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REPORT R-1677

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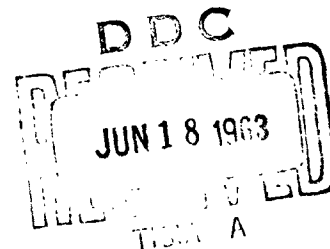
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DELAYED FRACTURE OF ALUMINUM ALLOYS BY
LIQUID ZINC AMALGAM

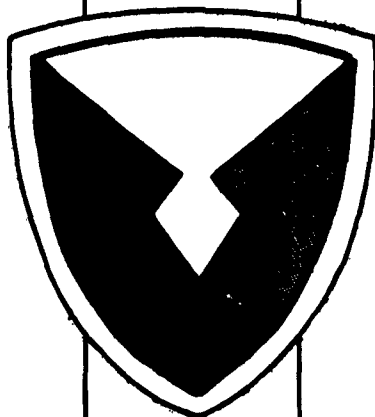
by

B. J. ROGUS

OMS Code 5010.11.80500.51.01



REPORT R-1677



APRIL 1963

PHILADELPHIA 37, PA.

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DELAYED FRACTURE OF ALUMINUM ALLOYS BY
LIQUID ZINC AMALGAM

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April 1963

OBJECT

To investigate the delayed fracture of aluminum alloys in the presence of a liquid zinc amalgam.

ABSTRACT

Delayed fracture studies were conducted on sheet specimens of aluminum alloys 2024 and 7075 in different conditions of temper upon wetting with a liquid 2% zinc amalgam. Delayed fracture data obtained at ambient temperature showed behavior characteristic of this type phenomenon in that the time to failure under sustained load varied inversely to the magnitude of applied stress. Tests on alloys 2024 and 7075 in the precipitation hardened condition revealed that embrittlement could occur rapidly at stress levels below the yield strength of the material while similar tests on these alloys in the annealed state showed that brittle fracture could not be induced at sub-yield stress levels regardless of time held at stress.

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INTRODUCTION

Occurrence of delayed fracture associated with liquid metal embrittlement has been noted previously by other investigators and was described in some detail in a book by Rostoker, McCaughey and Markus^{(1)*} With high stresses, failure upon wetting with liquid metal may be immediate. With lower stresses, however, a finite time may be required before failure occurs, the time being inversely proportional to the magnitude of applied stress. This time expended before failure occurs under constant stress is referred to as the delayed fracture time. This phenomenon has also been called static fatigue and has been noted in hydrogen embrittlement of steel as well as in stress corrosion cracking of both ferrous and non-ferrous metals.

The previous reported work on delayed failure had been done at 100° C on one aluminum alloy. This investigation was conducted in order to determine whether the phenomenon was operative at ambient temperature. In addition, more than one alloy and temper were studied.

MATERIALS

Delayed fracture tests were made on specimens machined from bare, commercial quality, 1/16 inch thick, sheet stock of aluminum alloys 2024 and 7075 in the T-3 and T-6 tempers respectively as well as in the annealed "O" condition. These alloys were selected since previous work by other investigators⁽¹⁾ had shown precipitation hardened grades of aluminum to be susceptible to liquid metal embrittlement. Average tensile properties of the alloys investigated are listed in table I.

A liquid 2% zinc amalgam was used to induce delayed failure of the alloys while in the stressed condition.

*See References

Table I. Average tensile properties of aluminum sheet material in the unwetted condition

<u>Alloy and Temper</u>	<u>Yield Strength, psi (0.2% Offset)</u>	<u>Tensile Strength psi</u>	<u>Elongation (% in 2")</u>
2024-0	11,000	33,000	22.5
2024 T-3	51,800	71,400	19.5
7075-0	15,000	34,000	18.5
7075 T-6	73,700	83,700	9.5

EXPERIMENTAL PROCEDURE

Experiments were conducted at room temperature using flat 1/16 inch thick specimens with a 2-inch gage length and 1/2-inch gage width. A table tensile machine was used which permitted stressing the specimens while held horizontally. The test machine was screw activated and was equipped with friction-type grips. Special sliding cross arm supports were designed and installed to support the jaws so that their weight would not impart any bending stresses to the specimens. Preliminary tests conducted on specimens with SR-4 strain gages on either side of the gage section confirmed that there was no bending of the samples in test and the loading was essentially uniaxial tension.

A calibrated SR-4 strain gage load cell was installed in the system to measure the applied loads. Accuracy of the load measuring system was determined to be ± 1 percent of the indicated value. A photograph of the test arrangement is shown in figure 1 while equipment used to record the time to failure is shown in figure 2. A strain analyzer and chart recorder were incorporated into the setup so that a graphical presentation of load and time was obtained. A mechanical drop-switch was activated by movement of the jaws when the test specimen broke, thereby recording the time to failure.

