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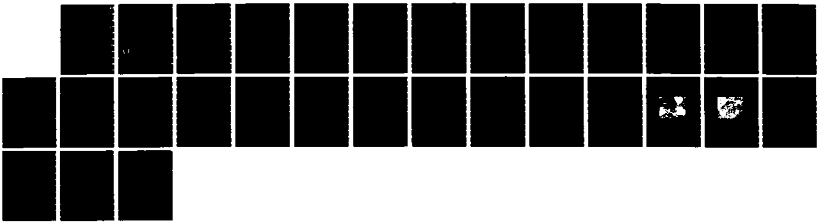

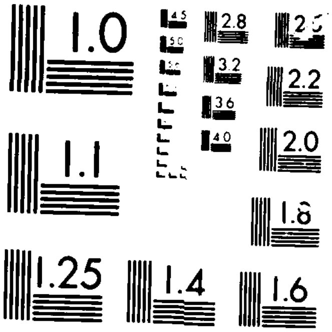

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TECHNICAL REPORT ARCCB-TR-86014

# EMBRITTLEMENT OF A HIGH AND A LOW STRENGTH STEEL IN LIQUID LEAD ENVIRONMENT

M. H. KAMDAR

APRIL 1986

U.S. ARMY
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CLOSE COMBAT ARMAMENTS CENTER
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A study has been made of the cyclic fatigue fracture behavior of single-edge notched specimens of 4340 type high strength (yield stress 160 Ksi) and alloy steel of low strength (yield stress 100 Ksi) with and without a fatigue precrack tested in liquid lead and argon at 700°F. The high strength steel specimens were severely embrittled by liquid lead with stress intensity at fracture some two orders of magnitude lower in liquid lead than in the argon

(CONT'D ON REVERSE)

20. ABSTRACT (CONT'D)

environment. The embrittlement susceptibility was the same for both the notched specimens and for the specimens that had a fatigue precrack at the root of the notch. On the other hand, identical as-notched specimens of low strength steel were immune to lead embrittlement. However, when these specimens were fatigue precracked, they were severely embrittled by liquid lead. This variation in susceptibility to embrittlement is discussed in terms of the prevalent "reduction in cohesion" mechanism of liquid metal embrittlement. The implications of these results in determining the embrittlement susceptibility or elimination are also discussed.

A-1

# TABLE OF CONTENTS

Page

ACKNOWLEDGEMENTS iii

INTRODUCTION 1

EXPERIMENT 2

RESULTS 5

DISCUSSION 6

SUMMARY 10

REFERENCES 11

## TABLES

I. TENSILE TEST DATA FOR 4340 STEEL TESTED IN TENSION IN LIQUID LEAD AND ARGON ENVIRONMENTS AT 700°F 12

II. STATIC FATIGUE TEST DATA FOR SMOOTH SPECIMENS LOADED TO VARIOUS STRESS LEVELS AND TESTED IN LEAD AND ARGON AT 700°F 12

III. STATIC FATIGUE TEST DATA FOR AS-NOTCHED SPECIMENS TESTED IN LIQUID LEAD AND SIMILAR SPECIMENS WITH A FATIGUE PRECRACK TESTED IN ARGON AT 700°F 13

IV. CYCLIC FATIGUE TEST DATA FOR SINGLE-EDGE NOTCH SPECIMENS FATIGUE PRECRACKED AT 70°F AND TESTED AT 700°F IN LIQUID LEAD AND ARGON 13

V. CYCLIC FATIGUE TEST DATA FOR SINGLE-EDGE NOTCH SPECIMENS WITHOUT A FATIGUE PRECRACK TESTED AT 700°F IN LIQUID LEAD AND ARGON 14

VI. CYCLIC FATIGUE TEST DATA FOR 4340 AS-NOTCHED SPECIMENS TESTED IN TENSION-TENSION LOAD OF 1950 LBS. IN ARGON AND IN LIQUID LEAD AT VARIOUS TEMPERATURES 14

VII. CYCLIC FATIGUE TEST DATA FOR AS-NOTCHED STEEL SPECIMENS OF CHROME-MOLY STEEL* TESTED IN TENSION-TENSION LOADS IN LIQUID LEAD AND PURIFIED ARGON ENVIRONMENTS AT 700°F 15

