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Contract No. DA-11-ORD-022-3108

FRACTURE OF METALS

ARF-B183-13
(Interim Report)

Commanding Officer
Frankford Arsenal
Philadelphia 37, Pennsylvania

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

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(Interim Report)

March 22, 1963 to June 22, 1963

for

Commanding Officer
Frankford Arsenal
Philadelphia 37, Pennsylvania

Attention: Mr. J. M. McCaughey
Pitman-Dunn Laboratories

July 2, 1963

FRACTURE OF METALS

ABSTRACT

Studies are in progress toward understanding the mechanism of delayed failure in the presence of low-melting liquid metals. Experiments utilize age-hardened beryllium copper wetted with mercury. It is shown that during the time delay period between application of sustained stress to wetted samples and the occurrence of failure, cracks can only be detected during the last minute or fraction thereof. The indications are that during the long preceding period, mercury diffuses into the surface developing a state of what is equivalent to surface age hardening.

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FRACTURE OF METALS

I. INTRODUCTION

This is an interim report for the period March 22, 1963 to June 22, 1963, describing the results of a continuing program of research on a special form of brittle fracture produced when structural metals under stress are wetted by certain liquid metals of low melting point. The presence of the liquid metal on the surface can produce brittle fracture below the yield point of high-strength materials that are normally very ductile. The liquid metal not only reduces the stress to initiate a crack but also, by following the crack, markedly reduces the energy to propagate a crack.

Studies of this phenomenon have largely dealt with failure under simple tensile loading, and fracture strength has been related to structure, heat treatment, and temperature. There is another important aspect--that of delayed failure under sustained loads--which has not been seriously investigated. Present efforts are being devoted to illuminating this aspect of the subject and to develop a mechanism for its occurrence.

II. GENERAL ATTRIBUTES OF DELAYED FAILURE

As a basis for illustration, we shall take the case of age-hardened beryllium copper* wetted with mercury. This alloy in the particular state of heat treatment used possesses a yield strength of about 180,000 psi, an ultimate strength of 190,000 psi, and 2.5% elongation at failure. When wetted with mercury a tensile specimen will fail immediately upon reaching a stress of 131,000 psi. Alternatively, the specimen can be preloaded to this stress level, and failure will occur as soon as wetting is effected. Note that the stress level for instantaneous failure is of the order of 70% of the engineering yield strength.

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* Composition: 1.94% beryllium, 0.25% cobalt, 0.12% iron, 0.005 nickel balance copper.

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If tensile specimens wetted with mercury are loaded to somewhat less than the critical 131,000 psi, failure will occur after a finite delay. The time for delayed failure is proportionate to the tensile stress level and becomes longer the lower the stress. It is characteristic of delayed failure by liquid-metal wetting that a threshold stress exists below which failure will not occur even after prolonged times of sustained loading. There is, therefore, a positive endurance limit in this kind of delayed failure phenomenon. A delayed failure curve for beryllium copper in one state of age hardening is given in Figure 1. The stress interval between instantaneous failure and the endurance limit is larger with age-hardened structures than with simple solid solutions. For example, in cold-worked 70/30 brass wetted with mercury, this stress interval is only about 15,000 psi.

Because of the character of the delayed failure curve, delayed failure times just above the endurance limit show considerable scatter. Minor differences in surface stress can obviously produce large differences in time to failure. Specimen alignment becomes a very important factor. With flat specimens which must undergo heat treatment, this involves considerable difficulty. In the present work, it was found that pressing the specimens between flat dies after treatment and before aging significantly reduced the scatter in failure times. However, this can only be done to correct very slight specimen curvature. The cold work generated by large changes in curvature can significantly reduce critical failure stresses.

Surface condition is also an important factor in failure time. An electropolished surface is recommended as a reference condition. By way of evaluation a series of age-hardened beryllium copper specimens were surface treated by electropolishing, chemical pickling in nitric acid, hand-lapped with metallographic polishing paper and abrasive, and hand-ground with 600 grit carborundum powder. The results of tensile tests after wetting with mercury are summarized in Table I. It is interesting that a surface containing parallel scratches clearly resolvable by eye is conducive to a higher fracture stress than an electropolished condition. This probably signifies that the surface is slightly cold worked to a very shallow depth and that, as a result, the surface has a slight residual compressive stress similar to what can be attained by shot peening. The existence of surface residual stresses is an important

