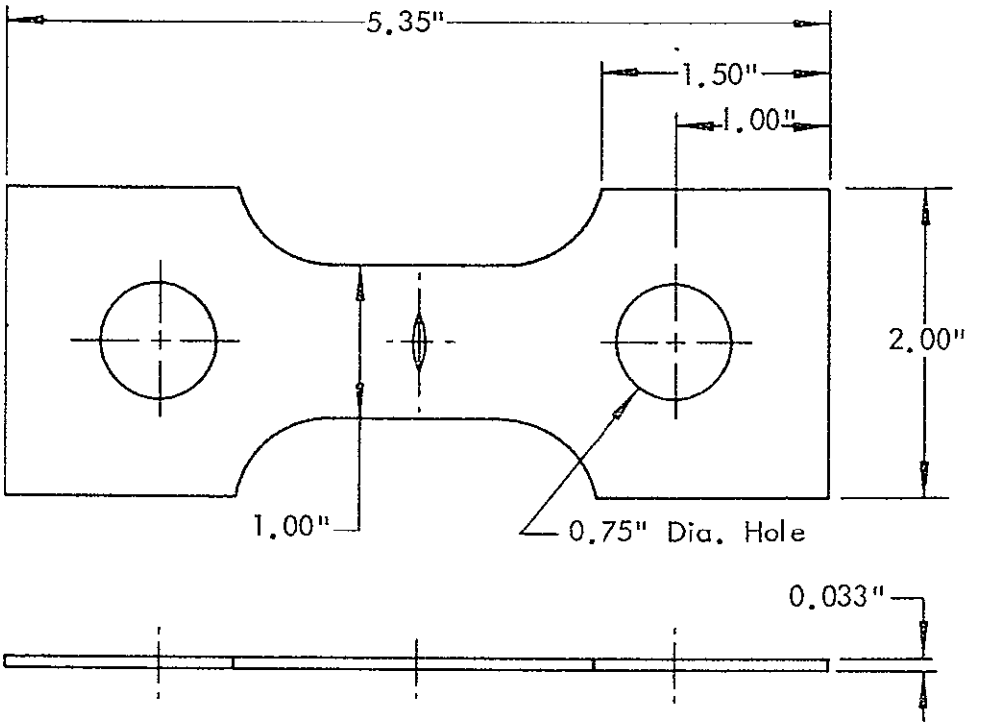


SERIAL NUMBERS 236, 238, 252 & 256

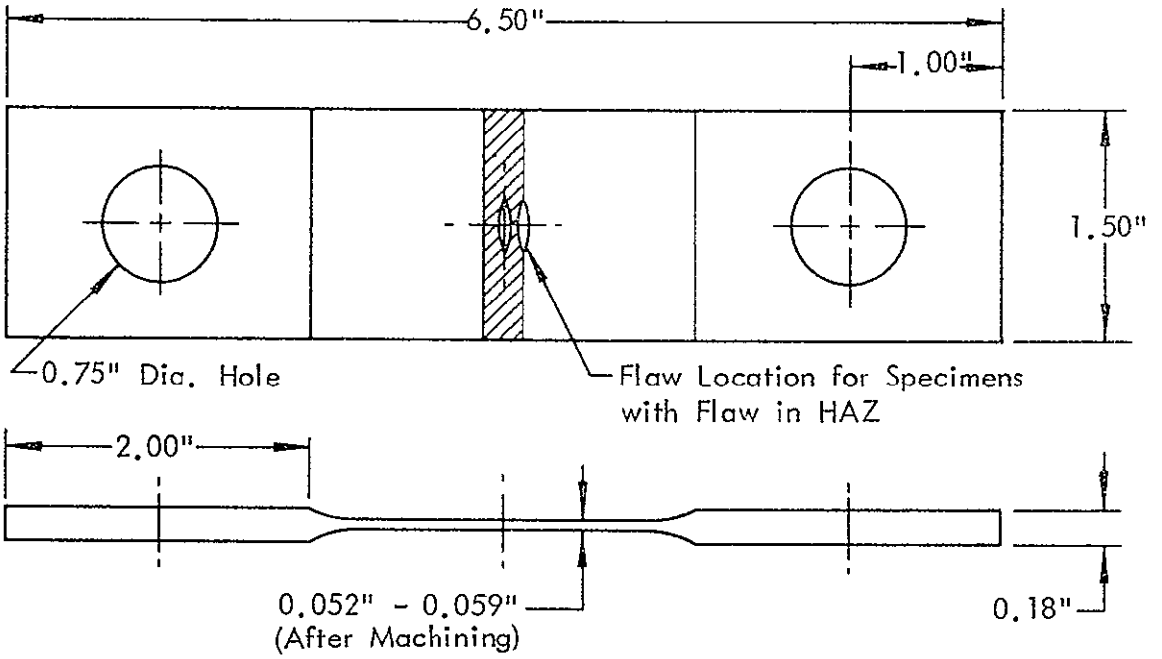


SERIAL NUMBERS 222, 229, 263 & 265

Figure 1 : SPECIMEN LOCATION AND ORIENTATION



BASE METAL



WELD METAL

Figure 2 : FRACTURE SPECIMEN CONFIGURATIONS

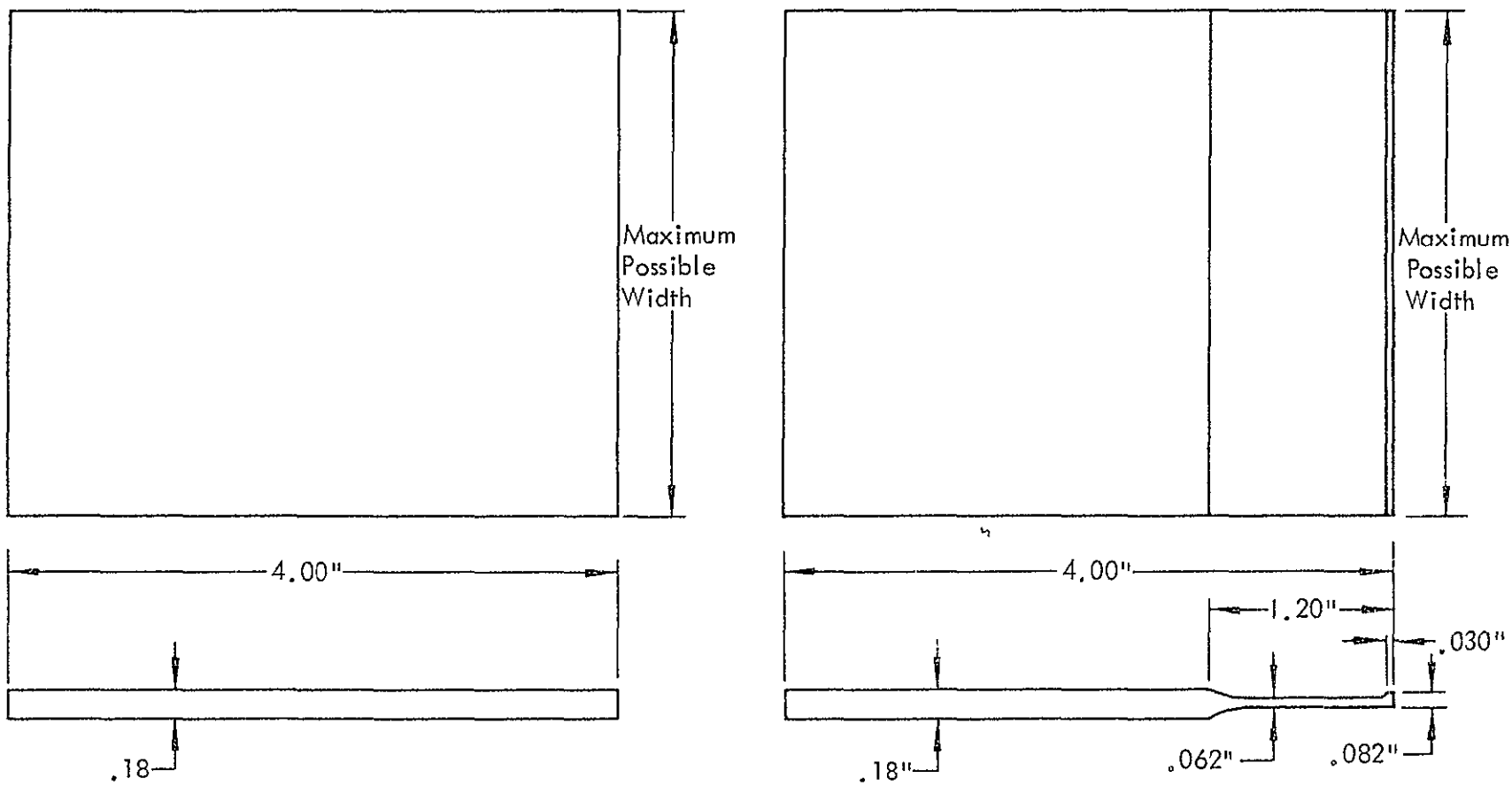


Figure 3: WELD PANEL CONFIGURATION

