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| TITLE | | |
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| ETHYLENE OXIDE FUEL CONTAINING $\frac{1}{2}$ OF 1% CARBON DISULFIDE: EFFECT UPON WELDED 61ST-6 ALUMINUM ALLOY | | |
| REPORT NO. MSD -3078 | DATE 12-4-56 | MODEL NO. XQ-5 |
| SUBMITTED UNDER (CONTRACT, SPEC., ETC.) | | |
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Form 375-2 LOCKHEED AIRCRAFT CORPORATION, CALIFORNIA DIVISION

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LOCKHEED AIRCRAFT CORPORATION
MISSILE SYSTEMS DIVISION

⑬ 14 Agency LMSD-3078

ENGINEERING TEST DEPARTMENT (72-30)

DATED December 4, 1956

Chemical-Metallurgical Section, Group 41

REF: SM/763

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MODEL XQ-5

REQUESTED BY J. H. Hummell

CHARGE 3-13/1-1362-02

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⑰ ETHYLENE OXIDE FUEL CONTAINING 1% OF 1% CARBON DISULFIDE: EFFECT UPON WELDED 61ST-6 ALUMINUM ALLOY

OBJECT:

The purpose of this investigation was to determine the effect of liquid ethylene oxide on 61S Aluminum Alloy which had been welded and heat treated to the T-6 condition. It was desired to ascertain if corrosion occurred and whether such corrosion applied to the entire assembly, or to the welded portion or both. It was also desired to determine whether the assembly acted as a catalyst to cause polymerization of the ethylene oxide.

INTRODUCTION:

It is planned by the XQ-5 Project to use 61S Aluminum Alloy to fabricate fuel tanks to contain ethylene oxide which will be used to power a motor-generator for use in a missile. The tank will be of welded construction and will be heat treated to the T-6 condition following the welding. The original test request specified that determinations should be made on the alloy in both the T-4 and T-6 condition. Later advice from the XQ-5 Project stated that the fuel tank was treated to the T-6 condition and tests on T-4 alloy would not be required.

CONCLUSIONS:

1. The original specimens, as received, were quite poorly made. After exposure to liquid ethylene oxide, a comparison was made with an unexposed sample, and there visually appeared to be little difference between the 61ST-6 specimens.
2. Exposure of iridited specimens of welded 61ST-6 to liquid ethylene oxide resulted in an almost complete bleaching out of the normal "gold" iridite color.
3. All specimens exposed showed a very small increase in weight. The iridited specimens appeared superior, their weight gain being 0.00793% as compared to 0.0204% for the plain specimens.

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CONCLUSIONS: (Continued)

4. Some polymerization occurred with both plain and iridited specimens. The iridited specimens were superior in this respect, showing a polymerization residue of 0.13% as compared to 0.27% for the plain specimens.
5. Wyandotte Chemicals Corporation have probably done more investigative work on the effect of ethylene oxide on various materials than any other concern in this country. They found that polymerization residue was a good criterion for the evaluation of different materials for use with ethylene oxide. They found that in general, polymerization residue increases with increase in temperature. After considerable work, they arrived at the following system for rating metals and alloys:

Polymerization Residue -

| | | |
|-------------------|---|----------------|
| Less than 0.15% | - | Excellent |
| From 0.15 - 0.30% | - | Fair |
| Greater than 0.31 | - | Unsatisfactory |

This system is based on a test period of four weeks and test temperatures of both 160°F and 86°F.

While our test temperature was 150°F, our test period was only one week, so that we can draw no direct conclusions since the test times were different. One would expect polymerization residue to increase with time. Wyandotte, however, found that results of 26-week tests were only slightly higher than those obtained at four weeks. They did not develop any information giving a comparison of one and four-week tests.

PROCEDURE:1. Samples:

Samples were supplied by the IQ-5 Project. They were 1" wide x 3" long and 0.032" in thickness. The weld was located one inch from one end. The specimens were stamped with a metal stamp for identification and a small hole drilled in the end of the specimen opposite the weld end. This was for convenience in cleaning, iriditing, etc. The thickness of metal used in the fuel tank is the same as that of the samples.

The samples were very poorly prepared. They showed excessive weld build-up on one side and either excessive penetration or no penetration on the other. Further, all welds showed cracks through the center on the reverse side. The cleaning and iriditing also were very poorly done. The surface was rough and appeared to be etched by having been left too long in a caustic solution.

