INVESTIGATION OF THE EFFECTS OF LIQUID OXYGEN ON CHEMICALLY TREATED SURFACES OF ALUMINUM TUBING

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PHASE REPORT

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Liquid oxygen
Aluminum tubing
Chemically treated surfaces
Infrared Spectrophotometry
Atomic Absorption Spectroscopy
Electron Microscopy
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PHASE REPORT

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INTRODUCTION

Aluminum transfer tubing used in the oxygen breathing system aboard aircraft has been free of chemical coatings as specified in reference (a). Recently an aircraft manufacturer has used chemically treated sections of aluminum tubing in the liquid oxygen system aboard the F-14. Since specific information was not available on the possible effects which liquid oxygen may have on chemically treated aluminum surfaces and the resulting quality of the pilots' breathing oxygen, it was decided to determine if residual deposits, gaseous contaminants and adverse surface structural changes could be anticipated. Atomic absorption, infrared analysis and electron microscopy were used to study the effects of pressurized liquid oxygen flowing through untreated, chromic acid anodized, and chromate conversion coated aluminum tubing.

MATERIALS AND TEST PROCEDURES

Three 1-foot lengths of aluminum tubing, 6061-T6 (MIL-T-7081) HYD ½ in. I.D. by 1/16 in. thickness with connector fittings, were treated in accordance with the requirements of MIL-C-5541B (reference (b)) so that a chromate conversion coating was obtained on the interior and exterior surfaces of the tubing. Three additional lengths of tubing from the same source as the above stock were treated in accordance with MIL-A-86244C, Type I, (reference (c)) to obtain a chromic acid anodized coating on the interior and exterior surfaces of the tubing. A length of "untreated" aluminum tubing from the same base stock was used as a control.

The sections of chromate conversion coated tubing were then assembled and connected to the outlet valve of a 500 gallon liquid oxygen (LOX) storage tank. The tank was pressurized to approximately 10 lbs. to produce a continuous flow of LOX through the transfer tubing for 10 minutes. The LOX was collected in a 4000 ml. glass beaker.

Upon filling, the beaker was removed and replaced by a Navy G-276 LOX Sampler Cylinder. A LOX sample was collected for analysis of contaminants in the gaseous state by infrared spectrophotometry.

The sections of transfer tubing were then disconnected and immersed in the beaker of collected LOX for one hour, until evaporation was complete. The residue was retained for analysis by atomic absorption spectroscopy.

The same procedures were repeated with the chromic acid anodized tubing and the untreated (control) tubing.
ANALYSIS AND RESULTS

1. Chromium by Atomic Absorption Spectroscopy

A measure of the chemical inertness of the chromate conversion and chromic acid anodize coatings would be indicated by resistance of these films to the severe solvent and abrasive action of liquid oxygen under the test conditions. If leaching occurs the presence of chromium in the LOX residues would be expected. The residues, therefore, were transferred from the collection beakers by washings with demineralized water and dilute hydrochloric acid into volumetric flasks for analysis by atomic absorption spectroscopy. Standard solutions of chromium were prepared for calibration. The analytical conditions were as follows:

**INSTRUMENT:** Perkin-Elmer Model 303  
**HOLLOW CATHODE LAMP:** Chromium  
**WAVELENGTH:** 3579A  
**FLAME:** Air-Acetylene  
**SENSITIVITY:** 0.1 PPM Cr for 1% Absorption

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>% CHROMIUM FOUND</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromate Conversion Coating</td>
<td>None detected</td>
</tr>
<tr>
<td>Chromic Acid Anodize Coating</td>
<td>None detected</td>
</tr>
<tr>
<td>Untreated</td>
<td>None detected</td>
</tr>
</tbody>
</table>

2. Gaseous Contaminants by Infrared Spectrophotometry

The samples of LOX collected in Navy G-276 Sampler Cylinders under the conditions of test were analyzed by infrared spectrophotometry for the presence of gaseous contaminants as specified in specification MIL-0-27210C (reference (d)). The presence of unusual infrared absorption bands not normally encountered in specification grade LOX would be indicative of contributions by the chemically treated transfer tubing.

**RESULTS OF ANALYSIS:** No unusual contaminants were detected.

3. Surface Examination by Electron Microscopy

The following conditions were represented by samples of tubes:

a. Untreated; unexposed  
b. Untreated; exposed to LOX  
c. Chromic acid anodized; unexposed  
d. Chromic acid anodized; exposed to LOX  
e. Chromate conversion coating; unexposed  
f. Chromate conversion coating; exposed to LOX
These were cut lengthwise and the interior surfaces replicated with two percent solution of collodion. These replicas were then shadowed with chromium followed by vapor deposited carbon. The collodion first replicas were dissolved with acetone and the remaining chromium-carbon replicas were examined in the electron microscope at magnification up to 36,000X, and then photographed at 12,000 magnification. Figure 1 shows electron micrographs of the interior surfaces of the tubings. Photos A and B show the interior surface of the untreated aluminum tubing before and after subjecting to LOX, respectively. No structural differences were observed between the two specimens. Photos C and D show the interior surfaces of the chromic acid anodized tubing before and after subjecting it to LOX. Examination of many areas, even up to 36,000X, gave only minor evidence of a smoothing of some of the asperities on the surface of the tubing subjected to the LOX. These differences are not apparent in photos C and D. Photos E and F show the interior surfaces of the chromic acid conversion coated tubing before and after subjecting it to LOX. A visible difference was observed in the smoothing effect of oxygen on the roughness of the chromate conversion coating. The change in surface smoothness, however, did not adversely affect the quality of LOX being transferred.

CONCLUSIONS

From the foregoing studies it is concluded that:

1. Liquid oxygen had no detrimental effects on the chemically treated aluminum transfer tubing.

2. The chemically treated tubing did not adversely affect the quality of the breathing oxygen.

3. No advantages in using chemically treated transfer tubing were apparent.

RECOMMENDATIONS

It is recommended that there be no mandatory requirement for use of chemically treated aluminum transfer tubing in the liquid oxygen systems of naval aircraft. Existing systems which use chemically treated tubing, however, may continue to be used effectively.
REFERENCES

(a) Military Specification MIL-D-19326, Installation and Test of Liquid Oxygen Systems in Aircraft

(b) Military Specification MIL-C-5541B, Chemical Conversion Coatings on Aluminum and Aluminum Alloys

(c) Military Specification MIL-A-8625C, Anodic Coatings for Aluminum and Aluminum Alloys

(d) Military Specification MIL-0-27210C(ASG), Oxygen, Aviator's Breathing, Liquid and Gas
FIGURE 1  ELECTRON MICROGRAPHS OF THE INTERIOR SURFACES