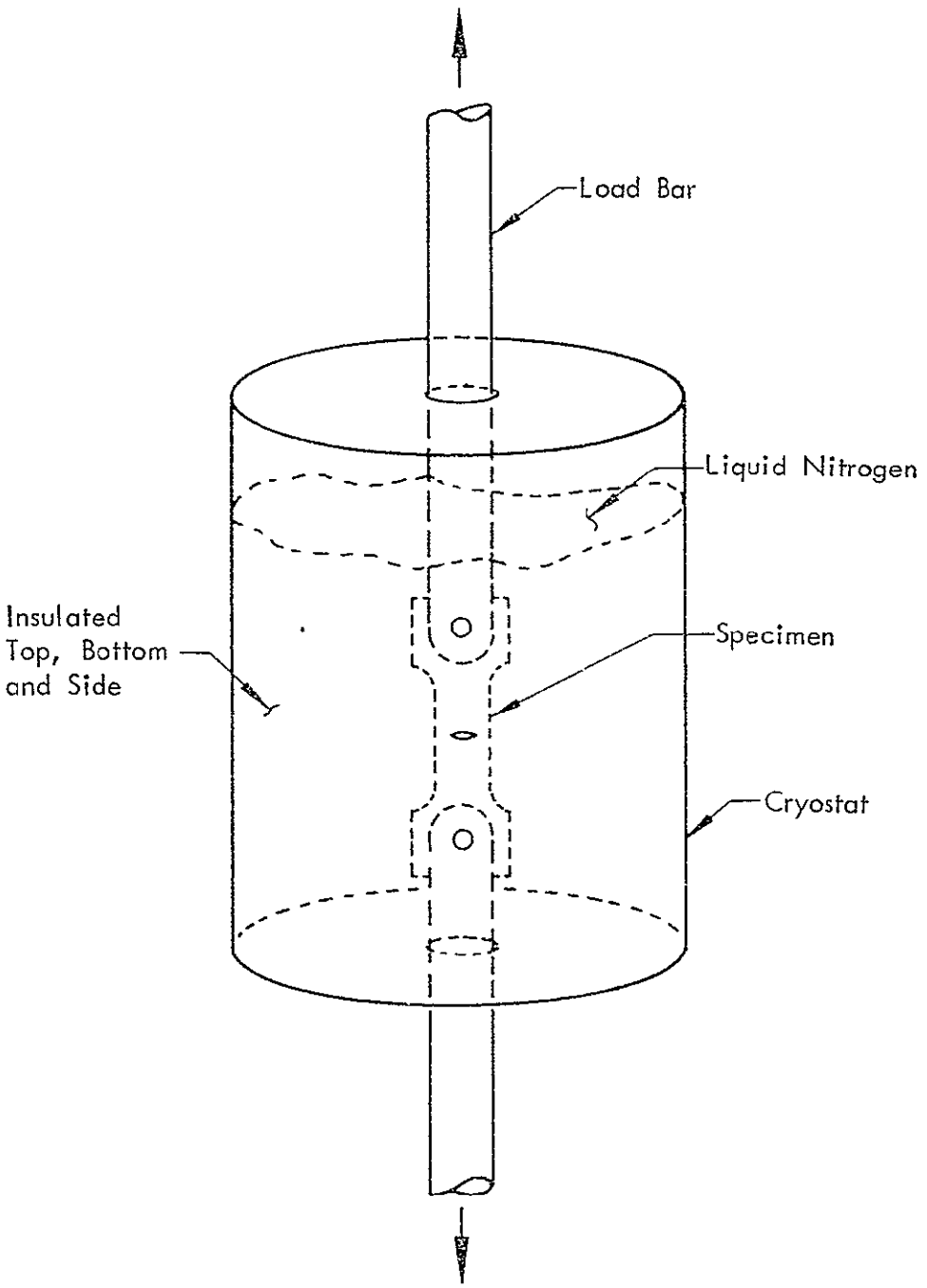


Figure 4 : LIQUID NITROGEN TEST SETUP

610-98

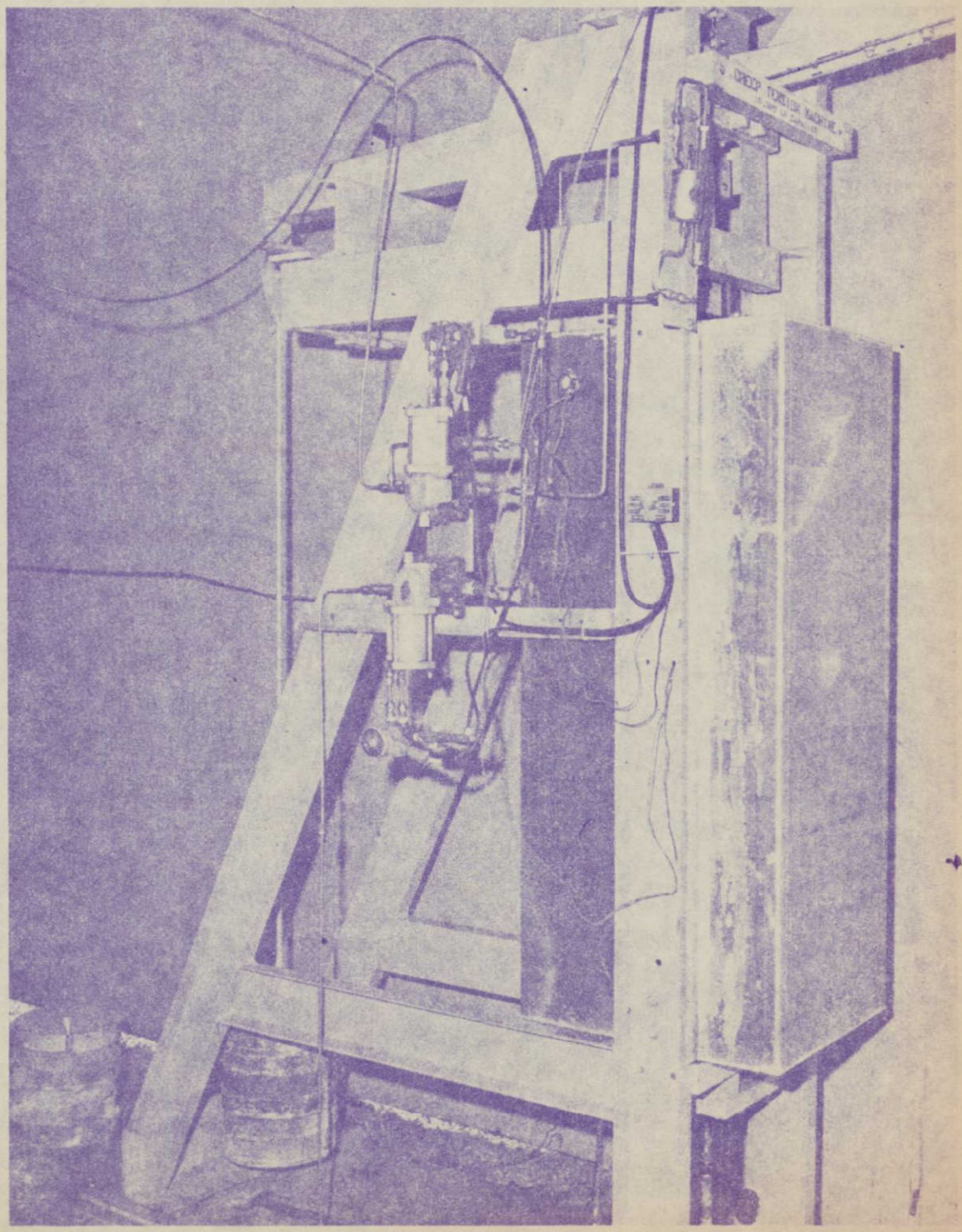


Figure 5 : SUSTAINED LOAD TEST SETUP - OVERALL VIEW

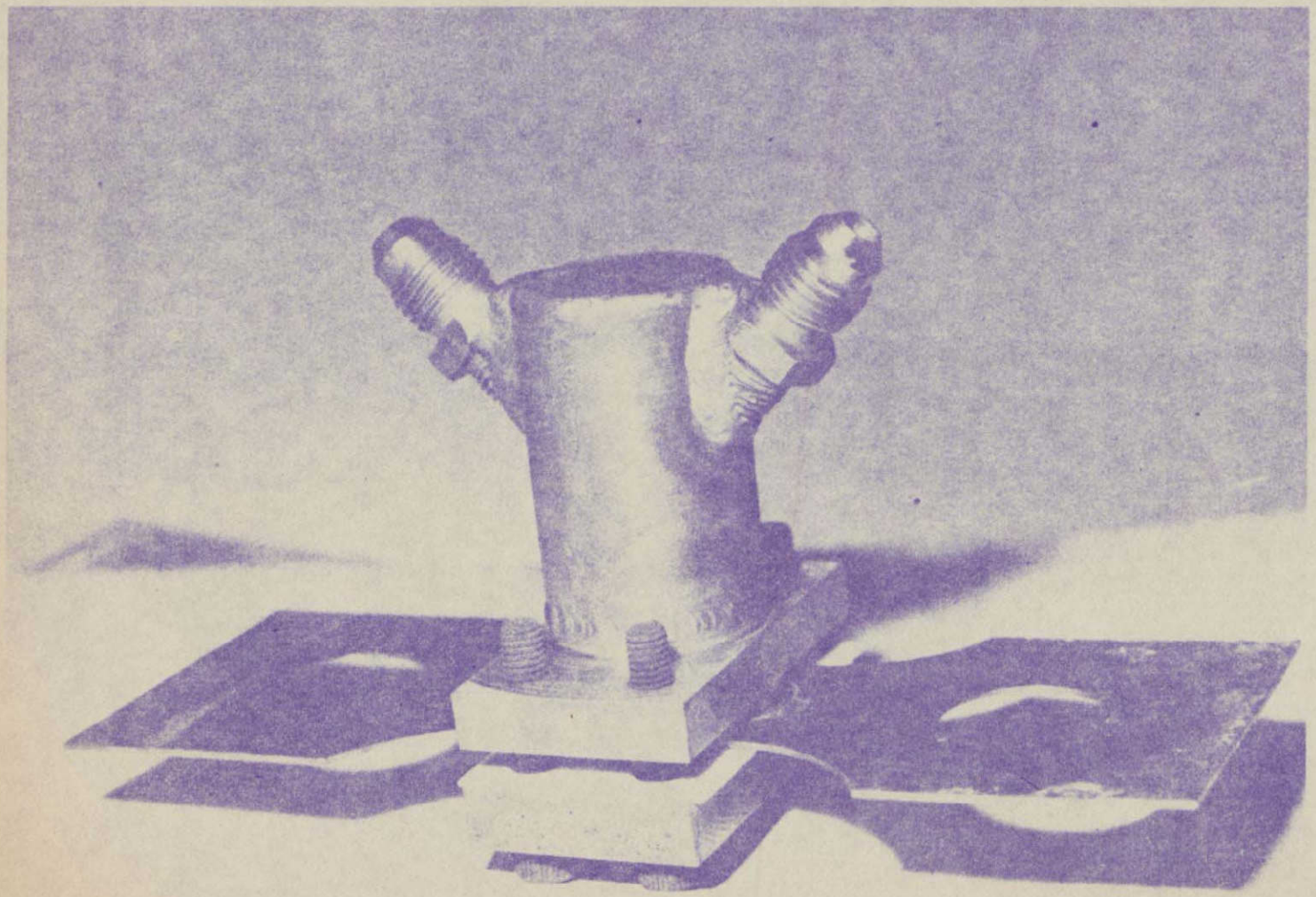


Figure 6 : SUSTAINED LOAD TEST SETUP - CUP MOUNTING DETAIL

610-98

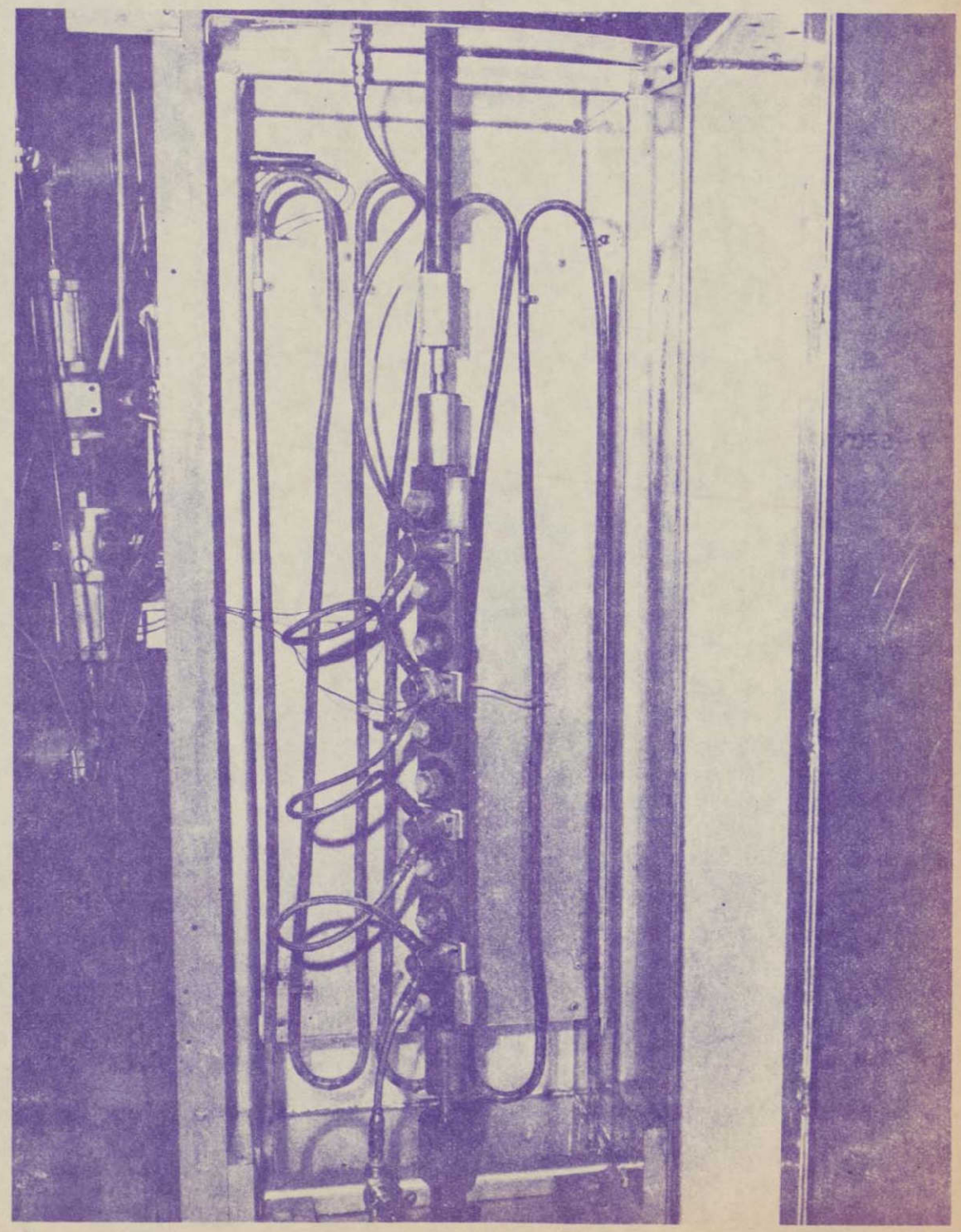