PROCEDURE: (Continued)1. Samples: (Continued)

Surface condition of specimens in tests of this type is extremely important, since where change in weight of the specimens is very small, a measure of the corrosive attack can frequently be made by visual comparison of surfaces. Surfaces of these specimens were in such poor condition to start with that this possibility was ruled out. The iridite coating was so poorly done that it was removed and a new coating applied in this laboratory. Figure 1 shows an enlarged view of the reverse side of one specimen. It clearly shows the excessive weld penetration in some areas and lack of penetration in others. The crack through the center of the weld is also plainly visible. This photograph is representative of the condition of all the specimens.

2. Equipment:

The pressure vessels used in the test work have been previously described (MSD Report No. 1817). Figures 2 and 3 show photos of the test equipment. Figure 3 shows the retaining nut holding the aluminum rupture disc, designed to blow out in case excessive pressure develops.

3. Tests:

Three specimens of plain alloy and three which had been iridited were available. One plain specimen and one iridited specimen were reserved as controls. Each test consisted of two specimens and the plain and iridited alloy were run separately.

The weighed specimens were placed in the pressure vessel which was then connected to the ethylene oxide storage tank. The storage tank was equipped with a siphon tube which allowed delivery of liquid ethylene oxide when the tank valve was opened. Ethylene oxide was then run into the pressure vessel to a mark somewhat above a predetermined level. The work was conducted in the fume hood since the ethylene oxide boils below room temperature. When sufficient ethylene oxide had boiled off to reduce the liquid level to the proper point, an amount of carbon disulphide sufficient to give $\frac{1}{4}$ of 1% based on the weight of the ethylene oxide was added from a pipette and the mixture stirred with a glass rod. The cap of the pressure vessel was then screwed in place and tightened.

The unit was then removed from the hood, placed in a vise and tightened further using a 2 $\frac{1}{2}$ " pipe wrench. The unit was checked for leaks and placed in a thermostatically controlled oven at 150°F for one week.

PROCEDURE: (Continued)

3. Tests: (Continued)

Before closing the bomb, the threads on both body and cap had been coated with aquadag which is a water suspension of colloidal graphite. This coating was allowed to dry thoroughly before the bomb was used.

When the test period was completed, the liquid ethylene oxide was discharged into a weighed 50 ml. beaker in several steps. The ethylene oxide was allowed to evaporate and the beaker then weighed and the residue determined by difference. When all the ethylene oxide had been removed from the test vessel, it was removed from the fume hood, opened and the test specimens removed and weighed. A photograph was made of the test specimens and the control samples.

RESULTS:

Sample No. 1 - Plain alloy - reserved as control

| | | |
|---|---|--------------|
| <u>Sample No. 2</u> - Plain alloy - weight after exposure | - | 4.4220 grams |
| weight before exposure | - | 4.4210 grams |
| Increase in weight | - | 0.0010 grams |
| % Increase in weight | - | 0.0226 |

| | | |
|---|---|--------------|
| <u>Sample No. 3</u> - Plain alloy - weight after exposure | - | 4.3932 grams |
| weight before exposure | - | 4.3924 grams |
| Increase in weight | - | 0.0008 grams |
| % Increase in weight | - | 0.0182 |

Average % Increase in Weight - Plain Alloy - 0.0204

Sample No. 4 - Iridited alloy - reserved as control

| | | |
|--|---|--------------|
| <u>Sample No. 5</u> - Iridited alloy - weight after exposure | - | 4.3022 grams |
| weight before exposure | - | 4.3020 grams |
| Increase in weight | - | 0.0002 grams |
| % Increase in weight | - | 0.00465 |

| | | |
|--|---|--------------|
| <u>Sample No. 6</u> - Iridited alloy - weight after exposure | - | 4.4450 grams |
| weight before exposure | - | 4.4445 grams |
| Increase in weight | - | 0.0005 grams |
| % Increase in weight | - | 0.0112 |

Average % Increase in Weight - Iridited Alloy - 0.00793

RESULTS: (Continued)Residue From Bomb No. 1 Containing Plain Alloy

| | | |
|-------------------------------|---|---------------|
| Weight of beaker plus residue | - | 44.4291 grams |
| Weight of beaker (empty) | - | 43.7143 grams |
| Weight of residue | - | 0.7148 grams |

Used 300 ml. ethylene oxide S.G. 0.8711
 Weight of ethylene oxide - 261.33 grams

% Polymerization Residue - 0.273

Residue From Bomb No. 2 Containing Iridized Alloy

| | | |
|-------------------------------|---|---------------|
| Weight of beaker plus residue | - | 48.7579 grams |
| Weight of beaker (empty) | - | 48.4099 grams |
| Weight of residue | - | 0.3480 grams |

% Polymerization Residue - 0.133

Figure 4 shows the appearance of the specimens before and after exposure.