VIII. CYCLIC FATIGUE TEST DATA FOR FATIGUE PRECRACKED CHROME-MOLY SPECIMENS (YIELD STRESS 70 Ksi, Rc 8) TESTED IN TENSION-TENSION LOAD OF 1950 LBS. IN LIQUID LEAD AND ARGON AT 700°F 16

# LIST OF ILLUSTRATIONS

Page

1. Edge-notch tensile specimen. 17

2. Smooth specimen tested in argon showing ductile failure. 18

3. Smooth specimen tested in liquid lead showing brittle intergranular failure. 19

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The author appreciates the assistance of Mr. Harry Nazarian in conducting the tests and Mr. C. Nolan for lead electroplating the test specimens. Assistance of Mr. McNamara was very helpful in the removal of lead from fracture surfaces and subsequent examinations in the SEM. Thanks are extended to Ellen Fogarty and Rose Neifeld for their incisive comments and for preparing the manuscript.

## INTRODUCTION

Lead is used as an alloying element in steel and is known to facilitate machining of high strength steel. Such steels are called free machining steels and are used in the manufacture of gears. Lead is also used as a lubricant for mechanical swage autofrettage of pressure vessel tubes to introduce compressive stresses and increase the fatigue life of the tubes. Lead-lithium alloys are considered for use in nuclear vessel heat exchange steel tubes. Thus, lead is used in a variety of ways in steel in both the solid and liquid state. However, lead is known to degrade mechanical properties of steel. Lead-containing gears are known to fracture by brittle intergranular mode. Pressure vessels autofrettaged with lead as a lubricant develop cracks during post-autofrettage thermal heat treatment near the melting point (m.p.) of lead at 700°F. This phenomenon of fracture is known as liquid lead embrittlement of steel. Leaded steels when heated near the m.p. of lead, as well as smooth steel specimens tested in liquid lead, are severely embrittled by lead (ref 1). However, little is known about the effects of static and cyclic fatigue test conditions on the liquid lead embrittlement of steel specimens that are smooth, notched, or notched and fatigue precracked. Also, the effects of yield stress on the embrittlement susceptibility are not known even though liquid metal embrittlement is sensitive to the strength level of the metal being embrittled.

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[1] P. Gordon, N. Breyer, L. Broutman, D. Albright, and W. Warke, "Environment Sensitivity of Structural Materials: Liquid Metal Embrittlement," Project Themes Progress Reports, Illinois Institute of Technology, Chicago, IL, 1969-1973.

The purpose of this investigation was to study the effects of yield strength on liquid metal embrittlement of high and low strength steel specimens. The specimens were smooth, notched, and notched with a fatigue crack at the tip. The results are discussed here in terms of the prevalent "reduction in cohesion" mechanism of liquid metal embrittlement and stress concentration at the root of the notches.

## EXPERIMENT

The material used in this investigation was a high strength, high purity 4340 steel, a low strength chrome-moly steel (Tables I and VII), and a high purity (99.99%) lead. Smooth round tensile specimens 0.25 inch in diameter and 1.25 inch in gage length were machined from steel and were used for crack initiation studies in monotonic and static fatigue tests. These specimens were mounted in a Rhiele tensile test machine and were enclosed in a furnace. The specimens were tested to failure at a constant load either in a flowing purified argon or liquid lead environment at desired temperatures. The load-elongation curves were recorded. Subsequently, the specimens were cooled in argon to room temperature. In static fatigue tests, smooth specimens and notched specimens with and without fatigue precrack and liquid lead environments were preloaded to 70°F to various stresses and heated to 700°F in purified argon while maintaining the stress constant. At 700°F, the stress on the specimen was kept constant for four hours and the time to failure or otherwise was noted.

In other tests from these steels, single-edge notched fracture toughness specimens having ASTM configurations, Figure 1, were machined for crack propagation studies in cyclic fatigue. The test specimens were 0.25 inch thick, 1.25 inches wide, and had loading pin holes 4 inches apart. They had a 60 degree notch with 0.005 inch root radius extending 0.525 inch through the width of the specimen. Some of the specimens were fatigued at 70°F to introduce a sharp (about 0.04 inch long) precrack. The same procedure was used to introduce fatigue precrack in specimens which were electroplated with a lead coating around the notch. The fatigue precrack was introduced underneath the lead plating thereby preventing oxidation or contamination of the precrack. This procedure assures wetting of the crack tip when lead is molten or liquid.