TABLE I

INFLUENCE OF SURFACE CONDITION ON THE TENSILE FRACTURE STRESS OF AGE-HARDENED BERYLLIUM COPPER SPECIMENS* WETTED WITH MERCURY

Surface Condition	Fracture Stress psi Determinations	Fracture Stress psi Average
Electropolished	118,000; 129,000	125,000
	128,000; 126,000	
Chemically pickled	133,000; 120,000	124,000
	118,000	
Hand lapped	126,000; 136,000	134,000
	135,000; 138,000	
	(edges not wetted)	
	132,000; 138,000; 135,000	
Hand ground	151,000; 153,000	158,000
	159,000; 167,000	
	(transverse scratches)	
	159,000	

* Solution treated at 1450°F, water quenched, and aged at 650°F for 2 hours.

aberration in experimental studies because their sign and magnitude can be highly variable and are generally unknown.

III. OBSERVATIONS ON CRACK NUCLEATION IN DELAYED FAILURE

In Figure 1 it can be seen that at a stress of 92,000 psi, failure in the gage section of sheet 0.050 in. thick occurs in about 60 minutes. The crack starts at the surface wetted by Hg and progresses inward. The Hg itself follows the crack to a depth of only about 0.010 in.; thereafter the crack runs away from the Hg because the alloy is very notch sensitive. Normal brittle fracture velocities are greater than Hg-induced cracks as has been shown in age-hardened high-strength Al alloys. Post examination of failed specimens usually shows the existence of several cracks in the wetted zone. These are always pointed transverse to the axis of stress and occupy only a fraction of the width of the specimen. The number of cracks generally increases with lower stresses.

The general story of failure seems to be as follows. Crack nuclei generate at one or more points at the solid-liquid metal interface. These cracks grow across the width of the specimen and into the thickness direction. The individual initiation times and growth rates are not exactly the same, and so one of these leads the rest in depth and total area of cleavage. As each crack grows, the stress intensity increases at the root, which in this case is a curved front. These stresses are clearly above the flow stress of the solid metal as indicated by the slip bands on either side of the fractured surface shown in Figure 2. These slip bands formed after the crack had nucleated because the nucleation stress is well below the yield point. The cracks follow an intergranular path until the notched tensile strength of the specimen is reached. Thereafter, fast brittle fracture without the assistance of Hg takes over and follows a transcrystalline path.

One of the first questions to ask in approaching the problem of defining the mechanism of time-dependent fracture is, When do the cracks nucleate? Do they nucleate immediately upon reaching the static stress level and grow slowly for a period of time, or do they form some time later? Two types of experiments have been conducted to investigate this question.