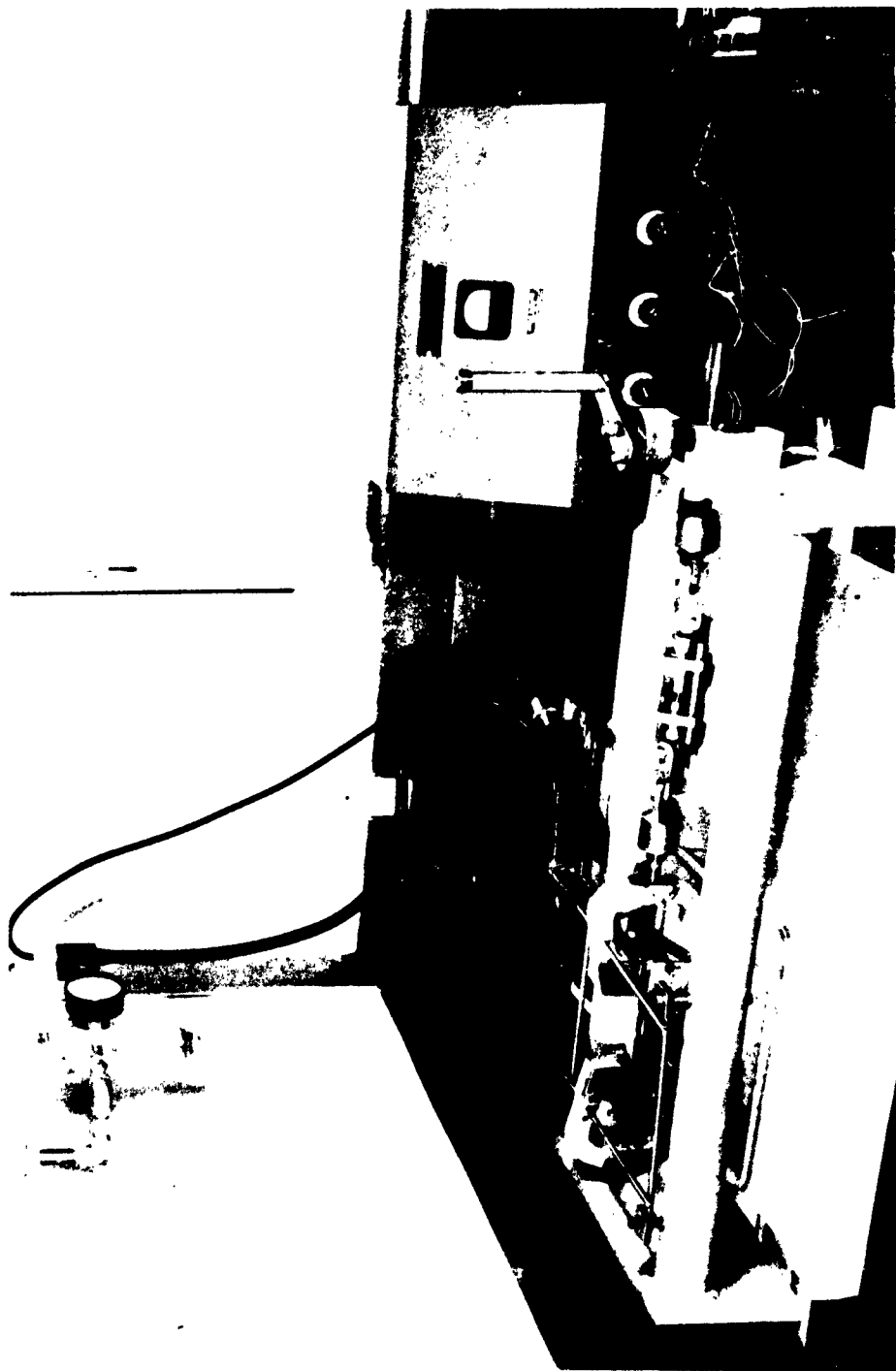


Figure 1. Tensile test machine used in the determination of delayed fracture phenomenon of aluminum alloys.

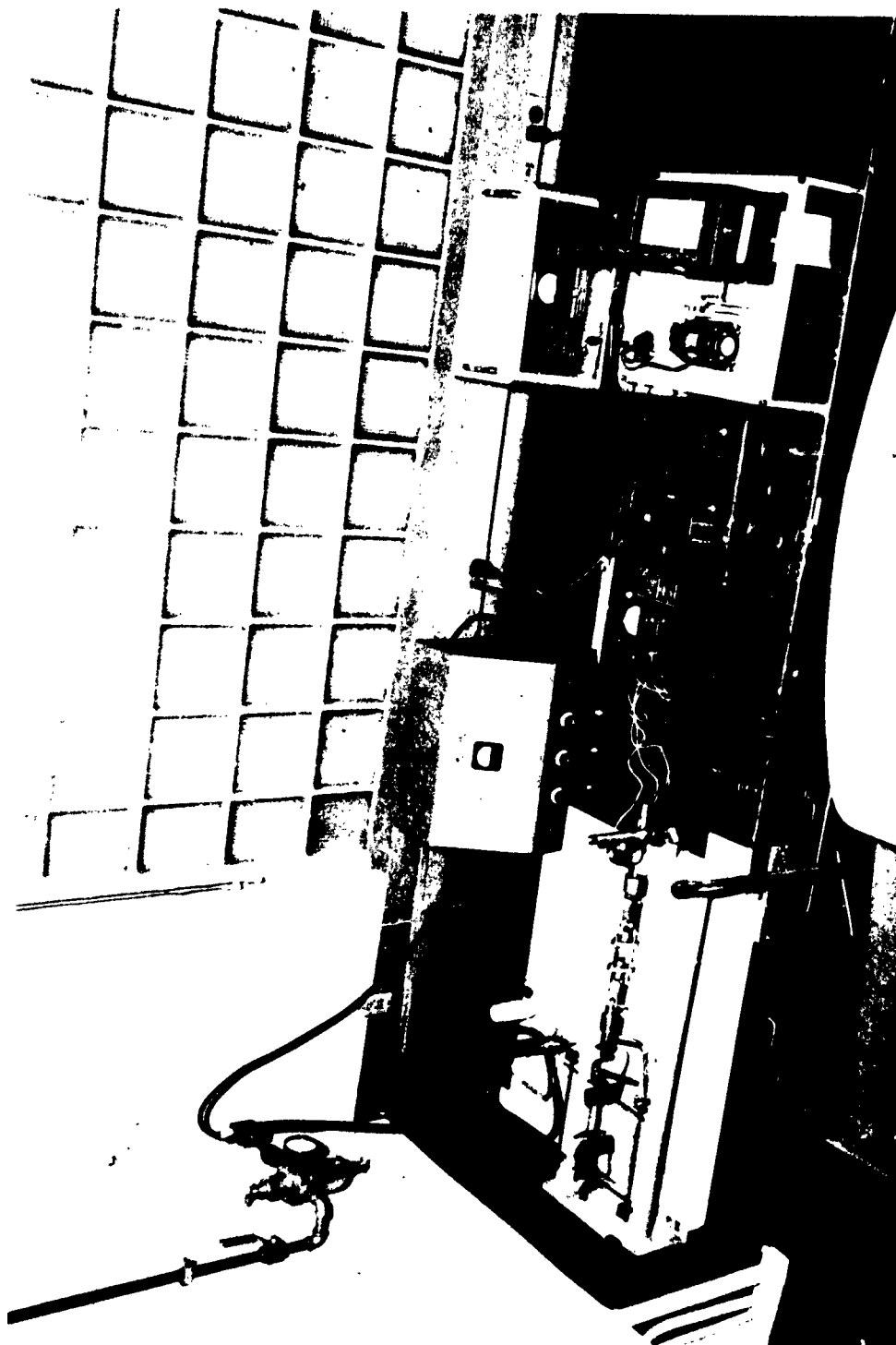


Figure 2. Test arrangement showing equipment used in the determination of the delayed fracture phenomenon on aluminum alloys.

Test procedure was as follows for the delayed fracture tests: the specimens were stressed to predetermined percents of the unwetted yield strength of the material and were then wetted in the center of the gage section. The material was tested as received or after annealing.