The specimens were cleaned in acetone, reverse etched (ref 2), and electroplated with ~ 40 mil thick coating of lead extending one inch on either side and all around the notch in the specimen. The specimen was mounted in a stainless steel cylindrical environment test chamber. The environment test chamber, 3 inches in diameter and 7 inches long, had a loading rod welded to its bottom. The loading rod extended inside the chamber. It had a slot at its end with holes where the specimen could be loaded by inserting a pin through the holes in the specimen and the loading rod. The rod extended outside the chamber and was threaded so that it could be screwed into the Sontag Fatigue testing machine. The cylinder was open at the other end and closed with a three-inch diameter screw cap. The cap had an opening for the specimen so that it could be connected to the upper loading rod by inserting a loading pin through the specimen and the loading rod. The screw cap had an inlet coupling for connecting the argon gas line which was cooled by liquid nitrogen

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[2] C. J. Nolan, Private Communication, U.S. Army Armament Research and Development Center, Benet Weapons Laboratory, Watervliet, NY, 1985.

to provide continuous flow of virtually oxygen-free argon inside the test chamber. The lead plated specimen was mounted in the environment chamber. A high purity lead was melted in another stainless steel container and was poured around the specimen so that the liquid lead covered the entire specimen including the volume at least two inches above the notch of the specimen. The test chamber containing the specimen and solidified metal environment was screwed into the Sontag Fatigue testing machine. The upper end of the specimen was connected to the upper loading rod by inserting a pin through the holes in the specimen and the slot in the loading rod.

The cap was screwed onto the cylinder and the whole assembly was enclosed in a fast heating three-zone split electric furnace. The heating of the three zones was controlled individually so that the liquid at the crack tip could be heated to 1200°F in one-half hour. The temperature of the metal bath was monitored by a thermocouple placed near the crack tip when the molten metal was poured earlier around the specimen. A small preload was applied to the specimen and was adjusted periodically so that the specimen would not break due to tensile stresses caused by the expansion of the specimen during heating or give erroneous results. When the liquid metal at the crack tip reached the desired temperature, the specimen was loaded to 1950 pounds for a tension-tension cyclic fatigue test. The specimen was tested at 1800 rpm in the Sontag machine to failure and the cycles to failure were recorded. The upper half of the specimen was lifted out of the molten bath immediately after failure by raising the upper loading rod to prevent excessive liquid metal from solidfying on the fracture surface. This facilitated subsequent removal of the solidified liquid metal from the fracture surface. The fracture surfaces were cleaned by various means including chemical removal and film stripping of the adherent metal. The cleaned surface of an area was examined in a scanning electron microscope and the fracture mode was determined.

## RESULTS

The tensile and static fatigue test data for crack initiation in a smooth specimen in argon and liquid lead environments are given in Tables I and II. The specimens tested in argon failed in a ductile manner, whereas those tested in liquid lead failed by brittle intergranular mode with total loss in ductility (Tables I and II and Figures 2 and 3).

Static fatigue results for notched specimens given in Table III show that the smallest stress intensity or K value for failure in liquid lead is 35 $\text{Ksi}\sqrt{\text{in.}}$ This value is four times lower than the stress intensity for fracture of 135 $\text{Ksi}\sqrt{\text{in.}}$ for steel in an inert argon environment.

The cyclic fatigue results for specimens with notches and with a fatigue crack at the root of the notches tested in liquid lead and argon at 700°F are given in Tables IV and V.

The initial stress intensity at the root of the notch, or the tip of the fatigue precrack at the point of failure reported above, and for the cyclic tests was calculated using measured crack length a, the width of the specimen b, the applied tensile stress $\sigma$, the equation of $K = \sigma\sqrt{\pi a}\ F(a/b)$, and the calibration curve for values of F for given values of a and b. The calibration curve and the equation for calculating K values for single-edge notched precracked flat bar specimens tested in tension were taken from ASTM publication No. 410 (ref 3). The results in Tables IV and V show that the K values

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[3] W. F. Brown, Jr. and J. E. Srawley, "Plain Strain Crack Toughness Testing of High Strength Metallic Materials," ASTM STP 410, 1966.

in liquid lead decreased by one order of magnitude to 7 Ksi$\sqrt{in.}$ and cycles to failure decreased by more than two orders of magnitude (to 2,000 cycles) as compared to those in argon.

