EMBRITTLEMENT OF METALS

BY ORGANIC LIQUIDS

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Submitted to:

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ABSTRACT

It is shown that the Rebinder effect involving the embrittlement of steel by organic liquids is in reality due to water absorption by the organic species. The organic liquids which normally cause early fatigue do not produce this effect in the presence of a strong dehydration agent.
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I. INTRODUCTION

This is a quarterly report summarizing progress in one portion of a composite program entitled "Fracture of Metals," identified under Contract No. DA-11-ORD-022-3108. This report covers the period September 1, 1964 to November 30, 1964.

This portion of the program on "Fracture of Metals" is directed to explore the existence and nature of embrittlement which might be produced by concurrent exposure of metals to organic liquids and tensile stresses. Organic liquids are defined for present purposes as pure liquid species, miscible liquid species, and solutions of solids in liquids. Embrittlement constitutes the premature incidence of cracking as experienced under continuously increasing load, static loading, or dynamic (cyclic) loading. "Premature" implies a lower maximum load, a shorter time, or fewer cycles than would be expected for the material stressed in air.

A special test has been developed to provide a broad scale upon which to measure embrittlement. This involves the use of sheet steel heat-treated to 44 Rc (about 200,000 psi yield strength) which has been deeply notched from both edges. The specimen is stressed by a combination of static and dynamic loads--12,500 psi static plus 4370 psi dynamic. This combination represents a condition below the endurance limit where this steel in this specimen geometry will sustain the combined static and cyclic loading indefinitely when surrounded only by normal air environment.

Embrittlement is induced by immersing the notched region in a candidate liquid using various pad or cup arrangements for containment. Under such circumstances failures can be made to occur in as little as 500,000 cycles. Testing is usually stopped for practical reasons if failure.
has not occurred in six to seven million cycles. If at that time the reservoir still contains liquid, this liquid species is adjudged to be nonembrittling. This caution is necessary because many of the candidates are volatile and in any given test run the sealing system may have been imperfect.

II. USE OF DEHYDRATION AGENTS

The results presented in the last quarterly report showed that when no attempt was made to seal off the candidate liquid around the test specimen from ambient air, the number of cycles to failure was quite erratic. It became apparent that water absorption was probably an interfering factor. The next question was to what extent the wetted pad, carefully wrapped with polyethylene and bound to the specimen with adhesive tape, was safe against water absorption since during spring and summer months this type of test scheme had also shown some erratic results.

The approach taken was to incorporate in the wrapped capsule around the notched zone of the specimen a quantity of anhydrous CaSO$_4$ known under the trade name of "Drierite." This is a strong dehydrating agent and likely to be otherwise inert to organic liquids. Normally, the laboratory grade of this material comes in 1/16-1/8 in. pieces.

Using Drierite as-received, the failure times in the presence of ethyl, methyl, and butyl alcohol reproduced prior results very well and the specimen-to-specimen variation decreased substantially. To improve the effectiveness of anhydrous CaSO$_4$ still further, a quantity was ground to fine powder and incorporated into test capsules. The first tests were run using methyl alcohol, which according to previous work failed in about 600,000 cycles. In the presence of fine ground anhydrous CaSO$_4$ failure did not occur in 9,000,000 cycles. Similar results were also obtained with ethyl and butyl alcohol.

The indication is that with very effective dehydration, the influence of organic liquids may be entirely precluded and that the previous trends involving carbon chain length primarily reflect relative absorption capacities or absorption rates for water vapor. To substantiate this, a
number of liquid species previously identified as very effective were rerun in the presence of finely ground Drierite. These included:

- n-propyl ether
- acetic acid
- isoamyl alcohol
- oleic acid
- dibutyl mercury
- triethyl aluminum in light mineral oil
- chlorobenzene
- 10% phenol in light mineral oil
- triethyl boron in light mineral oil
- benzonitrile
- diphenyl magnesium in heptane

In all instances cycling continued beyond 7,000,000 cycles without failure. It should be noted that isoamyl alcohol and oleic acid were the basis for the discovery of the Rebinde effect on steel reported by Rebinde and his associates. The conclusion seems inescapable that water remains the sole agent for embrittlement in all of these experiments. Furthermore, it seems that very small amounts of water are very effective and that larger amounts are relatively less effective, according to a "rate of increase of embrittlement with concentration" criterion.

One anomalous behavior has been discovered. The use of n-amyl mercaptan (C₅H₁₁S) in conjunction with finely ground Drierite gave failures in two separate tests of 1,400,000 and 1,401,000 cycles. These results will be checked with repeated experiments.

III. DEGRADATION OF MINERAL OILS

Since mineral oils are conventional lubricants, one might expect that the presence of water-absorbing soluble additives might act to degrade the fatigue performance of engineering components immersed therein. The final phase of this program will undertake a preliminary evaluation of this
possibility through the addition of small amounts of organic liquids which are completely miscible both in mineral oil and water. The binary solutions of mineral oil and additive will be presaturated with water before use in fatigue testing.

IV. PERSONNEL AND LOGBOOK RECORDS

The work reported is the result of collaboration of the writer with Mr. H. Nichols, Associate Metallurgist. Data are recorded in ITRI Logbook No. 14245.

Respectfully submitted

[Signature]
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