Wetting was accomplished both mechanically and chemically. By wetting is meant the removal or penetration of intervening oxide or other surface layers present between the aluminum metal and the liquid amalgam and the subsequent establishment of an effective true interface between the solid metal and the liquid.

For mechanical wetting, an ultrasonic probe was used, the tip of which vibrated at 28 kc. After the specimens were stressed, five drops of 2% Zn-Hg amalgam were placed on the gage section and the probe inserted into the pool. Upon activation, the vibrating tip effectively broke down the oxide layer and allowed the amalgam to wet the specimens. Effective wetting could be discerned both by the emanation of a distinct hissing sound from the amalgam pool and by visual observation of the manner in which the amalgam spread over the aluminum surface. The probe was passed through the amalgam over the gage width for 30 seconds. Care was exercised not to touch the specimen surface with the probe tip for trial tests had shown that erratic results are thereby obtained. Furthermore, the amalgam was kept away from the sides of the specimens to eliminate possible misleading edge effects.

For chemical wetting a combination of 10% sodium hydroxide solution and amalgam was used. Five drops of NaOH were placed on the center of the specimen gage section. After 30 seconds, during which time the NaOH dissolved the thin oxide film on the aluminum, five drops of amalgam were added into the center of the NaOH pool. The oxide film having been removed, the amalgam was able to make contact with the aluminum metal and thereby effect wetting.

The selection of a zinc amalgam rather than pure mercury was based on its superior wetting characteristics. Using pure mercury, it is extremely difficult to effect wetting mechanically since the mercury simply moves out from between the tip and the aluminum upon activation of the probe. This action is the result of a large contact angle between the mercury and the oxide on the

surface of the aluminum. With the addition of zinc powder to the mercury, a pasty amalgam is formed which readily can be used for wetting inasmuch as the contact angle is thereby reduced. It is believed that the presence of zinc in itself is not a required factor in the occurrence of delayed fracture phenomenon. Preliminary tests, employing chemical wetting indicated that behavior encountered with the amalgam was similar to that witnessed for pure mercury.

After the specimens had been stressed and wetted as described, times required for failure were measured. Time measurements were made from the completion of probe movement for mechanical wetting and from the instant of amalgam addition for chemical wetting.

RESULTS AND OBSERVATIONS

Preliminary to the delayed fracture studies, tests were conducted to determine the degree of embrittlement induced in the alloys by the presence of the liquid zinc amalgam. By embrittlement is meant the process wherein normally ductile metals are made to fail, in sudden manner, at stress levels below the yield strength of the material. Degree of embrittlement can be described by a ratio of the wetted fracture strength to the unwetted yield strength of the alloy. This ratio illustrates behavior best for those instances wherein brittle failure occurs at sub-yield stress levels. Average wetted fracture strength values, that is, the stress required to break a specimen immediately after wetting, are listed in table II for the alloys investigated.

Delayed fracture studies were then conducted at room temperature on the alloys 2024 and 7075 in the age hardened conditions. After the specimens were stressed to predetermined percents of the materials unwetted yield strength, the pieces were wetted with the amalgam using either mechanical or chemical means. Delayed fracture times were then recorded.

Table II. Wetted fracture strength values for aluminum alloys in the presence of a 2% zinc amalgam

Alloy	Chemical Wetting (psi)	Wetted Fracture Strength Values		Embrittle- ment Ratio
		Embrittle- ment Ratio	Mechanical Wetting (psi)	
2024-0	29,900	2.72	31,500	2.86
2024 T-3	31,800	0.62	47,000	0.91
7075-0	30,900	2.06	28,100	1.87
7075 T-6	18,000	0.24	31,300	0.42

Lower limits of stress, below which failure would not occur regardless of time, were noted for the 2024 T-3 and 7075 T-6 alloys, wetted mechanically with the probe. These limits were approximately 10,000 and 5,000 psi respectively. This lower limit is sometimes referred to as the static endurance limit.

Average values for the delayed fracture tests on alloy 2024 T-3 when wetted mechanically with the probe are given below in table III while the plot of values is shown in figure 3.

Table III. Ranges of delayed fracture time values (mins.) obtained on 1/16 inch thick 2024 T-3 aluminum wetted mechanically with a 2% zinc amalgam

	Stress Level (as percent of unwetted yield strength)								
	90	80	70	60	50	40	35	30	25
Min.	0.1	0.3	2.7	2.6	0.4	1.5	26.3	47.0	132.0
Max.	2.8	3.5	24.7	21.3	36.0	134.0	300.0	1000.0	422.0
Avg.	1.0	1.7	9.8	11.1	8.6	36.7	102.0	304.0	286.0