The K values in liquid lead were the same for specimens with machined notches only and for those with a sharp fatigue precrack at the root of the notch. This is a significant result. The threshold value for embrittlement was 5 Ksi$\sqrt{in.}$

The ductile-brittle transition data for 4340 steel are given in Table VI. It is seen that ductile-brittle transition occurs at 1200°F, some 500°F above the melting point of lead. These results suggest that a decrease in the yield stress of the steel may make steel immune to liquid lead embrittlement. To verify this, tests were performed with a low strength (yield stress 100 Ksi at 70°F) chrome-moly steel. The results of fatigue tests with as-notched specimens are given in Table VII. It is clearly seen that this steel is not embrittled by liquid lead. However, it was noticed that some specimens with high hardness which resulted from a variation in the heat treatment appear to be embrittled by liquid lead. This suggests that the specimens which are not embrittled by liquid lead, Table VII, may become susceptible to embrittlement if they had a fatigue precrack at the root of the notch. The results on fatigue precrack specimens are given in Table VIII. It is seen that the specimens are severely embrittled by liquid lead and the fatigue life in liquid lead is reduced to some 20 percent of that in an inert argon environment. The fracture mode changes from ductile to brittle intergranular mode.

## DISCUSSION

The smallest stress to initiate a crack in smooth specimens wetted with lead and tested in tension or in static fatigue is the same as the tensile yield stress of the steel, Tables I and II. This suggests that the simultaneous presence of plastic flow due to yielding, a tensile stress in the specimen, and liquid lead in intimate contact with steel are necessary for brittle crack initiation. These results or conditions are the same as the general prerequisites for the occurrence of liquid metal embrittlement proposed by Kamdar (refs 4 and 5). Additionally, the result that yielding must precede fracture suggests the following mechanism for crack initiation in a liquid lead environment. Slip bands produced upon yielding are blocked at grain boundaries inducing high stress concentrations at the grain boundary barriers. Adsorption of the liquid lead atoms at the grain boundary reduces the cohesive strength of the iron-iron bonds in the boundary, and as a consequence, a lower stress concentration is required for crack initiation. A brittle crack initiates in the grain boundary when the stress concentration is equal to or greater than the reduced cohesive strength of the steel.

Cyclic fatigue tests show the stress intensity at fracture is one order of magnitude less in liquid lead than in argon, Tables IV and V. Furthermore, this value (7 Ksi$\sqrt{\text{in.}}$) is half an order of magnitude lower than that determined in static fatigue tests. This suggests that initiation is a much more difficult step in fracture than in crack propagation. This, of course, is consistent with general observations on fracture both in liquid metal environments as well as in the absence of such chemically aggressive environments (ref 5).

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[4] M. H. Kamdar, "Liquid Metal Embrittlement," Treatise on Material Science and Technology, (C. L. Briant and S. K. Banerjee, eds.), Academic Press, Vol. 25, 1983, pp. 361-459.

[5] M. H. Kamdar, "Embrittlement by Liquid Metals," Prog. Mat. Sci., Vol. 15, 1973, p. 289.

It is significant to note that for a sharp fatigue crack or a blunt machined notch the stress intensity at fracture and the number of cycles to failure are the same, i.e., embrittlement is independent of the sharpness of the crack front or tip radii, whereas in argon the fatigue life is sensitive to the presence of a fatigue precrack, Tables IV and V. This effect is strikingly apparent in chrome-moly steel where as-notched specimens which are not susceptible to embrittlement become significantly susceptible to embrittlement when a fatigue precrack is present at the root of the notch, Tables VII and VIII. Westwood and Kamdar (ref 6) have explained such behavior by suggesting that embrittlement is a highly localized event occurring on an atomic scale only at the crack front. Thus, for a brittle crack to initiate in liquid metal environment, the only important consideration should be that the strength of the metal-metal atom bonds along the very crack front be reduced by the adsorbed liquid metal atoms. As far as the stress concentration being sufficient and corresponding to the reduced cohesive strength, embrittlement and crack propagation occur independent of the root radius at the crack front and are additionally independent of the liquid metal atoms adsorbed elsewhere near the crack front or on the crack surfaces. However, it is apparent that if the stress concentrations at the crack tip are lower than the reduced cohesive strength, plastic deformation will occur at the tip and apparent immunity to embrittlement will be observed as was noted in the case of as-notched chrome-moly specimens, Table VII. On the other hand, if the stress concentration is increased by introducing a sharp fatigue precrack or by increasing the strength of the alloy by heat treatment, etc.,

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[6] A.R.C. Westwood and M. H. Kamdar, Phil. Mag., Vol. 6, 1963, p. 787.

severe embrittlement will occur as observed, Table VIII.