Figure 7: SUSTAINED LOAD TEST SETUP - SPECIMEN MOUNTING ARRANGEMENT

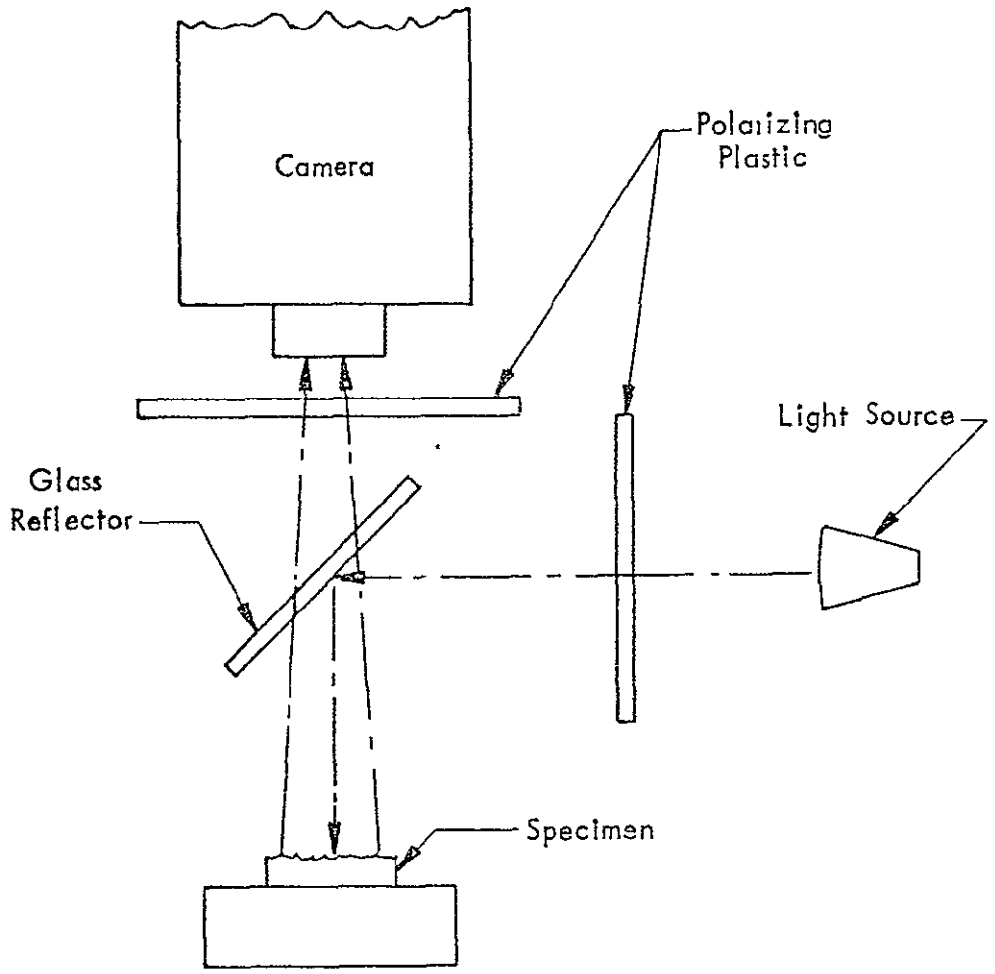


Figure 8: SCHEMATIC ILLUSTRATION OF FRACTOGRAPHIC SETUP

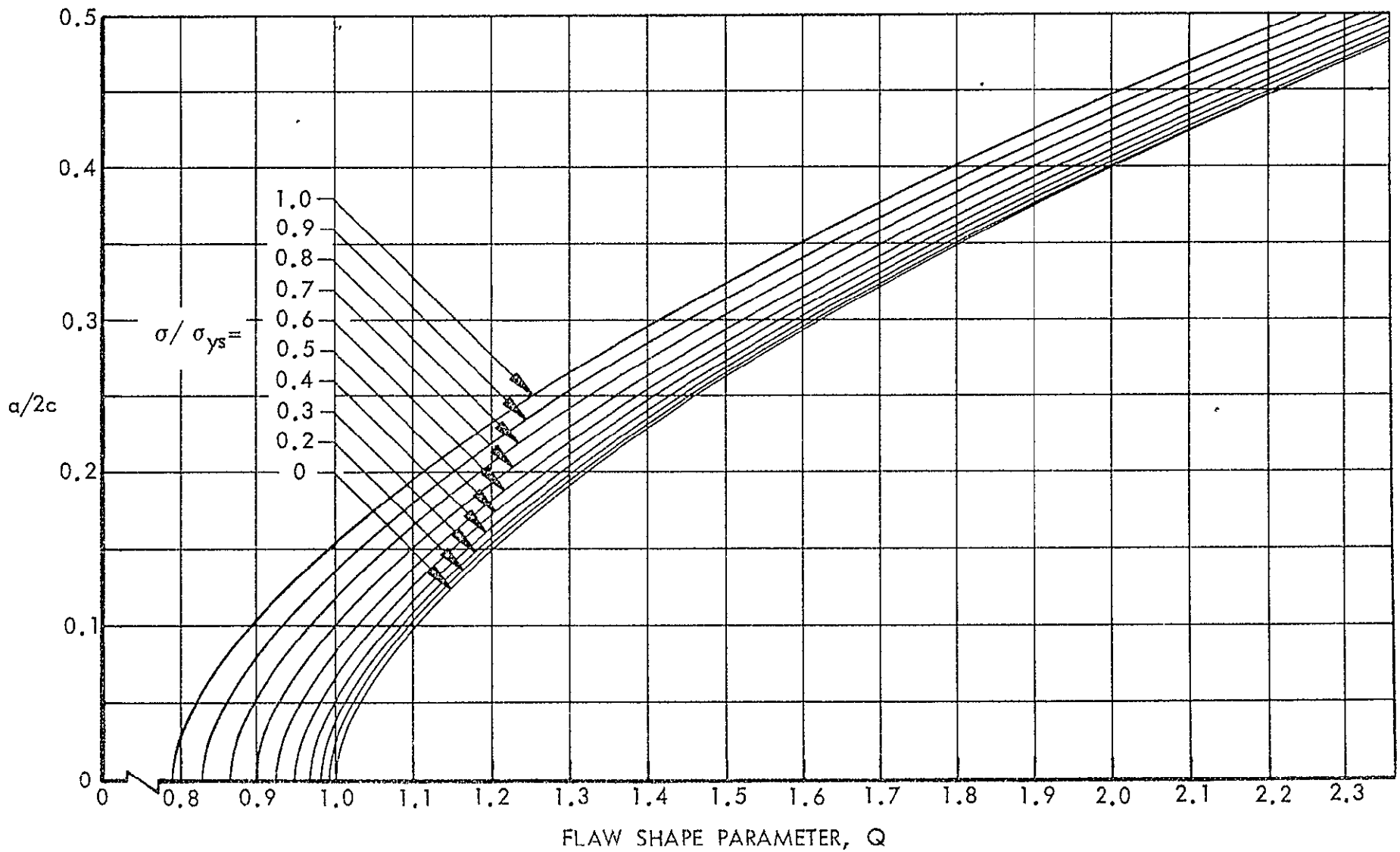


Figure 9: SHAPE PARAMETER CURVES FOR SURFACE AND INTERNAL FLAWS

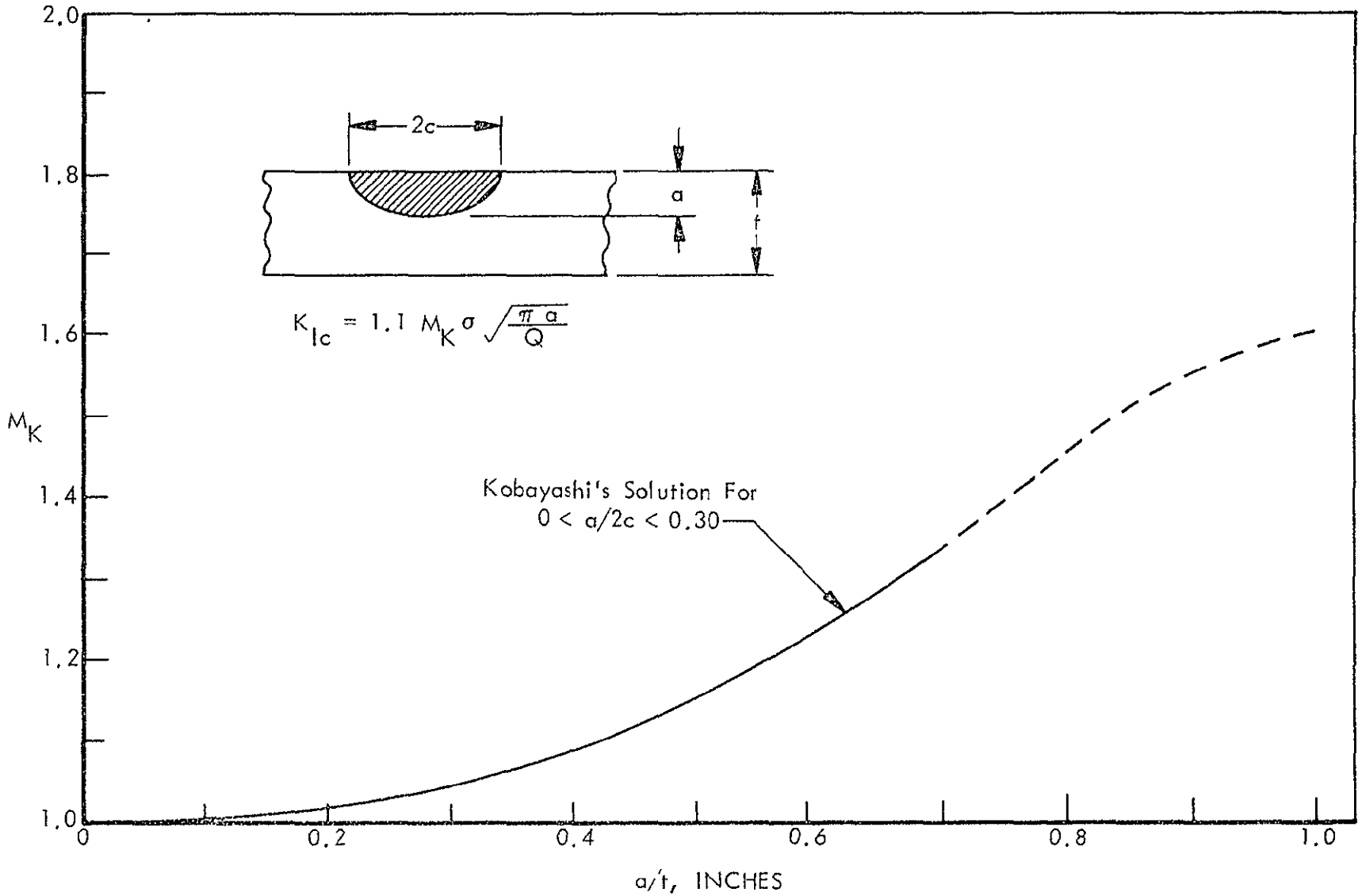


Figure 10: THE EFFECT OF FLAW DEPTH ON STRESS INTENSITY
(Thick Sections)