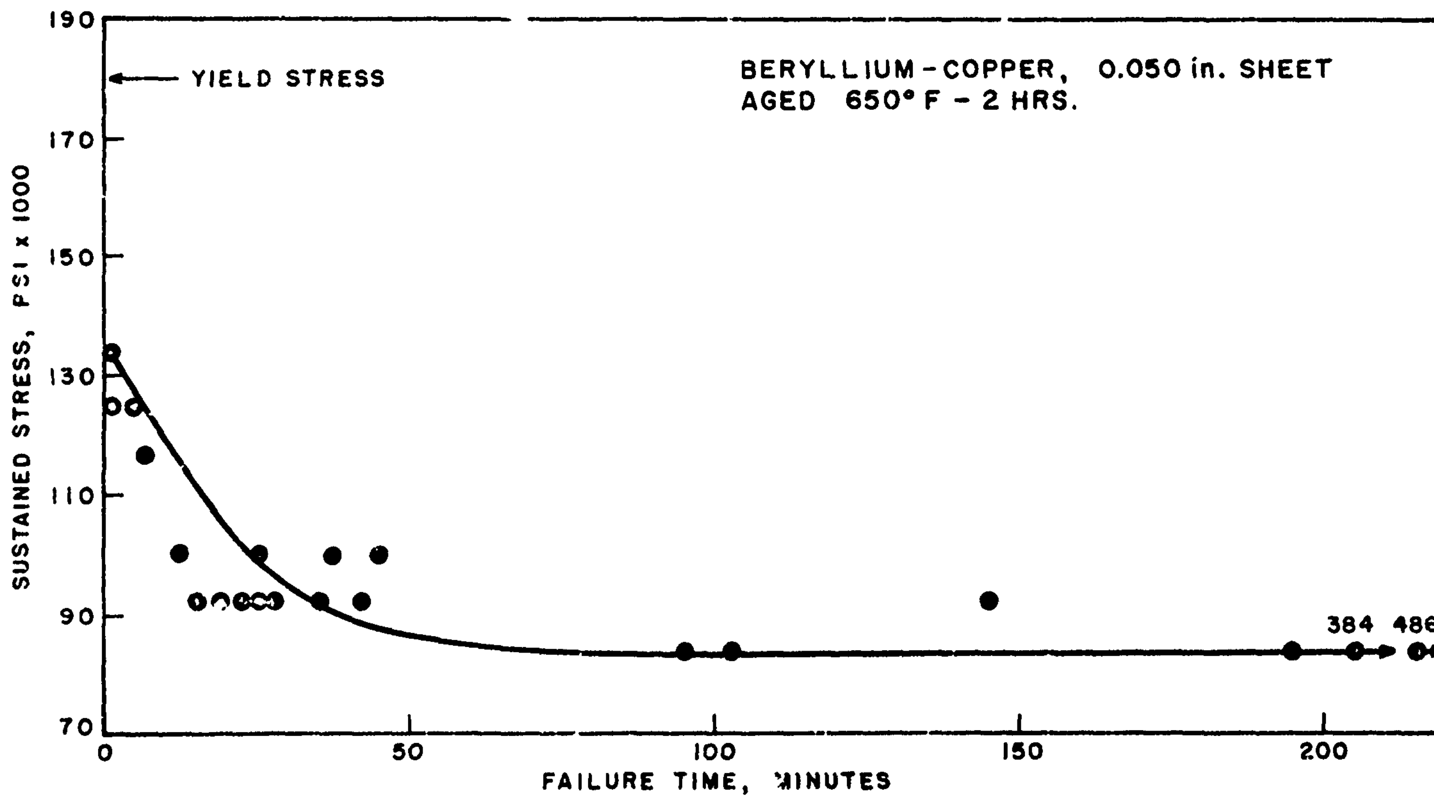
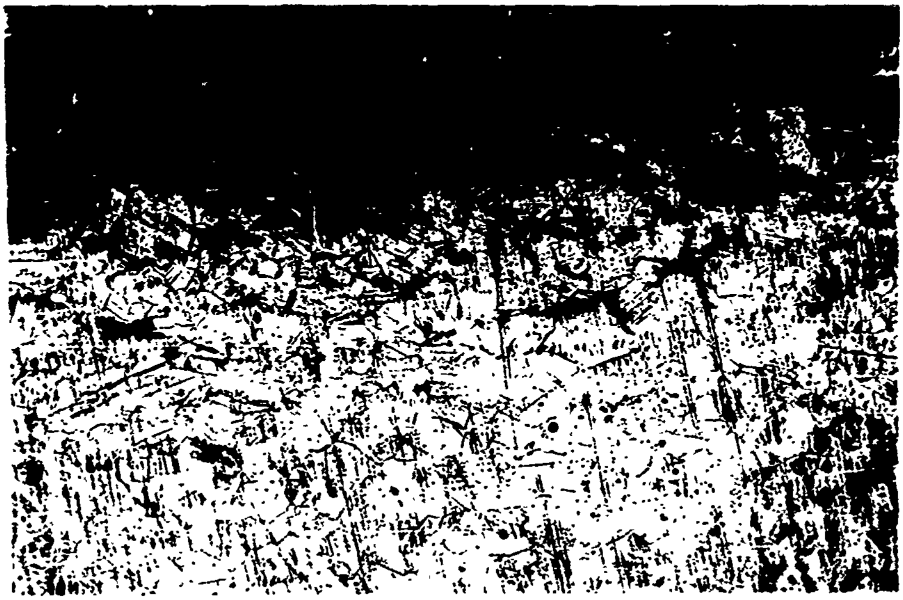


FIG. 1 - DELAYED FAILURE OF AGE HARDENED BERYLLIUM-COPPER WETTED WITH MERCURY ON ONE SURFACE, TESTED AT ROOM TEMPERATURE.



Neg. No. 24373 X70

FIG. 2 - EVIDENCE OF SLIP BANDS IN GRAINS OF BERYLLIUM COPPER SPECIMEN IMMEDIATELY ADJACENT TO THE FRACTURE PATH INDUCED BY WETTING WITH MERCURY AND STRESSING TO 130,000 PSI.