The embrittling action is highly localized and appears independent of the crack tip radii. Occurrence of embrittlement or otherwise will depend upon the stress concentrations at the crack front only. For stress concentrations equal to or greater than the reduced cohesive strength of the metal atom bonds by the liquid metal atoms, the crack will propagate in a brittle manner, whereas when the stress concentration is slower, a ductile behavior or no embrittlement will be noted. The continued propagation of a crack in liquid to failure may depend upon the surface diffusion of liquid metal atoms over adsorbed liquid metal atoms feeding the propagating crack tip. If the liquid is not able to do so and plastic blunting of the tip occurs due to emission of dislocations from the tip of the moving crack, then a brittle to ductile transition may be observed on the fracture surface. Alternatively, once a brittle crack is initiated by a liquid metal, further crack propagation may occur mechanically in the absence of the liquid at the tip of the crack, particularly in a high strength metal or alloy when a sharp crack may propagate to fracture instead of being blunted by plastic flow at the tip of the crack.

At the present time, it is not possible to calculate or even estimate the theoretical cohesive strength of metal atoms in the presence of adsorbed liquid metals (ref 5). The strength level or yield stress of the alloy is important in determining the stress concentrations at the crack front and in providing a rational explanation of the embrittlement behavior reported here

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[5] M. H. Kamdar, "Embrittlement by Liquid Metals," Prog. Mat. Sci., Vol. 15, p. 289.

for two steels in a liquid lead environment.

## SUMMARY

1. High strength 4340 steel is severely embrittled by liquid lead when tested in monotonic, static, or cyclic fatigue conditions. Such embrittlement is independent of the root radii investigated in this report.

2. The susceptibility of the chrome-moly steel to liquid lead environment depends upon the stress concentrations at the tip of the crack front and on the presence or absence of a fatigue precrack at the root of the notch.

3. Fatigue testing of precracked specimens provides the most severe conditions for the occurrence of embrittlement. It would be incorrect to assume immunity of embrittlement since test conditions such as stress, strain rate, temperature, microstructure, etc., can significantly alter embrittlement susceptibility.

4. Yielding is a necessary prerequisite for the initiation of liquid metal embrittlement.

5. The reduction in cohesion model of liquid metal embrittlement of Westwood and Kamdar (ref 6) and Stoloff and Johnston (ref 7) adequately explains the results presented in this report.

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[6] A.R.C. Westwood and M. H. Kamdar, Phil. Mag., Vol. 6, 1963, p. 787.

[7] N. S. Stoloff and T. L. Johnston, Acta Met., Vol. 11, 1963, p. 251.

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1. P. Gordon, N. Breyer, L. Broutman, D. Albright, and W. Warke, "Environment Sensitivity of Structural Materials: Liquid Metal Embrittlement," Project Themes Progress Reports, Illinois Institute of Technology, Chicago, IL, 1969-1973.
2. C. J. Nolan, Private Communication, U.S. Army Armament Research and Development Center, Benet Weapons Laboratory, Watervliet, NY, 1985.
3. W. F. Brown, Jr. and J. E. Srawley, "Plain Strain Crack Toughness Testing of High Strength Metallic Materials," ASTM STP 410, 1966.
4. M. H. Kamdar, "Liquid Metal Embrittlement," Treatise on Material Science and Technology, (C. L. Briant and S. K. Banerjee, eds.), Academic Press, Vol. 25, 1983, pp. 361-459.
5. M. H. Kamdar, "Embrittlement by Liquid Metals," Prog. Mat. Sci., Vol. 15, 1973, p. 289.
6. A.R.C. Westwood and M. H. Kamdar, Phil. Mag., Vol. 6, 1963, p. 787.
7. N. S. Stoloff and T. L. Johnston, Acta Met., Vol. 11, 1963, p. 251.