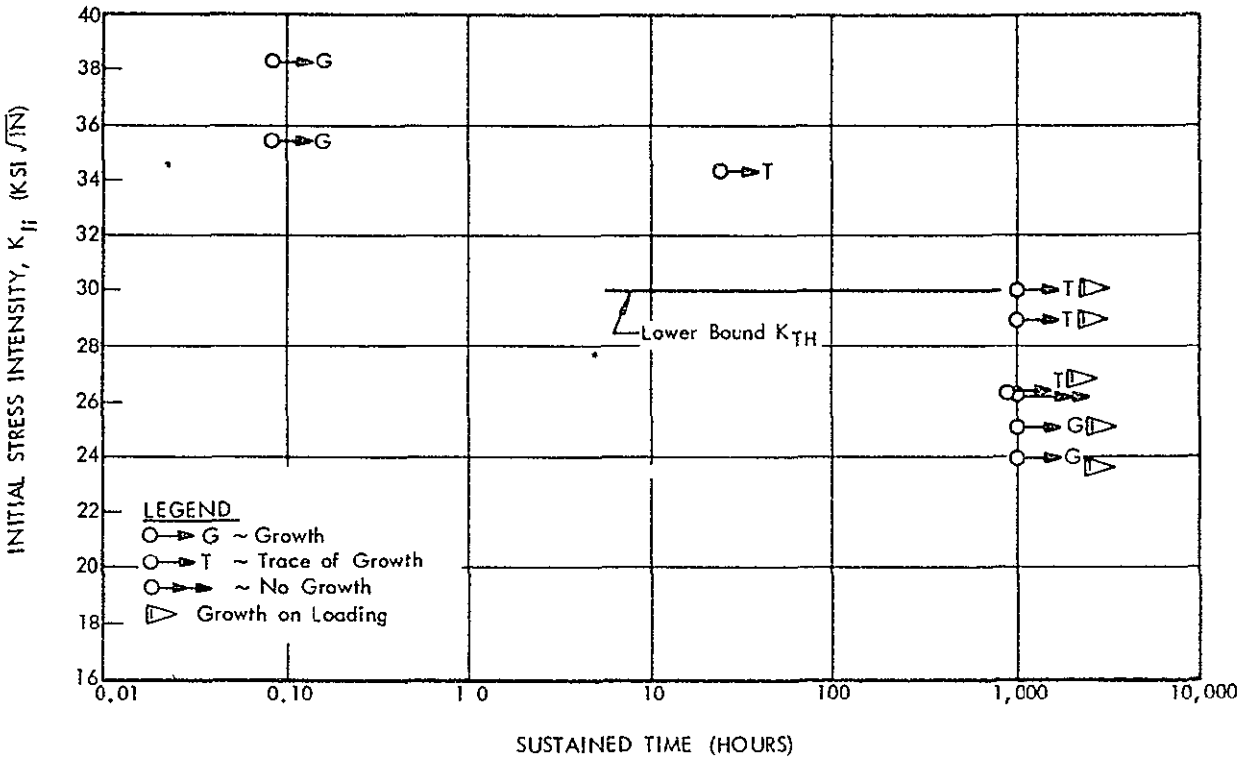


Figure 11: BASE METAL (Heat G-804) SUSTAINED LOAD FLAW GROWTH IN NITROGEN TETROXIDE

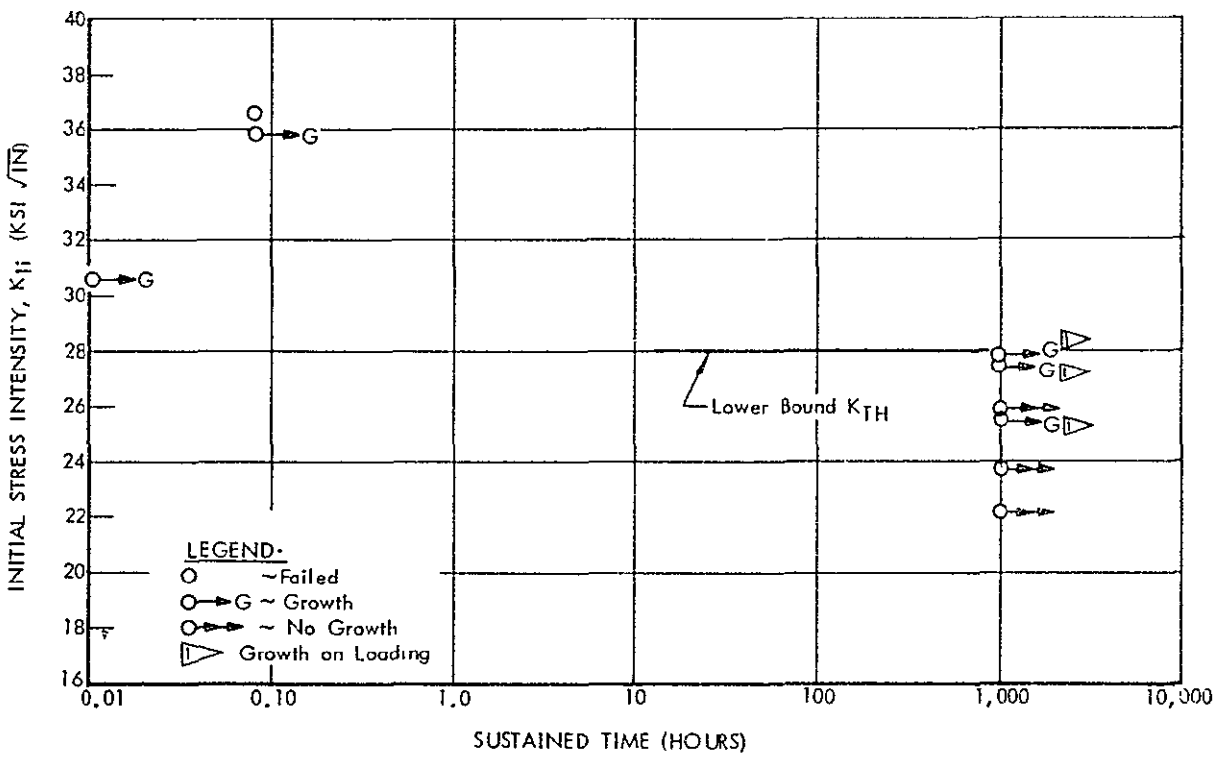


Figure 12: BASE METAL (Heat 301275) SUSTAINED LOAD FLAW GROWTH IN NITROGEN TETROXIDE