Microstrain measurements have been made continuously up until the time of failure. Microstrain changes capable of resolution to 10^{-6} inches per inch were monitored by a specimen micro-displacement transducer with suitable amplifier circuitry* feeding to a continuously recording potentiometer. The transducer itself was rigidly clamped to the specimen and monitored extensions over about 1/2 inch of gage length wetted with mercury. The purpose of this arrangement was to detect displacements occurring in the axis of tension during the period from load initiation to specimen failure. Whether the displacements represented plastic distortion or cracking was to be resolved by other experiments.

A typical plot of micro-displacement vs. time is shown in Figure 3. It is clear that within a resolution of 10^{-6} inches per inch no displacement of any kind occurs while the specimen is wetted with mercury and under load until something less than a minute before ultimate failure. During this latter period the generation and growth of cracks is clearly apparent.

Repeated attempts were made to identify the presence of microcracks in the surface of specimens by metallographic techniques. The following procedure was adopted. The gage section of a specimen was carefully polished either by lapping or by electropolishing to a metallographic finish. The specimen was then wetted with Hg by placing a drop of $HgCl_2$ solution on the polished surface. A film of metallic, liquid Hg was formed immediately. The water solution was taken off by absorbent paper and additional Hg laid down with an eye dropper. The wetted specimen was stressed to 91,000 psi for times of the order of 10, 20, 30, 40, and 50 minutes and then unloaded. The mercury had to be removed in order to examine the surface for the existence of cracks. This was accomplished by rapid evaporation in an evacuated retort at 650°F for a few minutes. This time is short enough to preserve the state of aging for repeated experiments. The surface of the specimen after the Hg had been evaporated off was usually lightly etched, sufficient to reveal the grain boundaries. In no case--examining the surface after evaporation, after repolishing, or after repolishing and etching--could any sign of surface cracks be detected.

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* Manufactured by Daytronic Corp., Dayton, Ohio. Model No. 103A-80 (linear displacement transducer); Model No. 300C (variable gain amplifier).