TABLE I. TENSILE TEST DATA FOR 4340 STEEL* TESTED IN TENSION IN LIQUID LEAD AND ARGON ENVIRONMENTS AT 700°F

| Specimen | Environment | Yield Stress (Ksi) | Fracture Stress (Ksi) | R.A. (%) | Fracture Mode |
|---|---|---|---|---|---|
| Smooth-Tensile | Argon | 125 | 515 | 70 | Ductile |
| Smooth-Tensile | Liq. Lead | 125 | 120 | 0 | Brittle |

| * C | Mn | P&S | Si | Ni | Cr | Mo | V |
|---|---|---|---|---|---|---|---|
| 0.32 | 0.45 | 0.005 | 0.50 | 3.00 | 0.85 | 0.66 | 0.10 |

(Yield Stress 160 Ksi at 70°F)

TABLE II. STATIC FATIGUE TEST DATA FOR SMOOTH SPECIMENS LOADED TO VARIOUS STRESS LEVELS AND TESTED IN LEAD AND ARGON AT 700°F

| Specimen | Environment | Stress (Ksi) | Time to Failure at 700°F | Fracture Mode |
|---|---|---|---|---|
| Smooth | Argon | 120-150 | No failure (4 hrs) | - |
| Smooth | Liq. Lead | 100 | 10 minutes | Brittle |
| Smooth | Liq. Lead | 90 | No failure (4 hrs) | - |

TABLE III. STATIC FATIGUE TEST DATA FOR AS-NOTCHED SPECIMENS TESTED IN LIQUID LEAD AND SIMILAR SPECIMENS WITH A FATIGUE PRECRACK TESTED IN ARGON AT 700°F

| Specimen | Environment | Stress Intensity $\text{Ksi}\sqrt{\text{in.}}$ | Time To Failure at 700°F | Fracture Mode |
|---|---|---|---|---|
| Fatigue Precracked | Argon | 135 | 3-4 hours | Ductile |
| Notched - no fatigue crack | Argon | 135 | No failure | - |
| Notched - no fatigue crack | Liq. Lead | 35 | | Brittle |

TABLE IV. CYCLIC FATIGUE TEST DATA FOR SINGLE-EDGE NOTCH SPECIMENS FATIGUE PRECRACKED AT 70°F AND TESTED AT 700°F IN LIQUID LEAD AND ARGON

| Tensile Load (lbs.) | Environment | Cycles to Failure | Stress Intensity $\text{Ksi}\sqrt{\text{in.}}$ | Fracture Mode |
|---|---|---|---|---|
| 1950 | Argon | $2\times10^5$ | 135 | Ductile |
| 1950 | Liq. Lead | $2\times10^3$ | 15 | Brittle |
| 800 | Liq. Lead | $2\times10^3$ | 7 | Brittle |

TABLE V. CYCLIC FATIGUE TEST DATA FOR SINGLE-EDGE NOTCHED SPECIMENS WITHOUT A FATIGUE PRECRACK TESTED AT 700°F IN LIQUID LEAD AND ARGON

| Tensile Load | Environment | Cycles to Failure | Stress Intensity Ksi$\sqrt{in.}$ | Fracture Mode |
|---|---|---|---|---|
| 1950 | Argon | $5\times10^5$<br>(no failure) | - | - |
| 1950 | Lead | $2\times10^3$ | 15 | Brittle |
| 1250 | Lead | $2\times10^3$ | 11 | Brittle |
| 800 | Lead | $2\times10^3$ | 7 | Brittle |
| 500 | Lead | $2\times10^3$<br>(no failure) | 5 | Threshold Value |

TABLE VI. CYCLIC FATIGUE TEST DATA FOR 4340 AS-NOTCHED SPECIMENS TESTED IN TENSION-TENSION LOAD OF 1950 LBS. IN ARGON AND IN LIQUID LEAD AT VARIOUS TEMPERATURES

| Environment | Temperature °F | Cycles to Failure | Fracture Mode |
|---|---|---|---|
| Argon | 1200°F | 30,000 | Ductile |
| Liq. Lead | 700°F | 2,000 | Brittle |
| Liq. Lead | 900°F | 3,000 | Brittle |
| Liq. Lead | 1000°F | 5,000 | Semi-Brittle |
| Liq. Lead | 1200°F | 20,000 | Ductile |