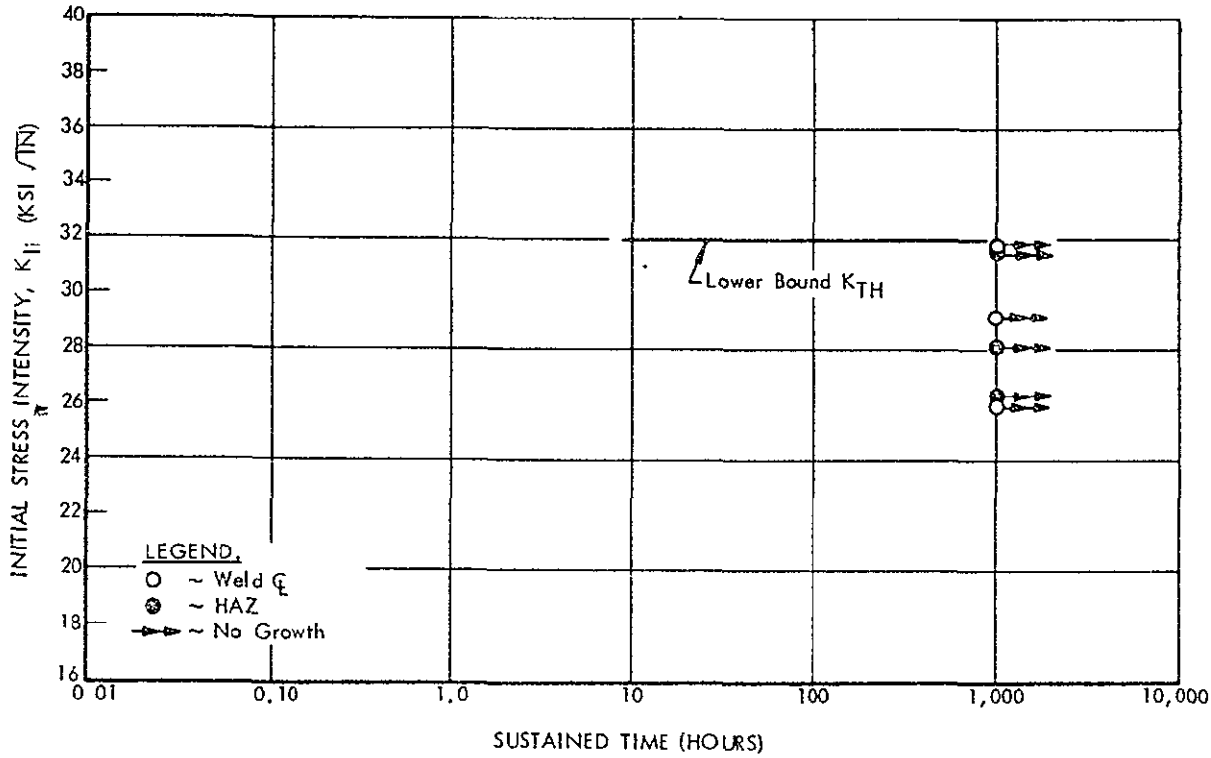


Figure 13: WELD METAL (Heat G-804) SUSTAINED LOAD FLAW GROWTH IN NITROGEN TETROXIDE

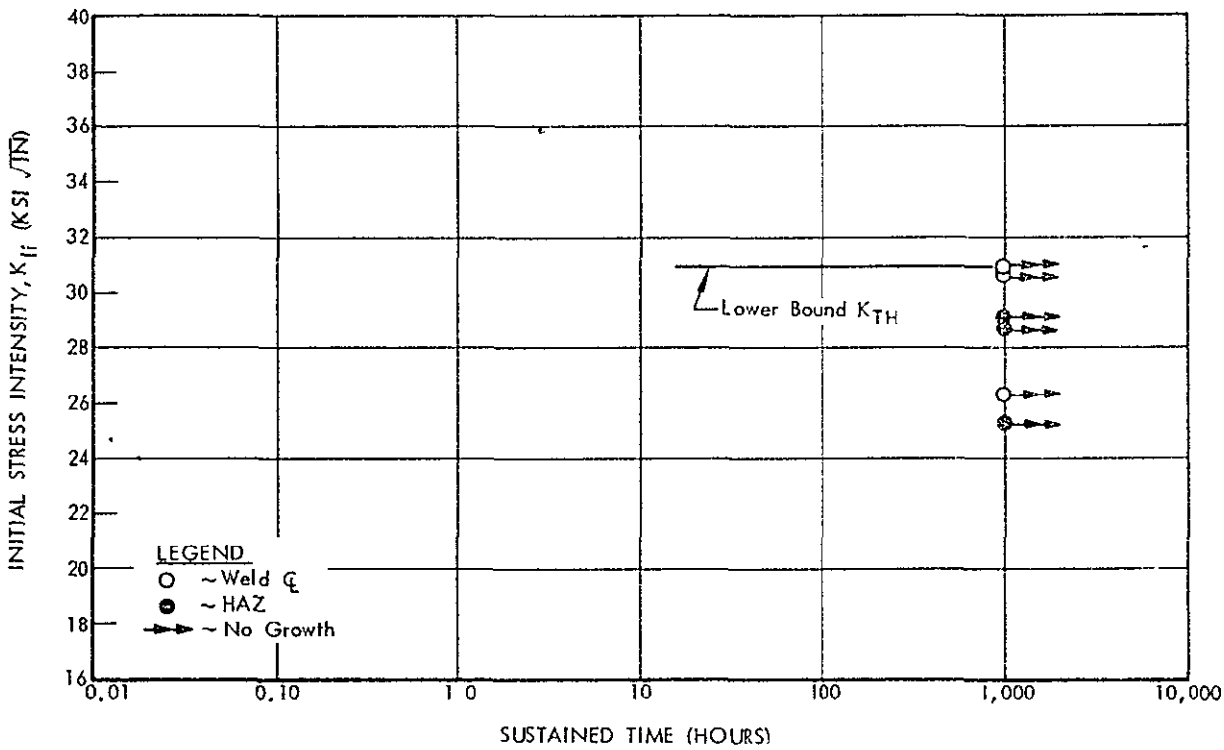
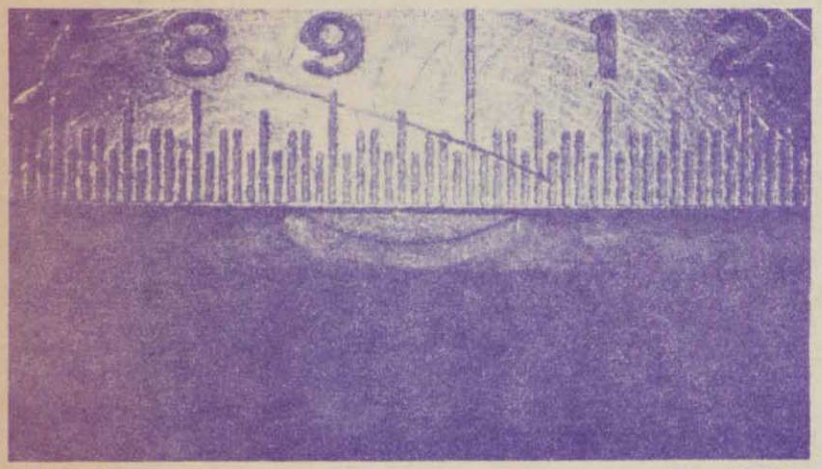
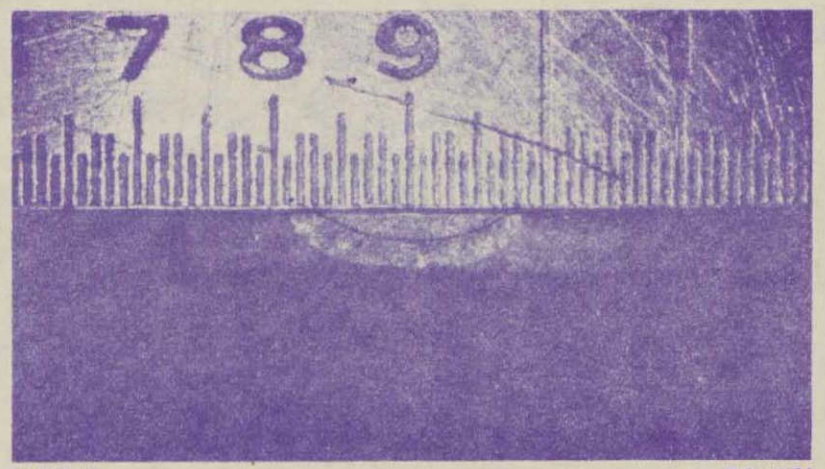