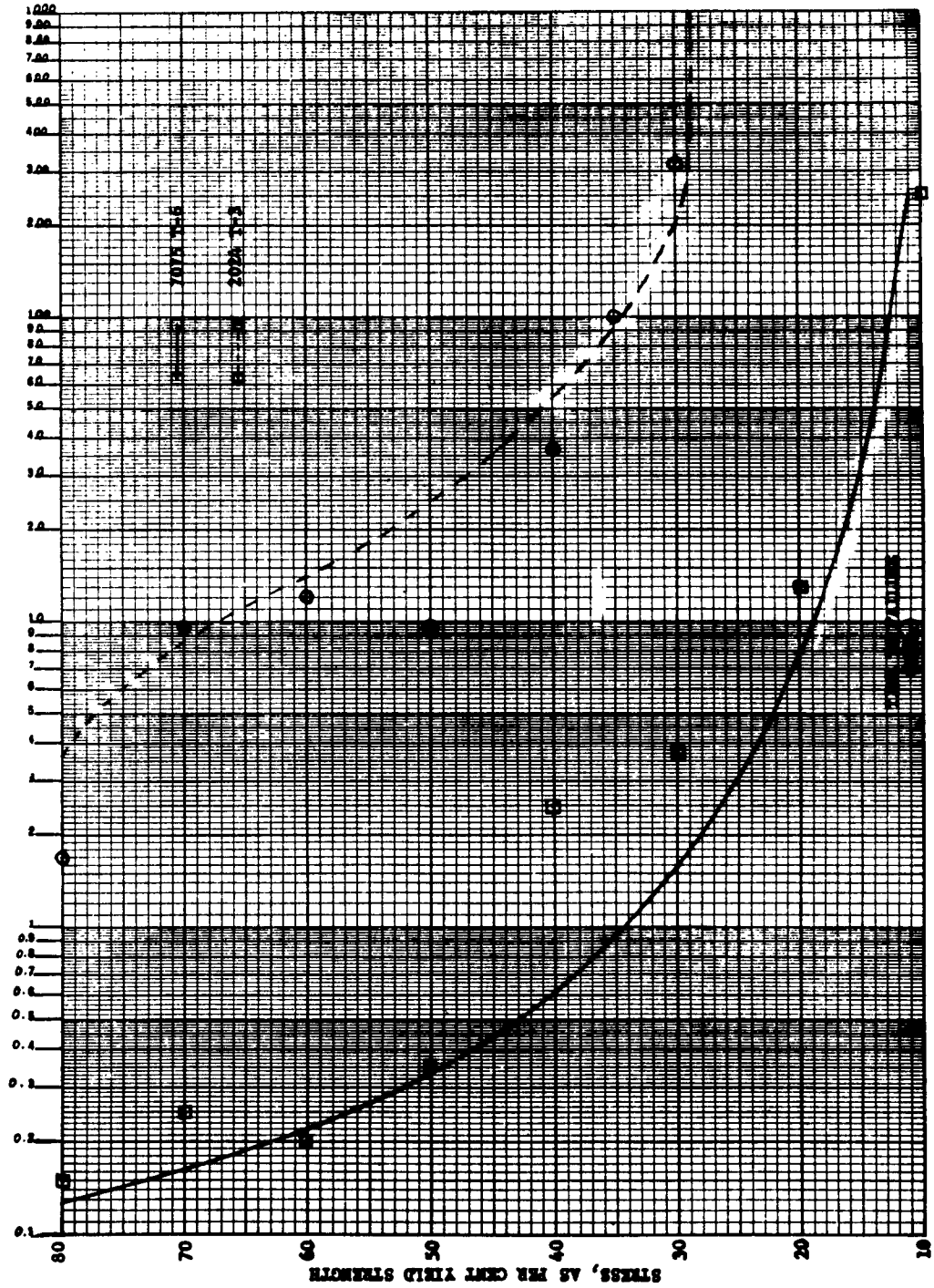


Figure 3. Times to failure of 1/16 in. thick aluminum sheet specimens wetted mechanically with 2% Zn - Hg amalgam.

Results for chemical wetting were similar in trend although the observed times to failure were generally less for corresponding stress levels. Average test values obtained on alloy 2024 T-3 wetted chemically are given in table IV and are shown plotted in figure 4.

Table IV. Ranges of delayed fracture time values (mins.) obtained on 1/16 inch thick 2024 T-3 aluminum wetted chemically with NaOH solution and a 2% zinc amalgam

	Stress Level (as percent of unwetted yield strength)										
	90	80	70	60	50	40	30	25	20	15	10
Min.	0.1	0.3	0.4	0.7	1.0	1.8	2.4	1.3	12.5	17.9	15.0
Max.	0.4	0.8	1.5	4.7	3.4	4.5	5.8	4.7	31.5	41.8	1460.0
Avg.	0.2	0.5	0.9	1.8	2.2	2.9	3.5	3.3	19.5	32.0	662.0

Similar tests were made on alloy 7075 in the T-6 temper. For both methods of wetting it was noted that times to failure were less for corresponding stress levels for the 7075 T-6 alloy than for the 2024 T-3 material. Delayed fracture data for alloy 7075 T-6 as obtained with mechanical wetting are shown in table V and are plotted in figure 3.

Table V. Ranges of delayed fracture time values (mins.) obtained on 1/16 inch thick 7075 T-6 aluminum wetted mechanically with a 2% zinc amalgam

	Stress Level (as percent of unwetted yield strength)							
	80	70	60	50	40	30	20	10
Min.	0.1	0.2	0.1	0.2	0.2	0.1	2.0	48.0
Max.	0.2	0.3	0.3	0.7	6.7	10.7	23.3	1140.0
Avg.	0.15	0.25	0.2	0.3	2.5	3.7	12.9	252.0