TABLE VII. CYCLIC FATIGUE TEST DATA FOR AS-NOTCHED STEEL SPECIMENS OF CHROME-MOLY STEEL* TESTED IN TENSION-TENSION LOADS IN LIQUID LEAD AND PURIFIED ARGON ENVIRONMENTS AT 700°F

| Specimen Number | Rockwell C Hardness | Environment | Tension-Tension Load | Test Cycles (1000) | Comments |
|---|---|---|---|---|---|
| 1 | 13 | Lead | 1950 | 15(F) | Brittle |
| 2 | 13 | Lead | 1950 | 20(F) | - |
| 3 | 13 | Lead | 1950 | 20(F) | Brittle |
| 4 | 8 | Lead | 1950 | 160(NF) | - |
| 5 | 8 | Lead | 1000 | 150(NF) | - |
| 6 | 8 | Lead | 1000 | 150(NF) | - |
| 7 | 8 | Lead | 1000 | 225(NF) | - |
| 8 | 25 | Lead | 1000 | 5(F) | Brittle, High Hardness 25 RC |
| 9 | 8 | Lead | 1950 | 140(NF) | - |
| 10 | 8 | Argon | 1000 | 150(NF) | - |
| 11 | 13 | Argon | 1950 | 200(NF) | - |

*Composition:

| C | Mn | P | S | Si | Cr | Mo |
|---|---|---|---|---|---|---|
| 0.12 | 0.44 | 0.014 | 0.018 | 0.018 | 2.17 | 1.00 |

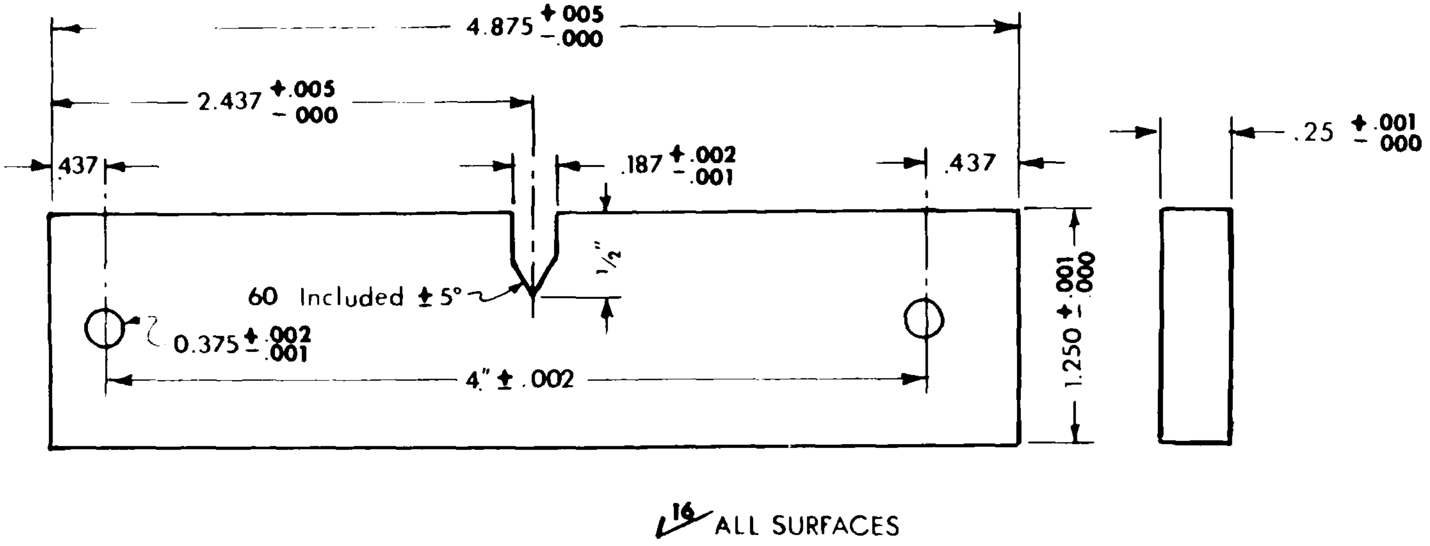
(Yield Stress 100 Ksi, Fracture Stress, 138 Ksi, R.A. 55% at 70°F)

(F): Fail
(NF): Did not fail

TABLE VIII. CYCLIC FATIGUE TEST DATA FOR FATIGUE PRECRACKED CHROME-MOLY SPECIMENS (YIELD STRESS 70 Ksi, Rc 8) TESTED IN TENSION-TENSION LOAD OF 1950 LBS. IN LIQUID LEAD AND ARGON AT 700°F*

| Environment | Cycles to Failure | Fracture Mode |
|---|---|---|
| Argon | 118K | Ductile |
| Argon | 113K | Ductile |
| Argon | 86K | Ductile |
| Liquid Lead | 29K | Brittle |
| Liquid Lead | 21K | Brittle |
| Liquid Lead | 29K | Brittle |
| Liquid Lead | 41K | Brittle |
| Liquid Lead | 21K | Brittle |
| Liquid Lead | 28K | Brittle |

*This test condition is the same as that used for tests reported in Table VII.