FURTHER WORK:

Since these tests were for only a week's duration, it might be advisable to repeat the tests and carry them on for a period of a month so that the Wyandotte system of rating could be applied. If this seems advisable, we should make certain that the samples used are much superior to the specimens used in this work.

REFERENCES:

1. Interoffice Notebook Pages 197866-197868 inclusive.
2. Discussions with following IQ-5 Personnel: J. Dodds, D. Raatz, J. M. Hamnell and J. Britten.
3. IDC from K. L. Kize (73-30) to I. Mayer (72-30) dated 2/17/56 on the subject "Corrosion Test of 61ST-4 and 61ST-6 Material in Ethylene Oxide Solution".
4. Ethylene Oxide Monofuel - A Summary dated 10 April 1953, Wyandotte Chemicals Corporation.
5. Liquid Monopropellant Investigation - 2, Detailed Report No. 7 dated 15 February 1954, Wyandotte Chemicals Corporation.
6. Liquid Monopropellant Investigation - 2, Detailed Report No. 8 dated 15 May 1954.
7. Work Folder 581/763.

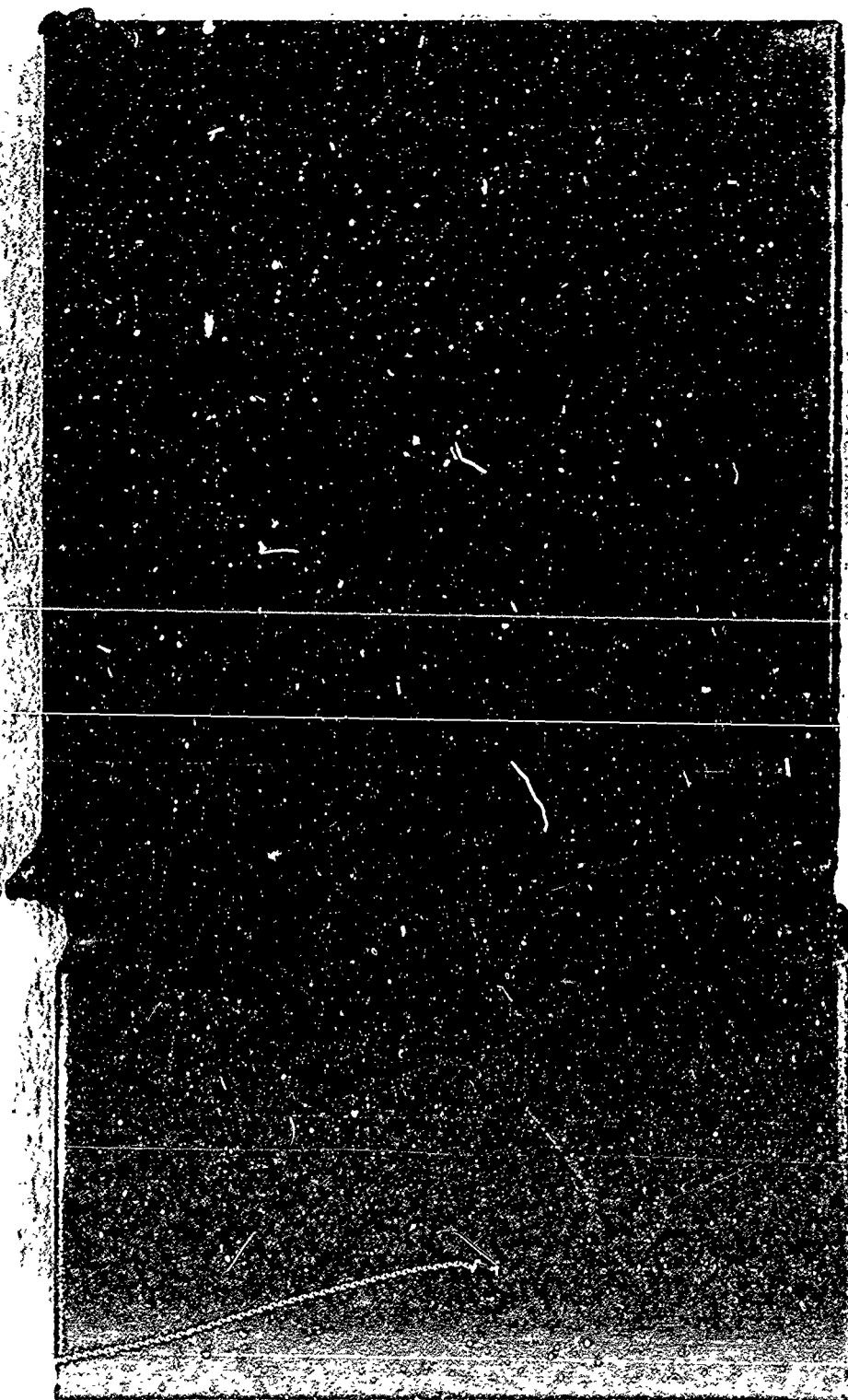
REFERENCES: (Continued)