Figure 14: WELD METAL (Heat 301275) SUSTAINED LOAD FLAW GROWTH IN NITROGEN TETROXIDE



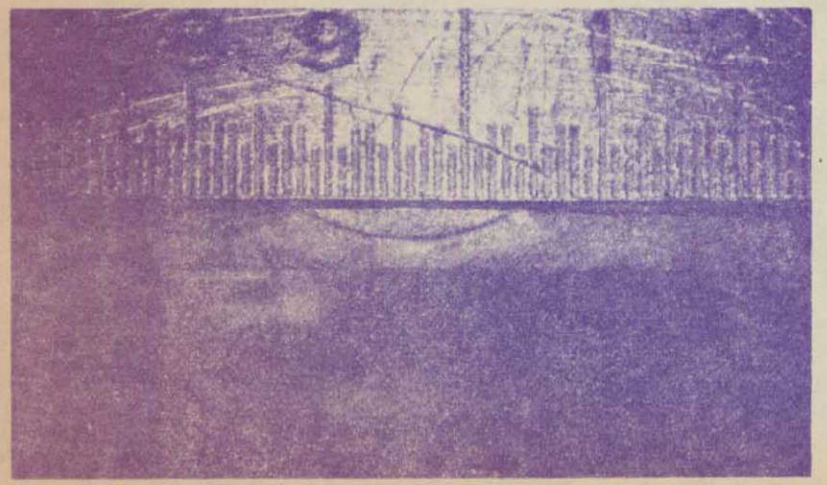
BWS-7 $\sigma = 60$ KSI

NO GROWTH



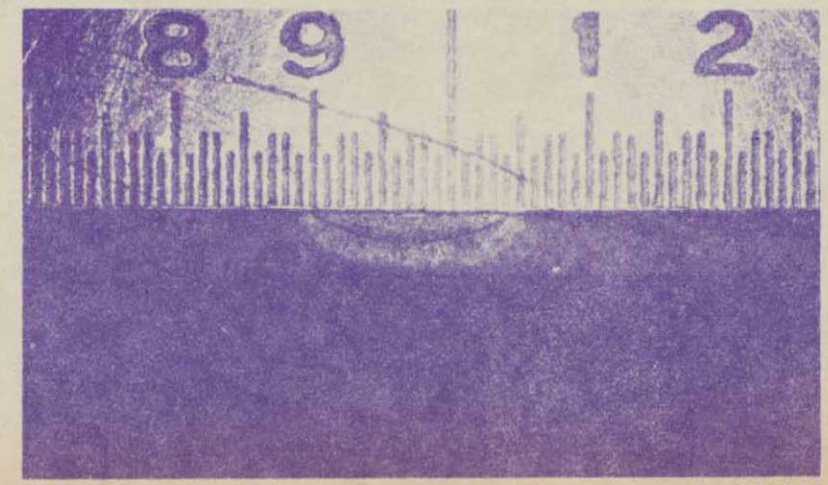
BWS-8 $\sigma = 70$ KSI

NO GROWTH



A1WS-7 $\sigma = 80$ KSI

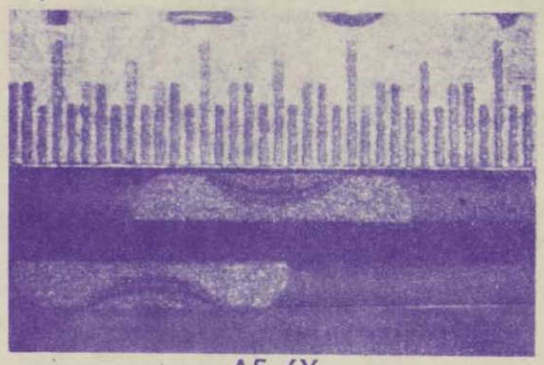
GROWTH



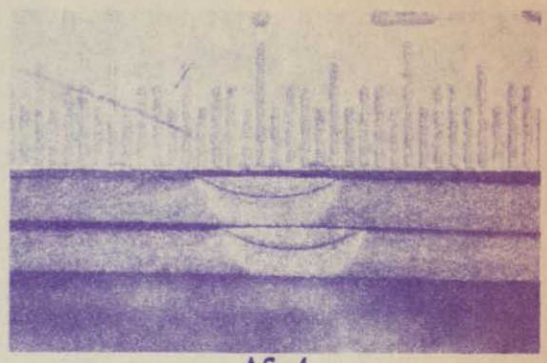
A1WS-8 $\sigma = 90$ KSI

TRACE OF GROWTH

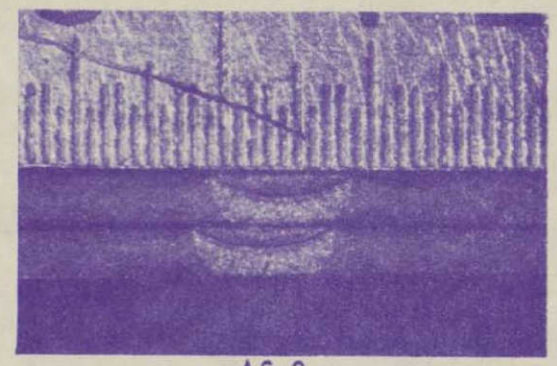
Figure 15 : FRACTOGRAPHS OF SPECIMENS USED TO DETERMINE THE EXTENT OF FLAW GROWTH DURING LOADING



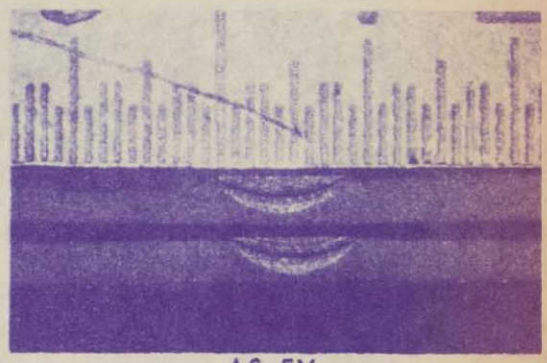
AS-6X



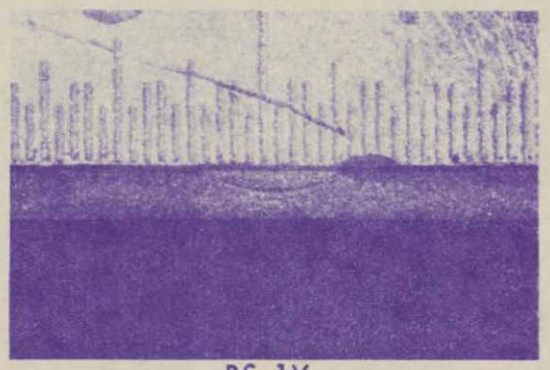
AS-4



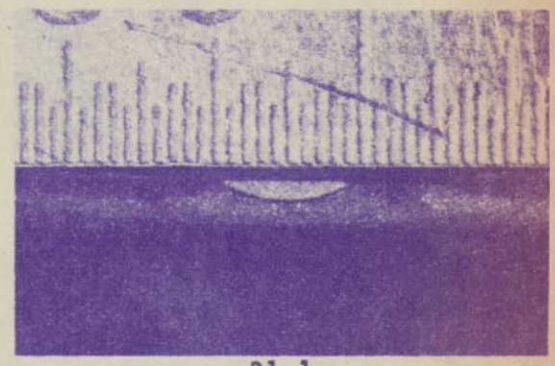
AS-2



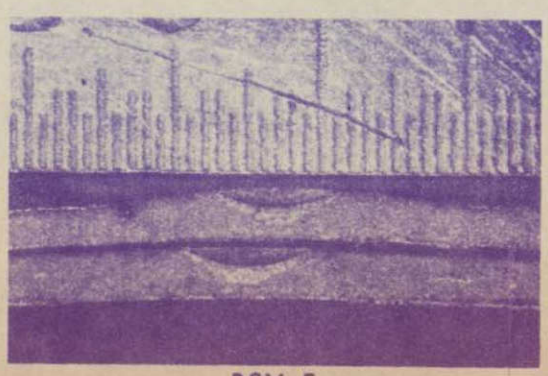
AS-5X



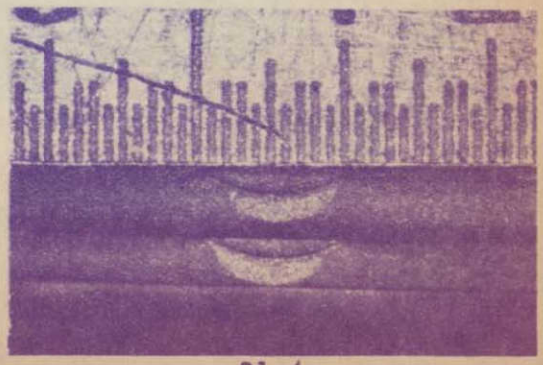
BS-1X



B1-1



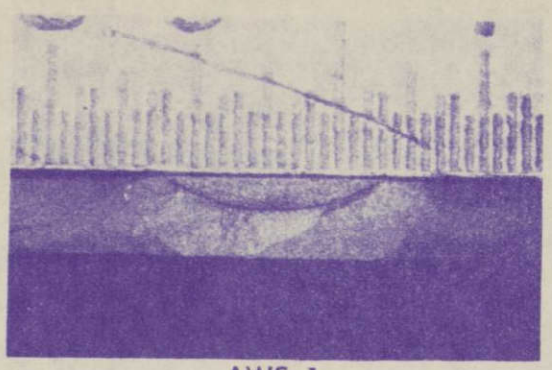
BSX-5



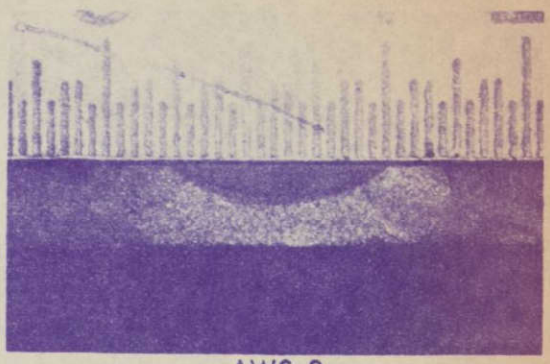
B1-4

Figure 16: FRACTOGRAPHS OF BASE METAL SUSTAIN LOADED SPECIMENS IN NITROGEN TETROXIDE (Polarized Light)