EDGE-NOTCH TENSILE SPECIMEN

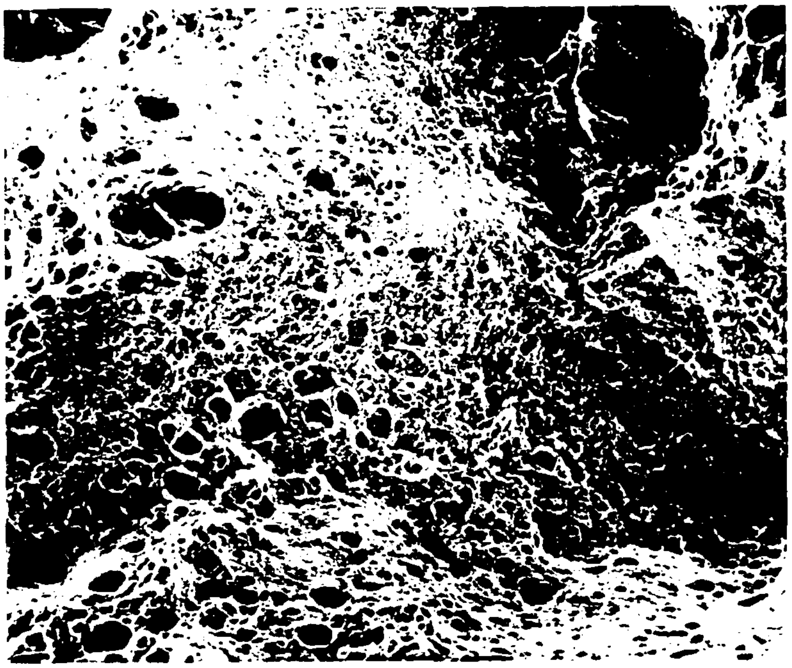


Figure 2. Smooth specimen tested in argon showing ductile failure (500X).

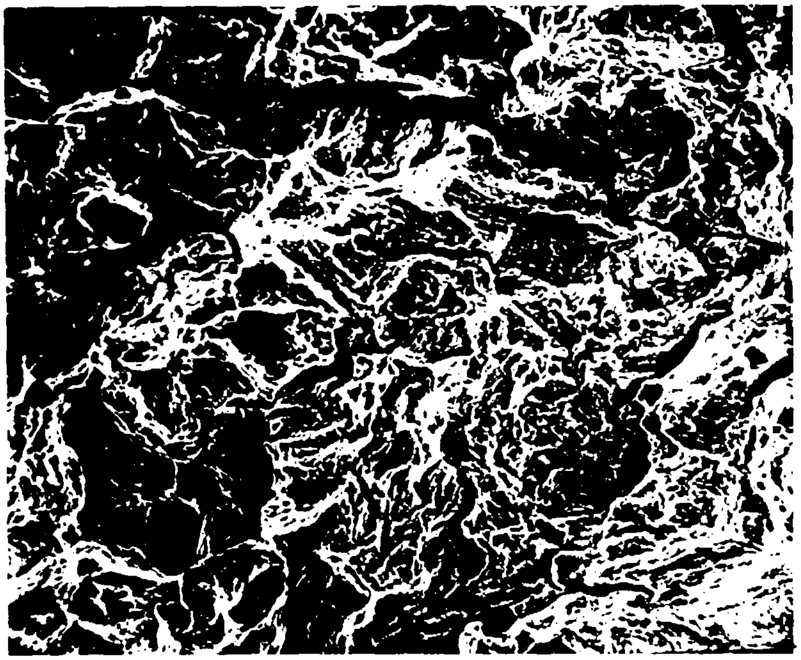


Figure 3. Smooth specimen tested in liquid lead showing brittle intergranular failure (500X).

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