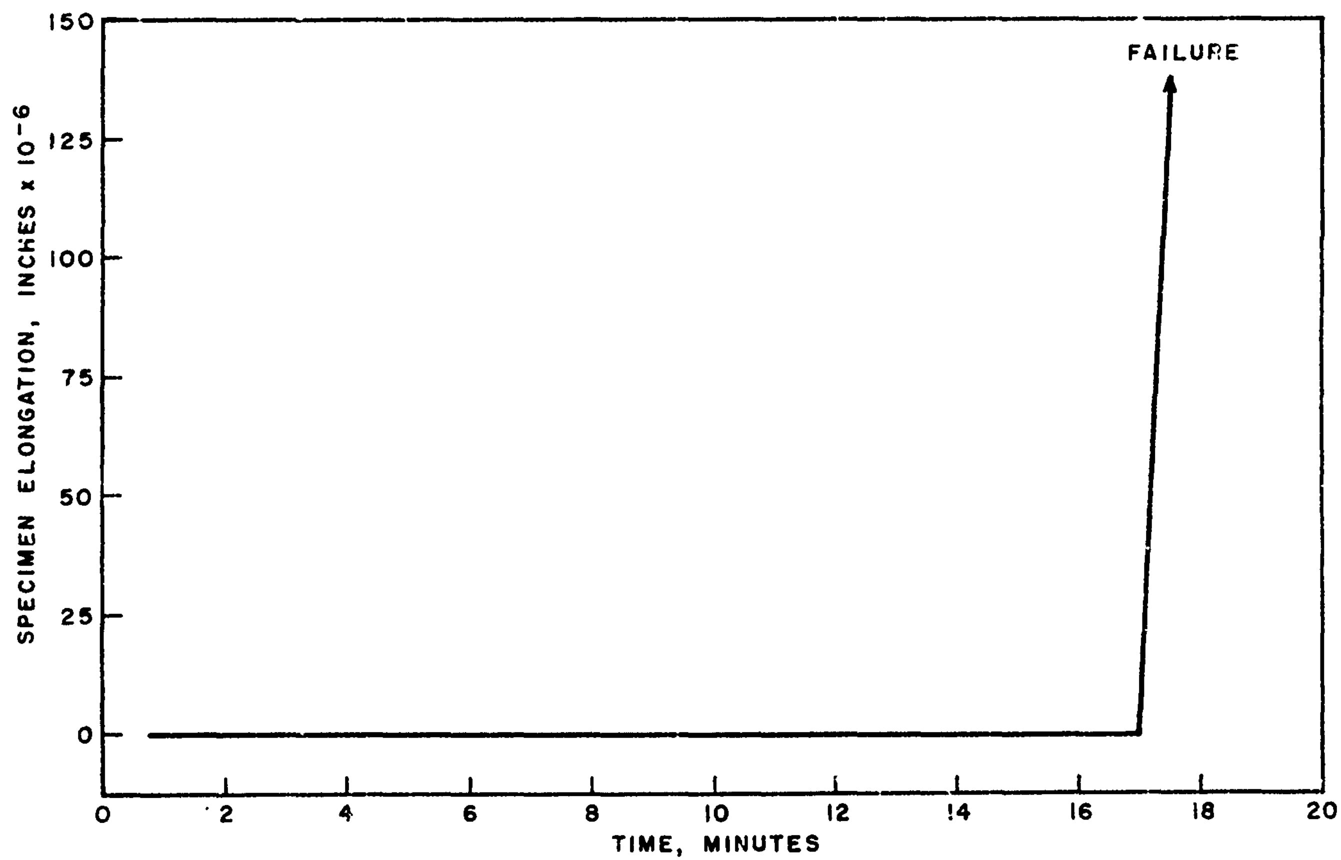

FIG. 5 - RECORD OF MICROSTRAIN LINEAR TRANSDUCER DURING DELAYED FAILURE TEST OF BERYLLIUM-COPPER SPECIMEN AT ROOM TEMPERATURE, WETTED WITH MERCURY AND UNDER A CONSTANT STRESS OF 92,000 PSI.

It seems clear from the microstrain-time measurements and the metallographic observations that the actual generation and growth of cracks is confined to the last minutes of the total time under stress to failure. It is necessary to identify other processes which are occurring over the major portion of the time and which somehow produce the conditions leading to crack initiation.

The existence of preparatory processes is revealed by a special etching procedure. A beryllium copper tensile specimen was wetted with Hg and stressed at 91,000 psi for a time just short of that expected to produce failure. The specimen was unloaded and the Hg evaporated off. In this condition there was no evidence of cracks. With repolishing and etching there was again no evidence of cracks or any other unusual feature. The specimen was then immersed in 50% nitric acid for a time sufficient to remove several mils from the thickness. This acid machining action brought certain structural features in sharp relief.

Figure 4 is a photograph of the surface previously wetted by mercury. On first examination, these surface markings appeared to be cracks transverse to the axis of loading. The action of focusing and defocusing under the microscope, however, demonstrated that they were not cracks but actually raised ridges which resisted acid attack more than the surrounding metal. Now when mercury diffuses into beryllium copper, the diffusion zone has a definitely increased resistance to acid attack. This experiment, therefore, suggests that under the influence of stress, there is locally accelerated mercury diffusion probably along certain grain boundaries. The influence of stress or associated slip band activity is revealed by the orientation of the diffusion zones with respect to the stress axis.

IV. SOME CRITICAL EXPERIMENTS IN DELAYED FAILURE

A number of simple tests have been performed to answer the following questions:

(a) To what extent does prolonged exposure of beryllium copper to liquid Hg in the unstressed condition influence the time to failure on subsequent stressing?