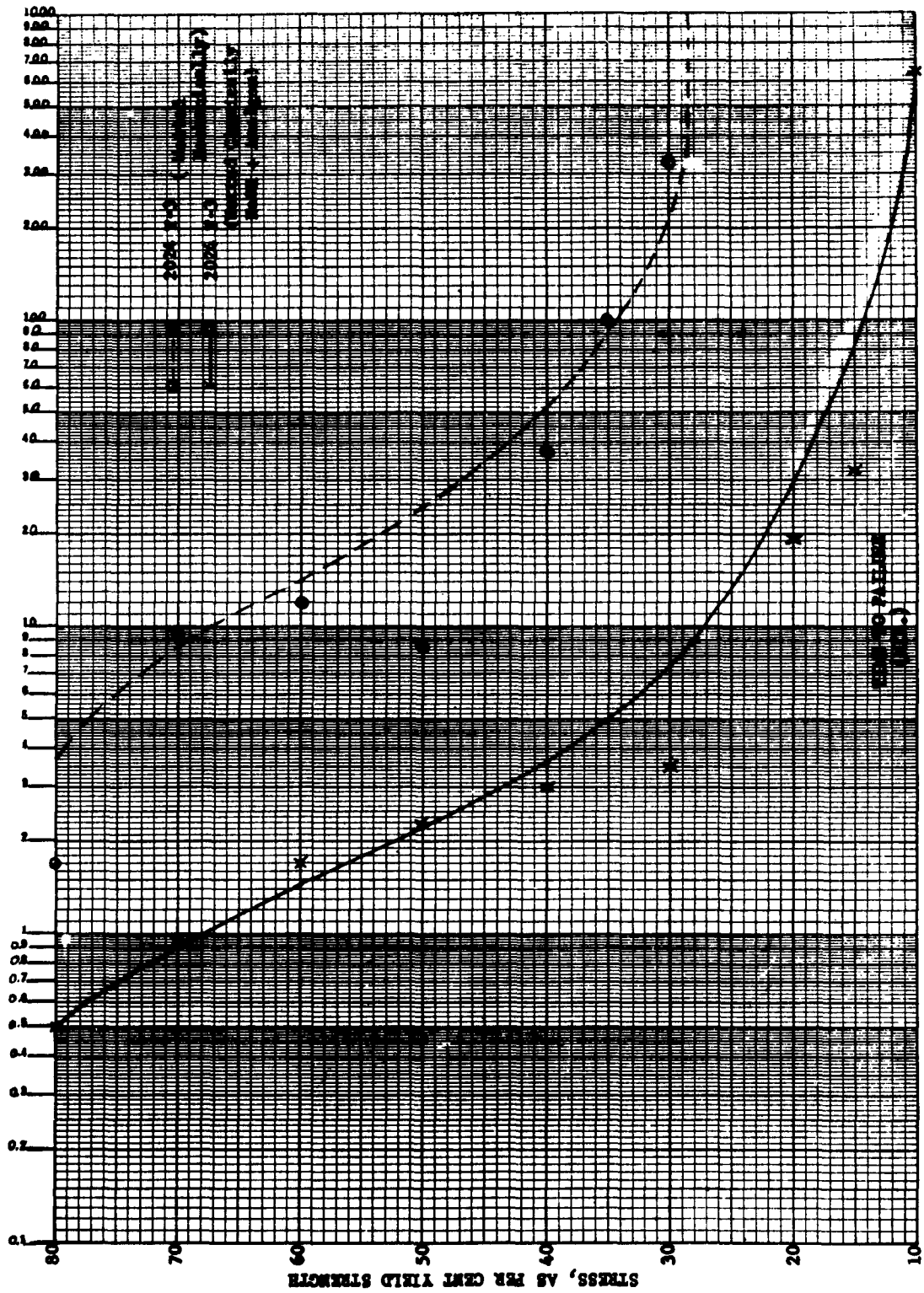


Figure 4. Times to failure of 1/16 in. thick 2024 T-3 aluminum sheet specimens wetted with 2% Zn - Hg amalgam.

Chemical wetting tests for this alloy were limited. Only sufficient specimens were tested to confirm the occurrence of behavior similar to that noted for mechanical wetting.

Extensive delayed fracture tests were not made on the alloys in the annealed or "O" condition since no embrittlement could be effected below the yield strength of the material regardless of the amount of time held stressed while in the wetted condition. A limited number of tests were conducted, however, at stress levels above the yield strength for alloy 2024-0. Average delayed fracture times were obtained on 1/16 inch thick 2024-0 material at stress levels indicated in table VI. The specimens were wetted mechanically. Unwetted yield strength for the material was 11,000 psi and wetted fracture strength, as determined immediately after wetting, was 31,500 psi.

Table VI. Delayed fracture times for
2024-0 aluminum alloy

<u>Stress, psi</u>	<u>Delayed Fracture Times (mins.)</u>
32,000	4.7
31,000	21.0
30,000	180
28,000	715

The tests suggested that the delayed fracture phenomenon appears operative even in material for which the wetted fracture strength is greater than the yield strength. Furthermore, the process is not peculiar only to age hardened states for which embrittlement ratios less than unity are obtained, but can occur in annealed material at elevated stress levels. It should be noted, however, that this test series differed somewhat from those for the precipitation hardened conditions. Inasmuch as the applied stresses were above the materials yield strength, additional straining of the specimens had to be effected to retain the cited stress levels.

DISCUSSION

Experimental work showed that delayed fracture of aluminum alloys can be effected at room temperature. Several aspects of the phenomenon were investigated.

Normally ductile precipitation hardened aluminum alloys were made to fail in a sudden manner. Analysis of the load-time plots for the delayed fracture tests showed no decrease in stress with time of loading up to the instant of failure. This fact indicated that, within the sensitivity of the equipment used, fracture was a catastrophic event upon inception.

Microstructural examinations were made of specimens from alloys 2024 and 7075 in the age hardened states upon embrittlement with the zinc amalgam. No evidence of deformation was seen at the fractured edges (see figures 5 and 6). This observation is typical of true embrittlement and is in agreement with the work of other investigators⁽²⁾ which showed no noticeable distortion along the fractured edges of embrittled twin crystals of brass (see figure 7).

In the present investigation, examination of the failed areas of the delayed fracture specimens of aluminum alloys showed the cracking for the precipitation hardened conditions to be both intergranular and transgranular (figures 5, 6 and 8). For the annealed material, the grain boundaries themselves were not clearly evident and the cracking associated with the failed edges appeared more localized. (figures 9 and 10).

Previous investigations (1, 2, 3) have related liquid metal embrittlement to an effective reduction in surface energies brought about by the presence of liquid metal. Once microcracks result in the stressed specimen, a reduction of energy can be effected through the continued formation of new crack surfaces, causing eventual complete failure, and thereby releasing the elastic energy present in the material as a result of stressing. In this light, the observed delayed fracture time may be associated only with the creation of conditions causing the first microcracks to occur.

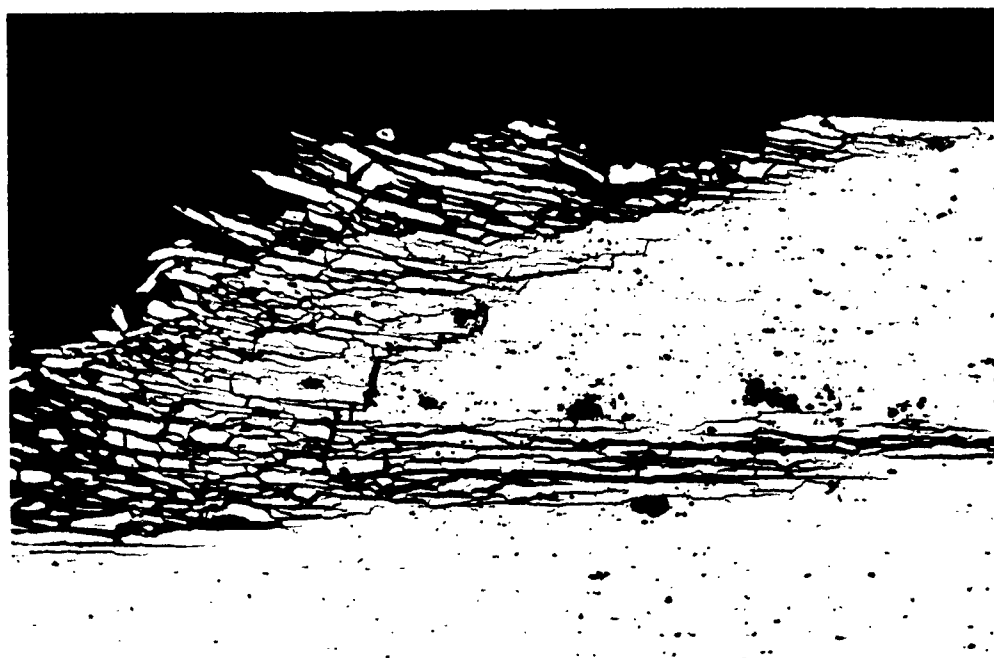


500X



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Figure 5. Photomicrographs of failed edges of 1/16 in. thick 2024 T-3 aluminum after having been wetted with 2% Zn - Hg amalgam and stressed to failure. Note lack of cold work or distortion along failure.