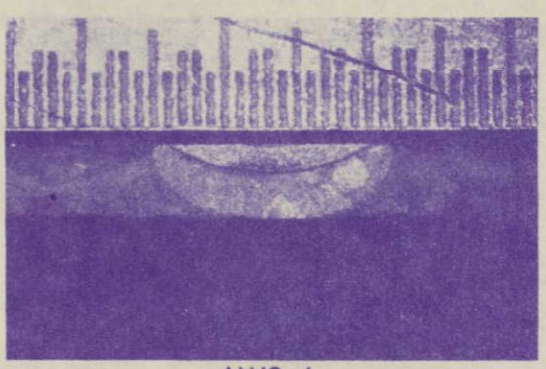
610-58



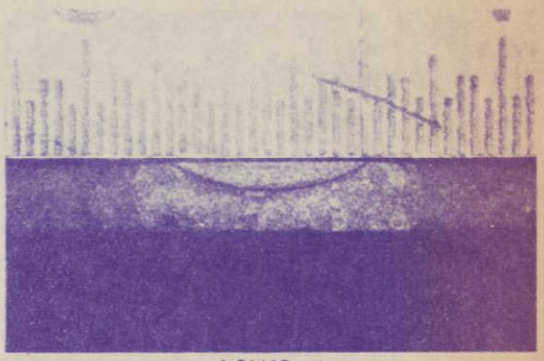
AWS-1



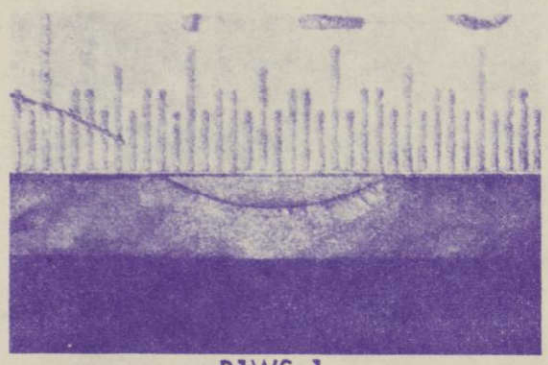
AWS-3



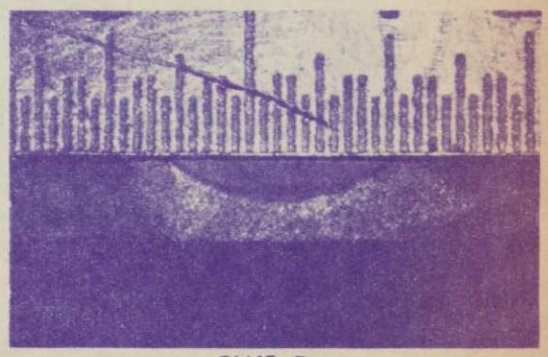
AWS-4



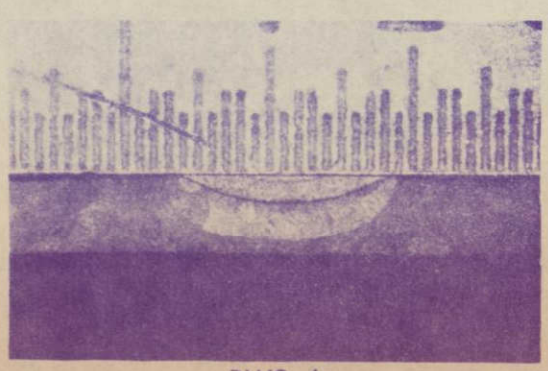
A1WS-6



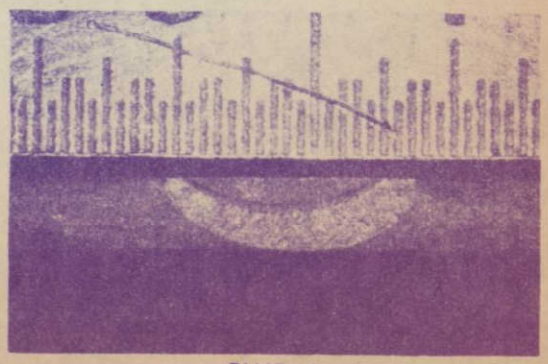
B1WS-1



BWS-3



BWS-4



BWS-6

Figure 17: FRACTOGRAPHS OF WELD METAL SUSTAIN LOADED SPECIMENS IN NITROGEN TETROXIDE (Polarized Light)

30

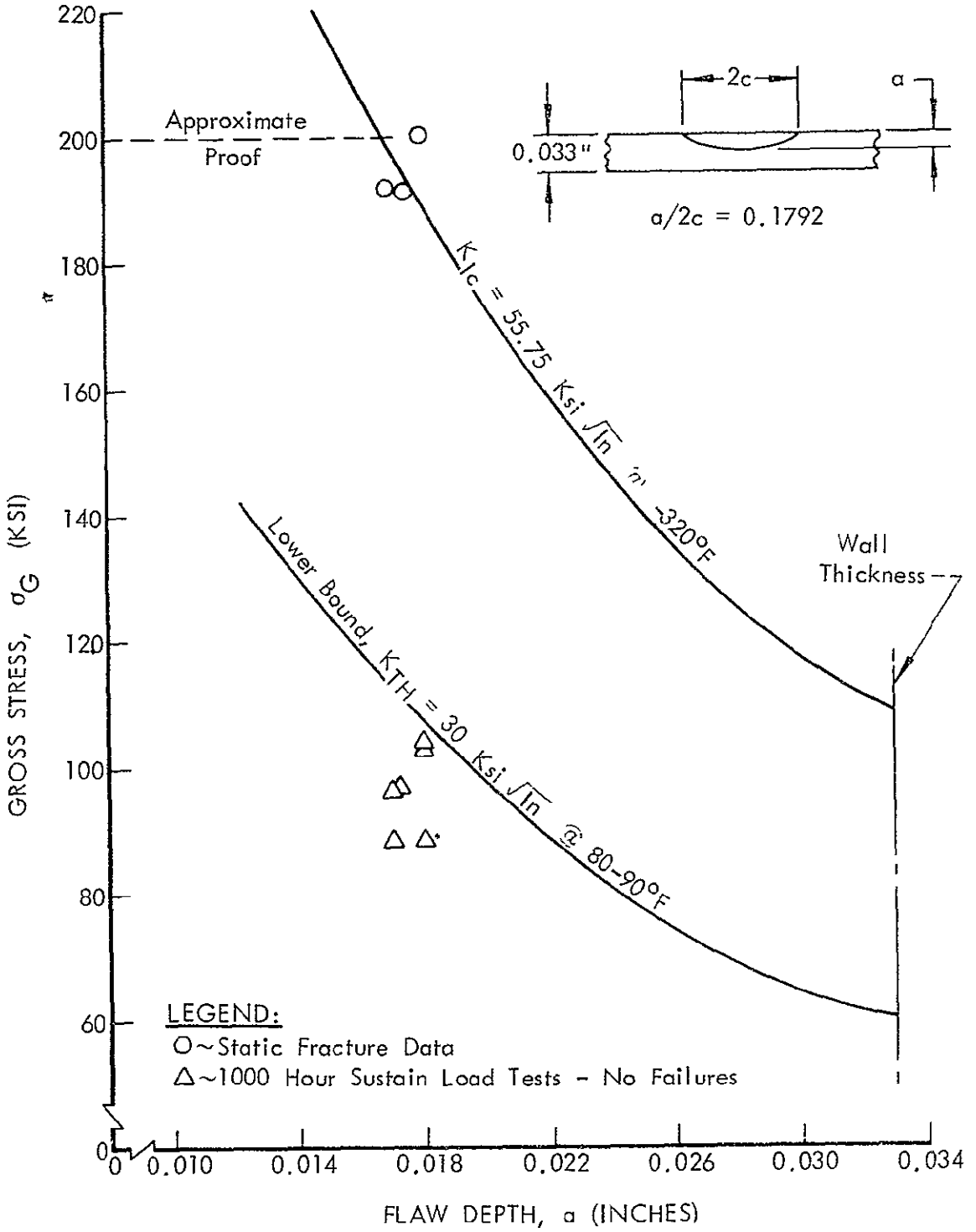


Figure 18: CRITICAL AND THRESHOLD FLAW DEPTH CURVES

Table I: CHEMICAL COMPOSITION (% By Weight)[1]

HEAT NUMBER	AL	V	Fe	O_2 [2]	C	N_2	H_2
G-804	6.3	4.3	0.16	0.162	0.022	0.008	0.0110
301275	6.3	4.3	0.14	0.176	0.024	0.008	0.0116

[1] Obtained From TMCA
[2] Exceeds Limitation Of JPL Specification ES504496A

Table II: COMPOSITION OF TEST FLUID (% By Weight)

COMPONENT	SPECIFICATION LIMIT	ANALYZED PRIOR TO 1000 HR TEST	ANALYZED AFTER 1000 HR TEST		
			SAMPLE #1 [1]	SAMPLE #3 [2]	SAMPLE #6 [3]
Nitrogen Tetroxide (N_2O_4)	98.7 min.	98.9	99.2	98.9	99.1
Nitric Oxide (NO)	0.80 ± 0.20	0.64	0.44	0.29	0.63
Water (H_2O)	0.10 max.	0.03	0.02	0.03	0.04
Chloride As Nitrosylchloride (NOCL)	0.08 max.	0.01	0.02	0.02	0.02
Particulate Matter	10 mg / l	2.4 mg / l	Not Determined		

[1] Contained Test Specimens AS-4, AS-9, BS-4 and BSX-5
[2] Contained Test Specimens AS-2, AS-3, B1-2 and B1-4
[3] Contained Test Specimens AWS-3, A1WS-6, BWS-3 and BWS-6

Table III: MECHANICAL PROPERTIES AT ROOM TEMPERATURE

MATERIAL	HEAT NUMBER	SPECIMEN ORIENTATION	SPECIMEN NUMBER	0.2% OFFSET YIELD STRENGTH (Ksi)	ULTIMATE TENSILE STRENGTH (Ksi)	% ELONGATION		% AREA REDUCTION
Base Metal [1]	G-804	Hoop Direction	S/N 114-1	161.0	183.0 [3]	13	In 1.0 Inch	45
			S/N 114-4	161.0	179.0 [3]	13		46
		Axial Direction	S/N 114-2	154.0	174.0	16		50
			S/N 114-3	144.0 [3]	164.0	16		52
		Boss Area	S/N 114-5	154.0	173.0	15		56
			S/N 114-6	146.0 [3]	164.0	16		50
	301275	Hoop Direction	S/N 270-1	160.0	177.0 [3]	15		53
			S/N 270-4	160.0	180.0 [3]	14		51
		Axial Direction	S/N 270-2	158.0	178.0 [3]	15		55
			S/N 270-3	155.0	173.0	15		58
		Boss Area	S/N 270-5	152.0	174.0	16		52
			S/N 270-6	150.0	166.0	16		50
Weld Metal [2]	G-804	Axial Direction	1	133.5	154.8	10	In 0.5 Inch	Not Determined
			2	133.0	152.4	10		
	301275	Axial Direction	3	135.0	155.2	10		
			4	140.7	156.6	7		

[1] Determined By Testing Engineers Incorporated
[2] Determined By The Boeing Company
[3] Not In Conformance With JPL Specification ES504496A