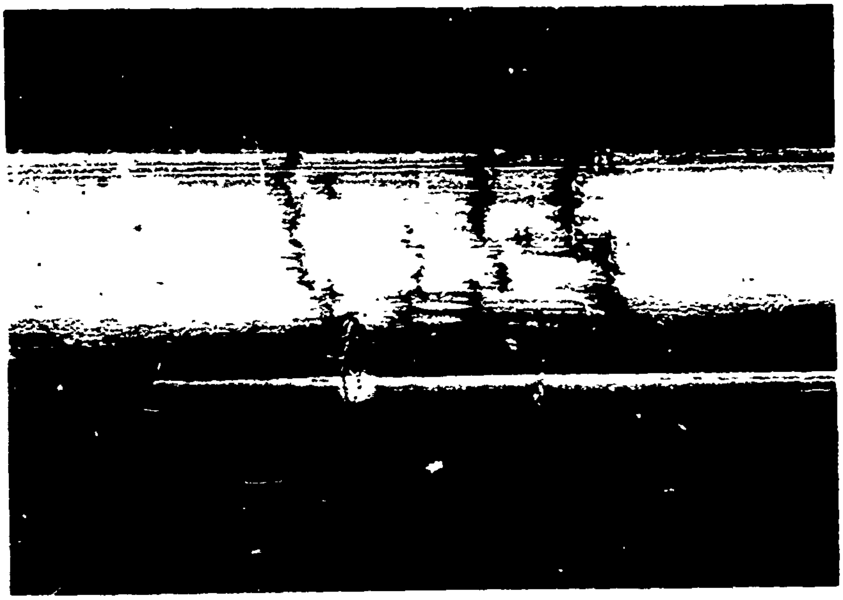

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FIG. 4 - ILLUSTRATION OF SELECTIVE INTERGRANULAR PENETRATION OF MERCURY INTO A STRESSED BERYLLIUM COPPER FATIGUE SPECIMEN. NOTE THE CRACK AT THE EDGE OF THE SPECIMEN.

(b) Is the time to failure influenced by the act of loading to the given stress level, or is this completely equivalent to wetting after the prescribed stress has been established?

(c) Does a prior stress excursion to higher than the prescribed stress level influence the time to failure at the latter stress level?

(d) To what extent does prolonged exposure of Be copper to stress in the unwetted condition influence the time to failure on subsequent wetting?

A series of tensile specimens 0.050 in. thick were prepared by hand polishing to a metallographic finish. Specimens wetted with mercury and loaded immediately to 100,000 psi failed on the average in 28 minutes. A series of specimens wetted with mercury and left unstressed for 60 minutes before loading to the same stress level failed on the average in 35 minutes. This is not a significant difference since the spread of data in both series of experiments includes both average times.

A third group of specimens were loaded to 100,000 psi and then wetted with mercury. The average failure time was 35 minutes. The remainder of the specimens were loaded to the yield point and unloaded to 100,000 psi, at which stress level they were wetted with mercury. The average failure time was 22 minutes. In this case the data were crowded to the lower limit of the other data bands, and there is probably a small but real effect from the very small amount of plastic distortion produced by the stress excursion to the yield point.

A new set of specimens was prepared from 0.125 in. thick sheet. Because the thickness was greater, the time to failure was longer and averaged 50 minutes for specimens for experiments where the specimen was loaded to 100,000 psi and then wetted within a few minutes. A second group of similar specimens were loaded to this stress level and held under sustained load for one hour before wetting with mercury. The average failure time for this group was 40 minutes. Again, the difference in average times is not significant.

In general, these experiments demonstrate that the independent prior actions of stress or exposure to liquid mercury do not influence the time to failure when stress and the wetted condition become simultaneously active.

Since all pertinent activity seems to begin at the point in time when stress and mercury act in concert, we must seek for evidence of physical changes from that point to failure. A series of experiments has been conducted with 0.125 in. thick specimens wherein they are loaded to 82,500 psi for various periods of time short of failure and then loaded to produce instantaneous failure. The wetted fracture stress after exposure to mercury under lower sustained loads can be taken as a sensitive indicator of physical changes in the surface of the specimen. The results of a large number of tests are shown in Figure 5.

These data reveal that the wetted fracture stress increases slightly but systematically under the influence of mercury wetting and sustained loading at 82,500 psi. The surface hardening curve is cut off by the incidence of cracking. This is a very interesting effect and must be weighed with the evidence for stress-induced mercury diffusion shown in an earlier section.

V. DISCUSSION

The experiments described in this report demonstrate that prior stress excursions, provided that no plastic strains are generated, and prior wetting with mercury have no influence on the delayed failure time. The conclusion may be drawn that the circumstances leading to delayed failure begin only with the simultaneous existence of a surface wetted with mercury and a stress of sufficient magnitude.

The time span of delayed failure may be separated into two parts. There is a long period during which the existence of cracks cannot be detected by microstrain devices or by direct metallographic observation. The culminating cracking period occupies only the last minute or fraction thereof.