100X



500X

Figure 6. Photomicrographs showing failed edges of aluminum alloy 7075 T-6 which had been wetted mechanically with 2% Zn - Hg amalgam. Note crack propagation pattern and lack of distortion along failed edges.



500X

Figure 7. Intersection of twin interfaces with an intergranular crack in 70/30 brass embrittled by wetting with mercury. (After Rostoker²)

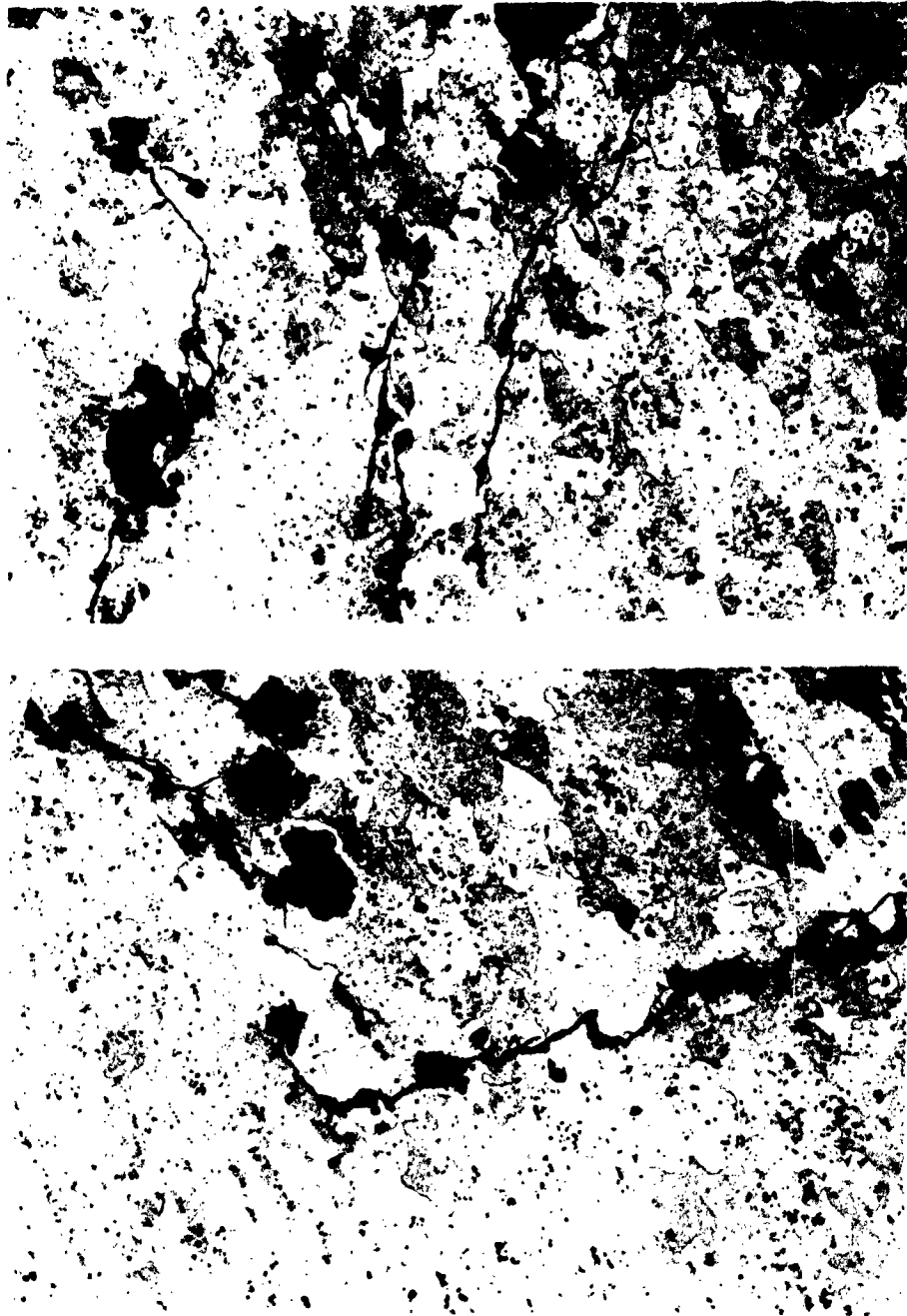
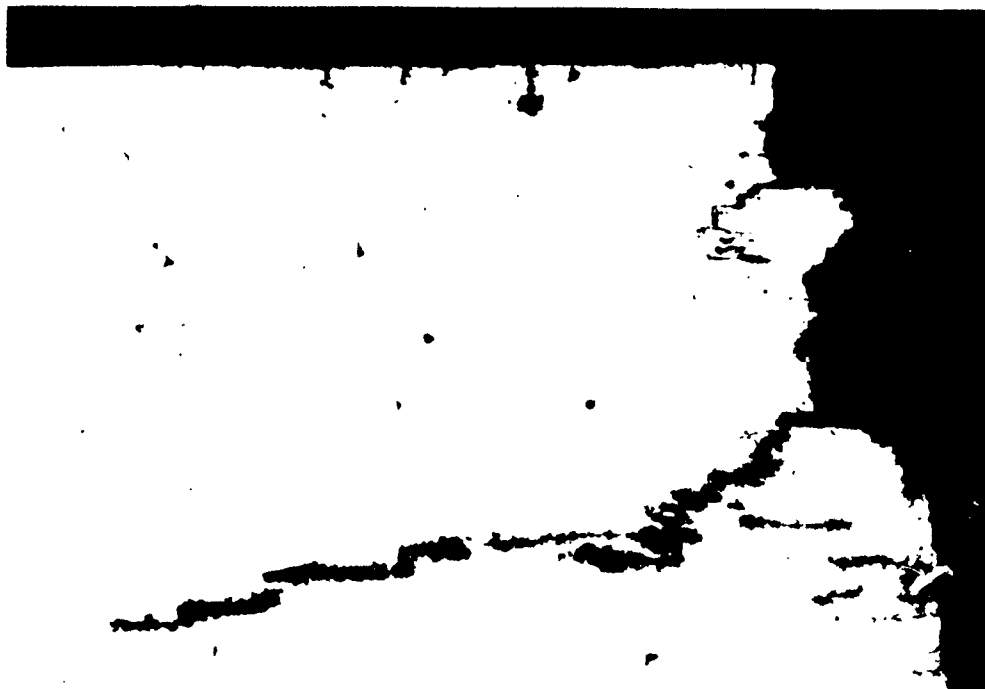
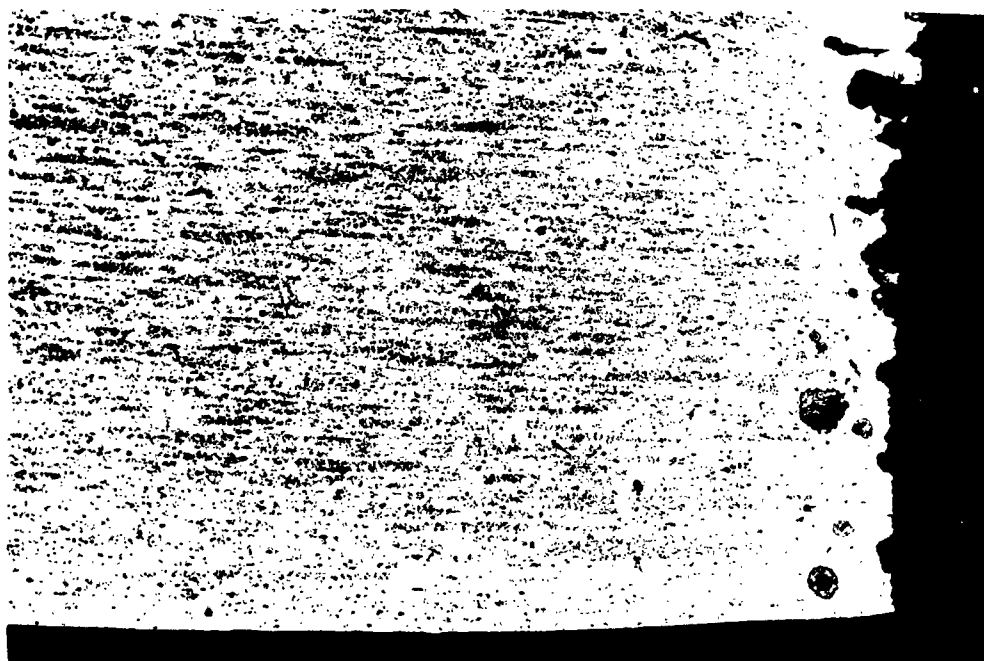