8. MSD Report No. 3066 entitled "Ethylene Oxide Fuel, Diaphragm Material, Califilm, Unplasticized MEL-F, For Use In IQ-5".
9. MSD Report No. 3005 entitled "Ethylene Oxide Fuel, Plasticized MEL-F (LP-20)".

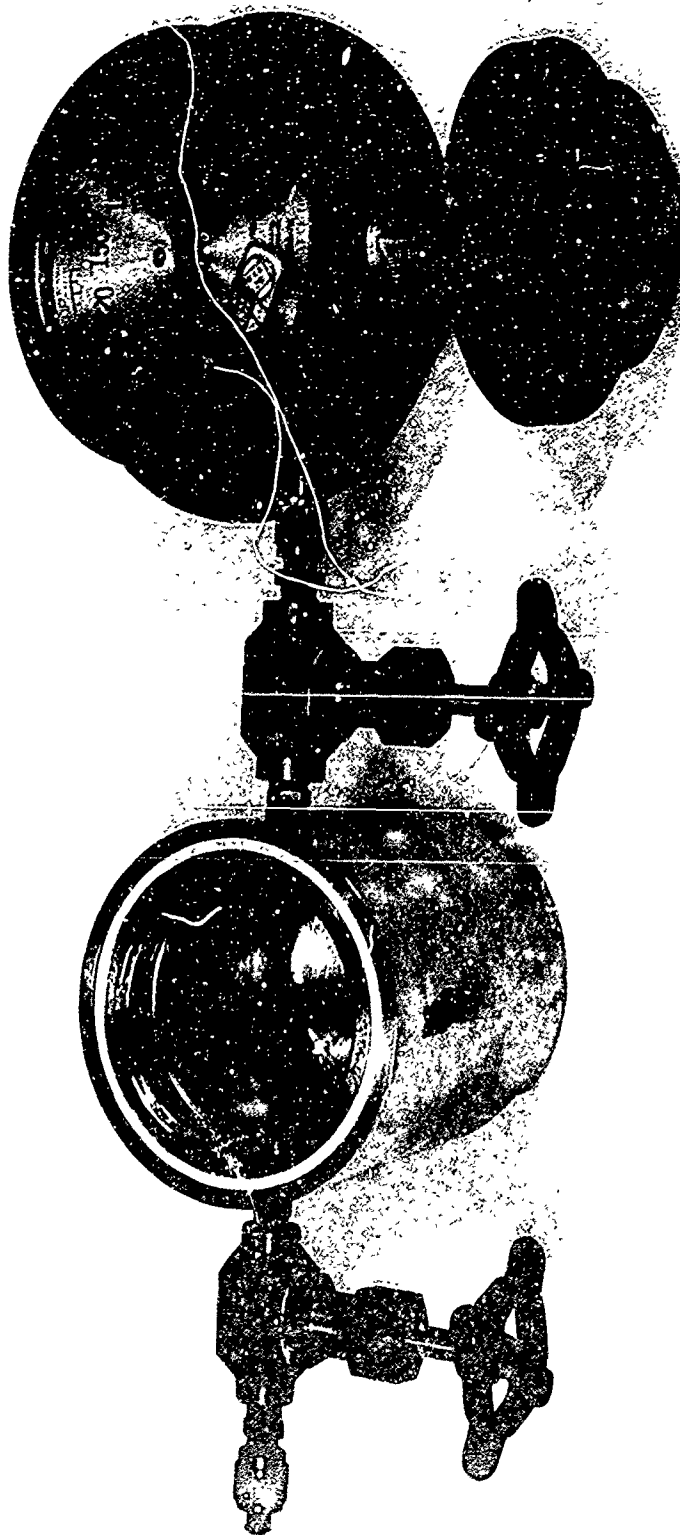
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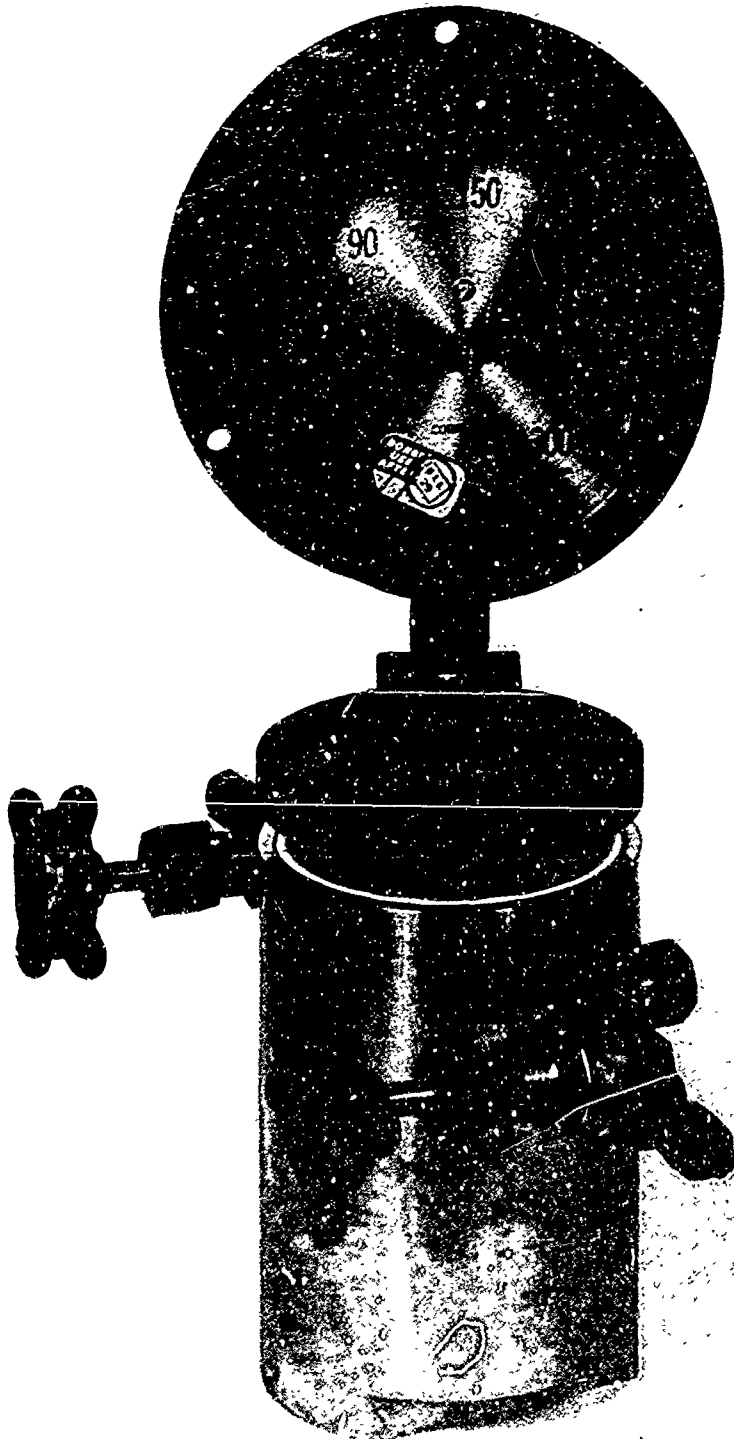
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ENLARGED VIEW OF REVERSE SIDE OF WELDED 61ST
SPECIMEN SUBMITTED FOR TESTS



DISASSEMBLED PARTS OF THE ALL-STAINLESS STEEL
PRESSURE TEST BOMB USED FOR HOLDING THE ETHYLENE OXIDE
TEFLON O-RING GASKET CAN BE NOTED



ASSEMBLED TEST BOMB EMPLOYED FOR HOLDING
THE ETHYLENE OXIDE DURING THE TESTS

Note the retaining nut holding the aluminum rupture disc
designed to blow out in case of excessive pressures.



FIGURE 4