The mechanism of delayed failure seems to be rooted in this long period of apparent inactivity. Two experimental observations indicate that mercury diffusion is active during this period. The differential attack by 50% nitric acid on a failed specimen revealed mercury-rich regions having the same geometry and relationship to applied stress that cracks possess. The small increase in wetted fracture stress during the sustained load period

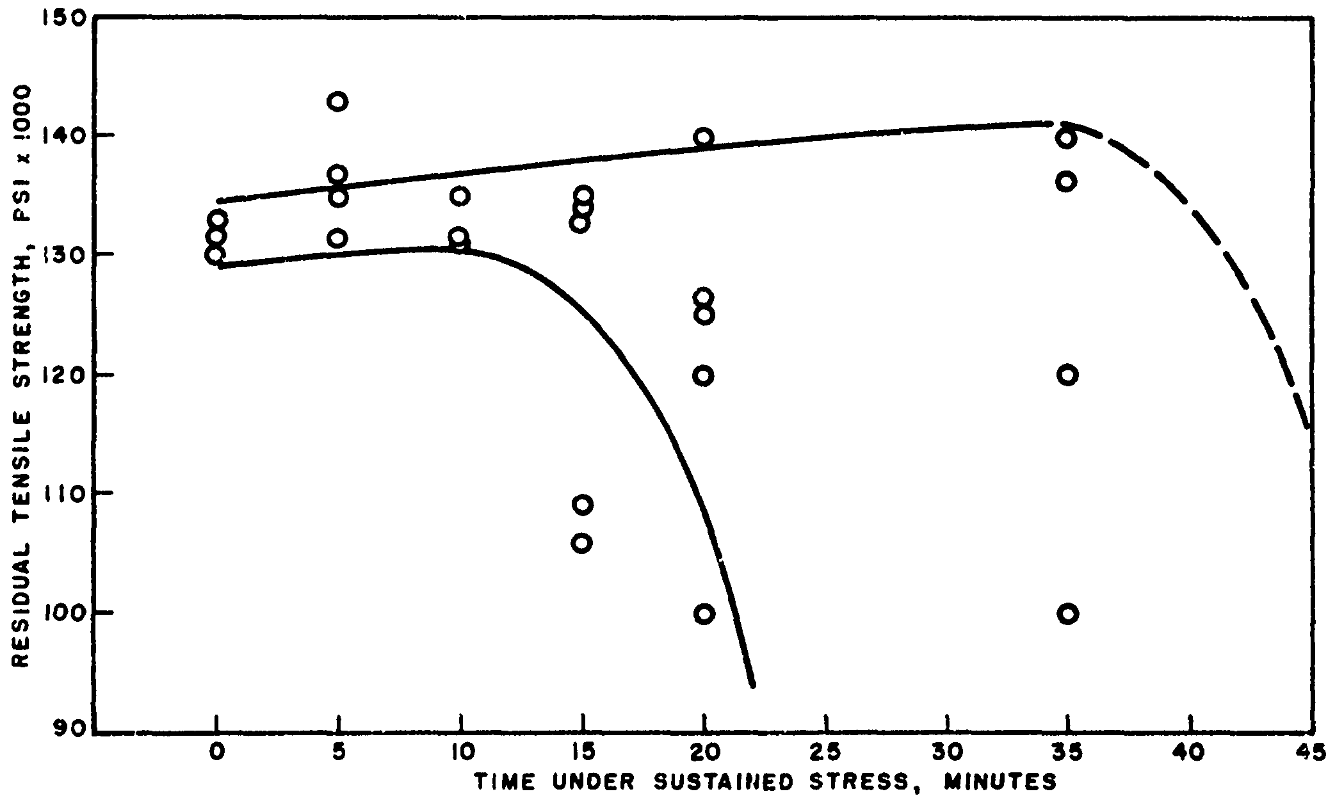

FIG. 5 - INFLUENCE OF TIME UNDER A SUSTAINED STRESS OF 82,500 PSI ON THE RESIDUAL TENSILE STRENGTH OF AGE HARDENED BERYLLIUM-COPPER WETTED WITH MERCURY.

indicates that this mercury diffusion into the surface produces a form of surface strengthening. This would mean that existing obstructed slip bands become locked by mercury atoms so that a higher stress is necessary to reactivate them. It would also imply that the increasing stress fields generated around obstructed slip bands by mercury atom aggregation would ultimately produce the critical local tensile stress in excess of the theoretical cleavage strength which leads to cracking.

VI. PERSONNEL AND LOGBOOKS

The work herein reported is the result of the cooperative efforts of Mr. H. Nichols, Associate Metallurgist, and the writer. Data are recorded in ARF logbooks No. C12787, C13011, and C13290.

Respectfully submitted,

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W. Rostoker

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