Figure 8. Photomicrographs showing cracks along failure of 1/16 in. thick 2024 T-3 aluminum after wetting ultrasonically with 2% Zn - Hg amalgam. Note intergranular and transgranular cracking. Magnification 100X, Keller's etch.

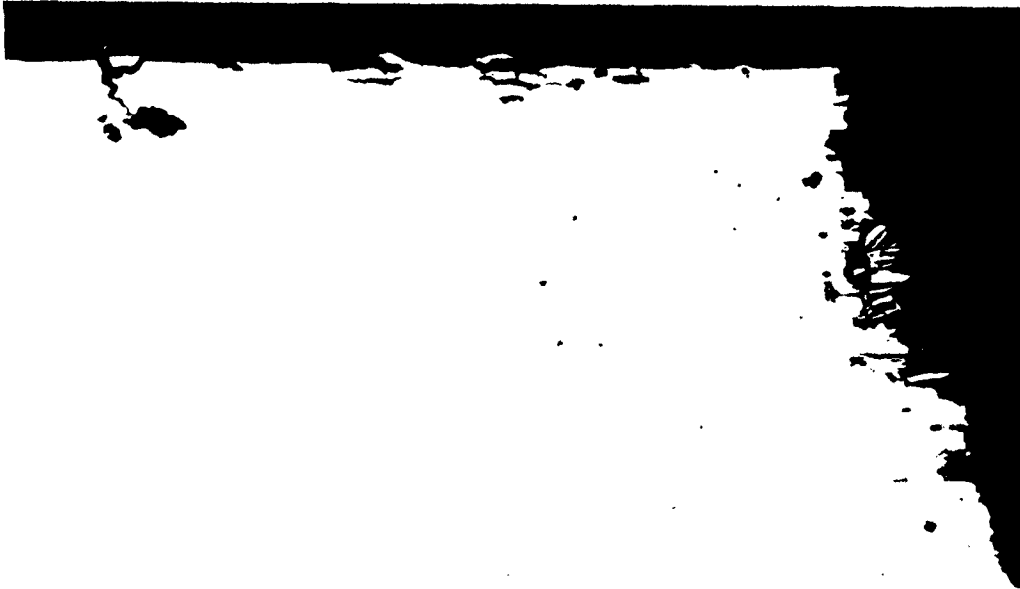


Unetched



Keller's etch.

Figure 9. Failed edges of annealed aluminum alloy 2024-O which had been wetted mechanically with 2% Zn - Hg amalgam.



Unetched



Keller's etch.

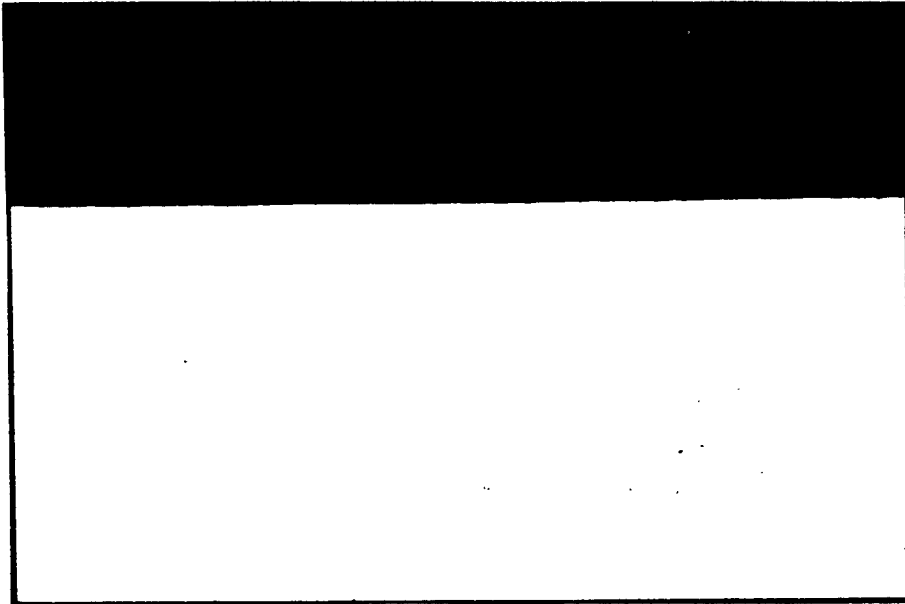
Figure 10. Failed edges of annealed aluminum alloy 7075-O which had been wetted mechanically with 2% Zn - Hg amalgam.