Table IV: BASE METAL PLANE STRAIN FRACTURE TOUGHNESS

HEAT NUMBER	SPECIMEN NUMBER	THICKNESS, t (IN.)	WIDTH, W (IN.)	FLAW DEPTH, a (IN.)	FLAW LENGTH, 2c (IN.)	TEST ENVIRONMENT	TEST TEMPERATURE (°F)	THRU-THE-THICKNESS			FAILURE			
								GROSS STRESS, σ_T (KSI)	FLAW SIZE, a/Q (IN.)	CALCULATED FRACTURE TOUGHNESS $(K_{Ic})_T$ (KSI $\sqrt{IN}$)	GROSS STRESS, σ_F (KSI)	FLAW SIZE, a/Q (IN.)	CALCULATED FRACTURE TOUGHNESS $(K_{Ic})_F$ (KSI $\sqrt{IN}$)	$\frac{a+\rho}{t}$ [1]
G-804	AK-3	0.0335	0.9998	0.0185	0.094	AIR	70	146.6	0.0167	44.03	149.3	0.0168	44.98	>1.0
	AK-4	0.0328	1.0002	0.0190	0.096	AIR	70	147.8	0.0171	45.91	148.3	0.0171	46.10	>1.0
	AK-5	0.0333	1.0005	0.0180	0.092	AIR	70	146.2	0.0162	42.98	149.5	0.0164	44.11	>1.0
301275	BK-1	0.0335	0.9991	0.0185	0.092	AIR	70	142.8	0.0161	42.14	148.8	0.0163	44.20	>1.0
	BK-2	0.0339	0.9998	0.0180	0.093	AIR	70	153.0	0.0163	44.76	153.0	0.0163	44.76	>1.0
	BK-3	0.0337	0.9998	0.0190	0.098	AIR	70	146.9	0.0169	44.87	149.5	0.0170	45.80	>1.0
	1	0.1540	1.0003	0.0470	0.163	AIR	70	-	-	-	146.7	0.0335	54.78	0.46
	2	0.1528	1.0009	0.0500	0.158	AIR	70	-	-	-	147.1	0.0333	55.22	0.49
G-804	AK-1	0.0333	0.9999	0.0170	0.096	LN_2	-320	-	-	-	191.9	0.0154	53.83	0.86
	AK-2	0.0338	1.0001	0.0180	0.099	LN_2	-320	-	-	-	200.3	0.0164	58.76	0.94
	AK-6	0.0334	1.0002	0.0175	0.098	LN_2	-320	-	-	-	191.3	0.0158	54.83	0.89
301275	BK-4	0.0328	0.9997	0.0170	0.097	LN_2	-320	-	-	-	161.6	0.0147	44.53	0.74
	BK-5	0.0335	1.0015	0.0175	0.096	LN_2	-320	-	-	-	167.0	0.0150	46 61	0.76
	BK-6	0.0336	1.0013	0.0170	0.097	LN_2	-320	-	-	-	174.4	0.0149	48.02	0.76

[1] ρ = Plastic Zone Size = $\frac{\pi}{16}\left(\frac{K_I}{\sigma_{ys}}\right)^2$

Table V: WELD METAL PLANE STRAIN FRACTURE TOUGHNESS

HEAT NUMBER	SPECIMEN NUMBER	FLAW LOCATION	THICKNESS, t (IN.)	WIDTH, W (IN.)	FLAW DEPTH, a (IN.)	FLAW LENGTH, 2c (IN.)	GROSS STRESS, σ_G (KSI)	FLAW SIZE, a/Q (IN.)	TEST ENVIRONMENT	TEST TEMPERATURE (°F)	FRACTURE TOUGHNESS, K_{Ic} (KSI $\sqrt{IN}$)
G-804	AWK-1	℄	0.0589	1.5030	0.0270	0.151	135.8	0.0256	AIR	70	47.48
	AWK-2	℄	0.0566	1.5035	0.0250	0.150	136.1	0.0243	AIR	70	45.99
	AWK-4	HAZ	0.0547	1.5047	0.0250	0.142	143.5	0.0238	AIR	70	48.39
	A1WK-5	HAZ	0.0555	1.5039	0.0260	0.146	140.5	0.0246	AIR	70	48.53
301275	BWK-1	℄	0.0588	1.5033	0.0320	0.160	123.3	0.0282	AIR	70	47.93
	BWK-2	℄	0.0590	1.5037	0.0270	0.158	130.8	0.0260	AIR	70	46.13
	B1WK-5	HAZ	0.0545	1.5039	0.0235	0.146	141.5	0.0232	AIR	70	46.38
	B1WK-6	HAZ	0.0555	1.5046	0.0280	0.146	138.9	0.0257	AIR	70	50.10
G-804	AWK-3	℄	0.0573	1.5037	0.0240	0.153	145.0	0.0219	LN_2	-320	45.96 ▷
	A1WK-6	HAZ	0.0567	1.5035	0.0255	0.148	130.3	0.0221	LN_2	-320	42.17
	AWK-7	HAZ	0.0550	1.5038	0.0270	0.148	101.6	0.0222	LN_2	-320	33.76
301275	BWK-3	℄	0.0577	1.4944	0.0290	0.155	108.1	0.0237	LN_2	-320	37.45
	BWK-4	℄	0.0586	1.5010	0.0255	0.157	124.1	0.0224	LN_2	-320	40.07
	BWK-7	HAZ	0.0564	1.5023	0.0230	0.145	107.0	0.0199	LN_2	-320	32.14

▷ Incomplete Flaw

Table VI: BASE METAL SUSTAINED LOAD FLAW GROWTH DATA IN NITROGEN TETROXIDE

HEAT NUMBER	SPECIMEN NUMBER	THICKNESS, t (IN.)	WIDTH, W (IN.)	INITIAL FLAW DEPTH, a (IN.)	INITIAL FLAW LENGTH, 2c (IN.)	SUSTAINED GROSS STRESS, σ_G (KSI)	FLAW SIZE a/Q (IN.)	TEST DURATION (HRS.)	AVERAGE TEST TEMPERATURE (°F)	INITIAL STRESS INTENSITY, K_{Ii} (KSI $\sqrt{IN}$)	AMOUNT OF GROWTH
G-804	AS-1	0.0331	1.0162	0.0160	0.097	130 6	0.0149	0.08	84	35.41	Slight
	AS-2	0.0336	1.0001	0.0170	0.097	88.8	0.0144	1002	84	23.96	Slight [2]
	AS-3	0.0335	1.0034	0.0180	0.099	88.8	0.0150	1002	84	25.02	Slight [2]
	AS-4	0.0328	1.0228	0.0180	0.100	104.2	0.0154	1002	83	30.02	Trace [2]
	AS-5X	0.0329	1.0210	0.0170	0.095	96.6	0.0144	1003	84	26.31	None
	AS-6X	0.0331	1.0155	0.0180	0.098	130.6	0.0161	0.08	84	38.32	Moderate
	AS-8	0.0330	1.0173	0.0170	0.095	96.6	0.0144	1003	84	26.28	Trace [2]
	AS-9	0.0335	1.0040	0.0180	0.097	103.9	0.0152	1002	83	29.50	Trace [2]
	AS-10	0.0335	1.0034	0.0190	0.097	115.2	0.0160	22.5	88	34.29	Trace
301275	B1-1	0.0332	1.0428	0.0170	0.096	111.8	0.0147	[1]	84	30.66	Signif. [3]
	B1-2	0.0324	1.0660	0.0170	0.102	86.5	0.0145	1002	84	23.74	None
	B1-3	0.0329	1.0503	0.0170	0.097	93.8	0.0144	1003	84	25.51	Slight [2]
	B1-4	0.0330	1.0495	0.0160	0.090	86.3	0.0133	1002	84	22.13	None
	BS-1X	0.0335	1.0334	0.0175	0.097	126.8	0.0154	0.08	84	35.89	Slight
	BS-2X	0.0334	1.0360	0.0175	0.096	93.8	0.0146	1003	84	25.82	None
	BS-3	0.0337	1.0273	0.0180	0.098	126.8	0.0158	0.08	84	36.56	Failed
	BS-4	0.0335	1.0339	0.0170	0.098	100.9	0.0146	1002	83	27.46	Slight [2]
	BS-X5	0.0339	1.0213	0.0180	0.088	100.9	0.0144	1002	83	27.78	Slight [2]

[1] Specimen Failed in the Grip Immediately After Reaching Load

[2] Growth On Loading

[3] Flaw Grew Through The Thickness on Loading

Table VII: WELD METAL SUSTAINED LOAD FLAW GROWTH DATA IN NITROGEN TETROXIDE

HEAT NUMBER	SPECIMEN NUMBER	FLAW LOCATION	THICKNESS, t (IN.)	WIDTH, W (IN.)	INITIAL FLAW DEPTH, a (IN.)	INITIAL FLAW LENGTH, 2c (IN.)	SUSTAINED GROSS STRESS, σ_G (KSI)	FLAW SIZE a/Q (IN.)	TEST DURATION (HRS.)	AVERAGE TEST TEMPERATURE (°F)	INITIAL STRESS INTENSITY, K_{Ii} (KSI $\sqrt{IN}$)	AMOUNT OF GROWTH
G-804	AWS-1	℄	0.0558	1.5173	0.0275	0.156	92.2	0.0238	1002	85	31.77	None
	AWS-2	℄	0.0566	1.4995	0.0275	0.156	85.5	0.0235	997	85	29.14	
	AWS-3	℄	0.0570	1.4895	0.0280	0.157	75.9	0.0236	1002	84	25.98	
	AWS-4	HAZ	0.0525	1.5770	0.0260	0.145	94.3	0.0224	1002	85	31.61	
	A1WS-5	HAZ	0.0554	1.4955	0.0250	0.145	87.6	0.0216	997	85	28.05	
	A1WS-6	HAZ	0.0532	1.4850	0.0250	0.146	81.6	0.0214	1002	84	26.32	
301275	B1WS-1	℄	0.0574	1.4987	0.0275	0.156	90.8	0.0237	1002	85	30.97	
	B1WS-2	℄	0.0555	1.5480	0.0300	0.157	84.5	0.0248	997	85	30.68	
	BWS-3	℄	0.0575	1.4952	0.0295	0.156	75.0	0.0241	1002	84	26.32	
	BWS-4	HAZ	0.0545	1.5360	0.0235	0.146	93.3	0.0210	1002	85	29.14	
	BWS-5	HAZ	0.0555	1.5077	0.0265	0.145	86.8	0.0224	997	85	28.73	
	BWS-6	HAZ	0.0538	1.5555	0.0260	0.146	77.0	0.0218	1002	84	25.22	None

Table VIII: FLAW GROWTH DUE TO LOADING

HEAT NUMBER	FLAW LOCATION	SPECIMEN NUMBER	THICKNESS, t (Inch)	WIDTH, w (Inch)	INITIAL FLAW DEPTH, a_i (Inch)	INITIAL FLAW LENGTH, $2c_i$ (Inch)	MAXIMUM GROSS STRESS, σ_G (Ksi)	FLAW SIZE, a/Q (Inch)	TEST TEMPERATURE (°F)	INITIAL STRESS INTENSITY, K_{Ii} (Ksi $\sqrt{In}$)	AMOUNT OF GROWTH
301275	℄	BWS-7	0.0432	1.4850	0.0290	0.157	60.0	0.0233	70	23.42	None
	HAZ	BWS-8	0.0442	1.4505	0.0245	0.145	70.0	0.0206	70	23.40	None
G-804	℄	A1WS-7	0.0439	1.5523	0.0285	0.157	80.0	0.0238	70	30.95	Moderate
	HAZ	A1WS-8	0.0440	1.5295	0.0260	0.144	90.0	0.0221	70	32.05	Trace