Recent theories of fracture nucleation maintain that microcracks can be generated when a critical number of dislocations on a slip plane pile up against a barrier, such as a grain boundary, as the result of active shear stresses. Experiments have corroborated this in that correlations between wetted fracture strength and grain size have been established (3, 4). In view of this, the delayed fracture time might be that required for the dislocations to coalesce at the barriers to form microcracks. Therefore, if specimens were stressed for prolonged periods prior to wetting, less time may be expended for fracture to occur upon wetting with amalgam. To investigate this aspect, tests were conducted on 2024 T-3 material to determine whether stressing for sustained time intervals prior to wetting was of any consequence.

Specimens were stressed to 20,000 psi and were wetted immediately while others were held stressed as long as 120 hours before wetting. However, analysis of obtained delayed fracture times failed to indicate any significant difference between tests on specimens wetted immediately after stressing and those which had been stressed for prolonged periods before wetting.

That delayed fracture is not the result of a continued chemical attack of the aluminum by the liquid amalgam was shown in two ways. Specimens from alloy 2024 T-3 were wetted with amalgam using the ultrasonic probe and were then dewetted immediately by washing with water. The pieces were then subjected to microscopic examination to detect the extent of any possible amalgam penetration. As shown in the photomicrographs, see figure 11, no penetration was evident within the resolution of the microscopic equipment used.

Moreover, it was noted that there was no detectable degradation of properties during delayed fracture tests up to the instant of failure. Specimens from alloy 2024 T-3 material which had been wetted and held stressed for time intervals up to the expected instant of fracture, as determined by previous similar tests, were removed from the test machine and dewetted by washing with water. The pieces were then subjected to conventional tensile tests. Mechanical properties obtained on these specimens were comparable to those obtained on unwetted material. This fact was in agreement with the previously mentioned observation on the catastrophic nature of fracture in that there was no decrease in sustained load up to the instant of failure. Typical results for specimens which had been dewetted were as follows:



Unetched



Keller's etch.

Figure 11. Photomicrographs depicting edge of 1/16 in. thick 2024 T-3 aluminum which had been wetted with 2% Zn - Hg amalgam and probe and then dewetted immediately. Transverse to rolling direction. Magnification 200X.

	<u>Yield Strength, psi</u> <u>(0.2% Offset)</u>	<u>Ultimate Tensile</u> <u>Strength, psi</u>	<u>Elongation</u> <u>% in 2"</u>
Unwetted	50,200	70,900	20.0
	51,200	70,600	20.5
After wetting and holding 24 hours unstressed and then dewetted	51,300	70,200	19.5
	51,500	70,900	20.0

Several facets of the delayed fracture process for aluminum alloys were clarified by observations noted in this work.

Tests showed that a liquid metal wetting agent must be present for the phenomenon to occur. This was indicated by the fact that unwetted specimens could be stressed for long periods of time without failure, whereas similarly stressed specimens fractured shortly after wetting. Secondly, the initiation of delayed fracture appears to be associated with an interaction between the solid and liquid metals at the interface. This aspect was illustrated by the facts that no penetration of the amalgam into the aluminum base metal was noted as a result of the wetting process. Moreover, no permanent damage was recorded on previously wetted specimens which had been dewetted and then tensile tested. Conditions associated with crack initiation and fracture propagation appear to be explained best in terms of the reduced surface energies brought about by the presence of liquid metal.

CONCLUSIONS

Delayed fracture of aluminum alloys 2024 T-3 and 7075 T-6 can be effected at ambient temperature in the presence of a liquid zinc amalgam.

Delayed fracture occurrence is not limited to precipitation hardened states. Delayed failure can be effected on alloy 2024 in the annealed condition although only at stress levels significantly higher than the annealed yield strength.

Other factors which influence delayed fracture behavior include the particular alloy type, prior thermal history of the aluminum, and mode of wetting arrangements used.

Application of stress for prolonged periods of time before wetting is effected does not appear to influence the time required for failure after wetting.

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AD- R-1677 Frankford Arsenal, Research and Development Group, Philadelphia 37, Pa. DELATED FRACTURE OF ALUMINUM ALLOYS BY LIQUID ZINC AMALGAM by R. J. Rogus Report R-1677, April 1963, 27 pp incl illus. OAS Code 5010.11.80500.51.01 UNCLASSIFIED Delayed fracture studies were conducted on sheet specimens of aluminum alloys 2024 and 7075 in different conditions of temper upon wetting with a liquid 2% zinc amalgam. Delayed fracture data obtained at ambient temperature showed behavior characteristics of this type phenomenon in that the time to failure under sustained load varied inversely to the magnitude of applied stress. Tests on alloys 2024 and 7075 in the precipitation hardened condition revealed that embrittlement could occur rapidly at stress levels below the yield strength of the material while similar tests on these alloys in the annealed state showed that brittle fracture could not be induced at sub-yield stress levels regardless of time held at stress. DISTRIBUTION LIMITATIONS: None, obtain copies from ASTIA. UNCLASSIFIED	AD- R-1677 Frankford Arsenal, Research and Development Group, Philadelphia 37, Pa. DELATED FRACTURE OF ALUMINUM ALLOYS BY LIQUID ZINC AMALGAM by R. J. Rogus Report R-1677, April 1963, 27 pp incl illus. OAS Code 5010.11.80500.51.01 UNCLASSIFIED Delayed fracture studies were conducted on sheet specimens of aluminum alloys 2024 and 7075 in different conditions of temper upon wetting with a liquid 2% zinc amalgam. Delayed fracture data obtained at ambient temperature showed behavior characteristics of this type phenomenon in that the time to failure under sustained load varied inversely to the magnitude of applied stress. Tests on alloys 2024 and 7075 in the precipitation hardened condition revealed that embrittlement could occur rapidly at stress levels below the yield strength of the material while similar tests on these alloys in the annealed state showed that brittle fracture could not be induced at sub-yield stress levels regardless of time held at stress. DISTRIBUTION LIMITATIONS: None, obtain copies from ASTIA. UNCLASSIFIED
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