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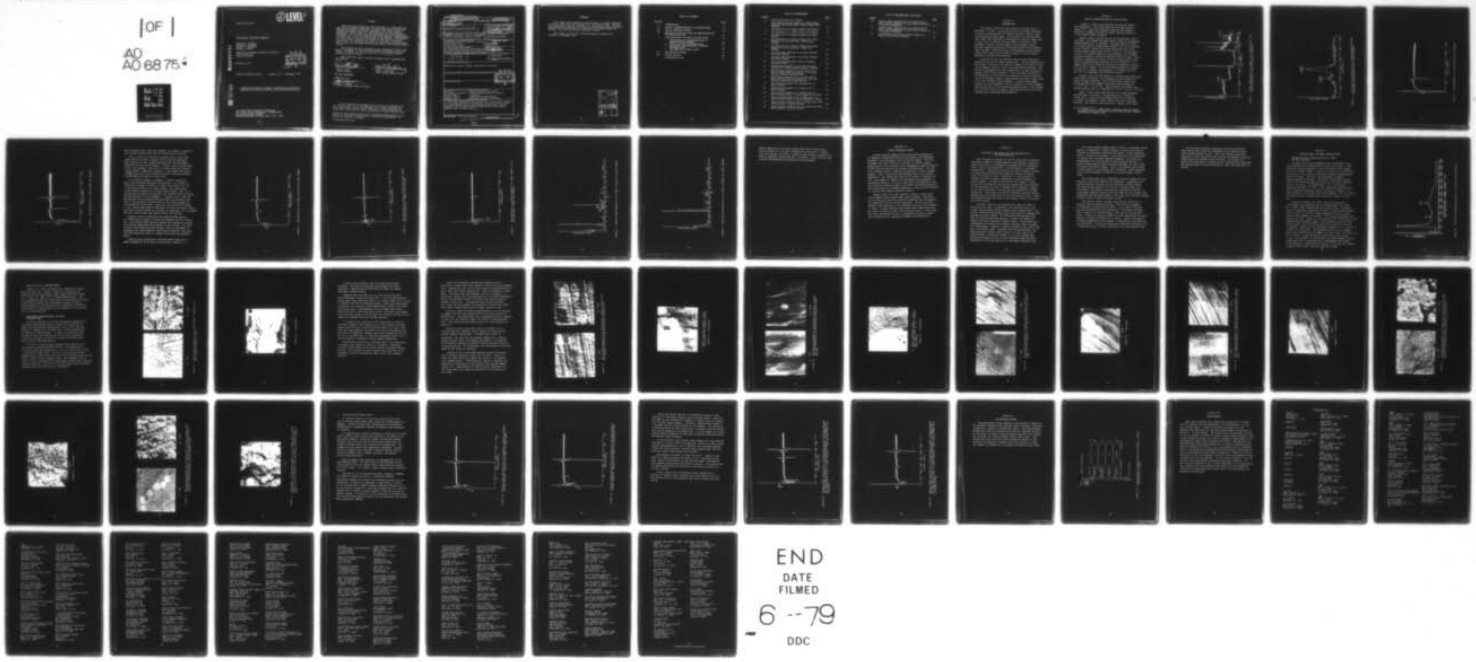

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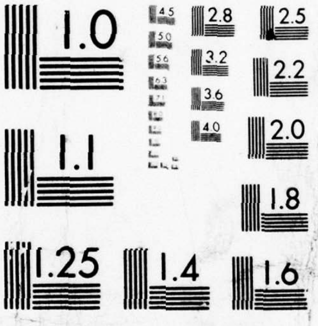

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NON-AQUEOUS ELECTRODE RESEARCH

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February 1979

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Passive films	

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes work carried out in the first 12 months of a 33 month contract to characterize the passive films formed on anodes commonly used in non-aqueous battery cells, and to identify cell reaction products formed at the carbon cathode commonly used in these cells.

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FOREWORD

This report was prepared by the University of Dayton Research Institute under Air Force Contract No. F33615-77-C-3156. The work was adminstered under the direction of the Aerospace Power Division, Energy Conversion Branch, Air Force Aero Propulsion Laboratory, with Mr. D.H. Fritts acting as contract monitor.

This report covers work conducted from 25 October 1977 through 31 September 1978.

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SECTION I

INTRODUCTION

This report covers the period from 25 October 1977 to September 31, 1978. During this period, the three techniques of Electron Spectroscopy for Chemical Analysis (ESCA), Auger Electron Spectroscopy (AES), and Secondary Ion Mass Spectroscopy (SIMS) were employed to characterize lithium anodes prior to their use in thionyl chloride ($SOCl_2$) batteries. Subsequently, several lithium samples were exposed to $SOCl_2$ and then examined using both the Scanning Electron Microscope (SEM) and AES. Additional work was performed to help characterize black spots which formed on several lithium samples exposed to nitrogen environments.

A helium dry box was obtained from the Aero Propulsion Laboratory for use in sample preparation. Several modifications were made to the box in an effort to improve its operation and insure sample integrity during long term lithium storage. Two pieces of equipment were also constructed for use during later phases of the current program. A four cylinder gas manifold was designed to allow selective exposure of clean lithium surfaces to nitrogen, oxygen, carbon dioxide and water vapor while in the AES and ESCA vacuum chambers. A high vacuum sample chamber was also designed to allow transfer of lithium samples from the dry box to the various analysis systems. This chamber will later be used to transfer anode samples from GTE Sylvania laboratories in Waltham, Massachusetts.

SECTION II

INITIAL CHARACTERIZATION OF LITHIUM METAL

A supply of lithium was obtained from the Aero Propulsion Laboratory. The lithium metal was received stored in mineral oil. Since the techniques used are very surface-sensitive, attempts were made to remove residual mineral oil on the samples before analyzing.

Figure 1 shows a survey scan of the lithium metal taken with ESCA. This sample was wiped several times with a methanol soaked Kimwipe prior to analysis. The high levels of sulfur and carbon indicate an appreciable amount of oil still remained on the surface. Figure 2 is a high resolution scan of the kinetic energy region from 1144 eV to 1169 eV. The photopeak at 1151.7 eV kinetic energy can be attributed to Li 1s photoelectrons excited by the Mg Kα radiation from the anode. Unfortunately, a photoline at 1155.9 eV can be noted, which is due carbon 1s photoelectron excitation by Al Kα radiation. It is thought that the Al radiation noted was attributable to excitation in the Al window. In all future ESCA studies Be windows will be used. However, the measured Li 1s binding energy of 55.9 eV is in agreement with literature values.[1]

Figures 3 and 4 are AES spectra of lithiuim metal before and after argon ion-etching. This specimen was also cleaned with methanol before analyzing. The ion-etching was carried out within the AES vacuum chamber which had a base pressure of 5×10^{-10} Torr (7×10^{-8} Pa). While etching, the chamber was backfilled with argon to a pressure of 5×10^{-5} Torr (7×10^{-3} Pa).

Figure 3 shows that carbon was the only element detected with AES prior to etching. The spectrum given in Figure 4 (taken after 2 hours of ion-etching) shows that in addition to carbon, the elements lithium, oxygen, and sulfur were also detected. The level of carbon was still, however, quite high.

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[1] K. Siegbahn et. al., ESCA Atomic, Molecular and Solid State Structure Studies by Means of Electron Spectroscopy, Almquist and Wiksells, Uppsala (1967).

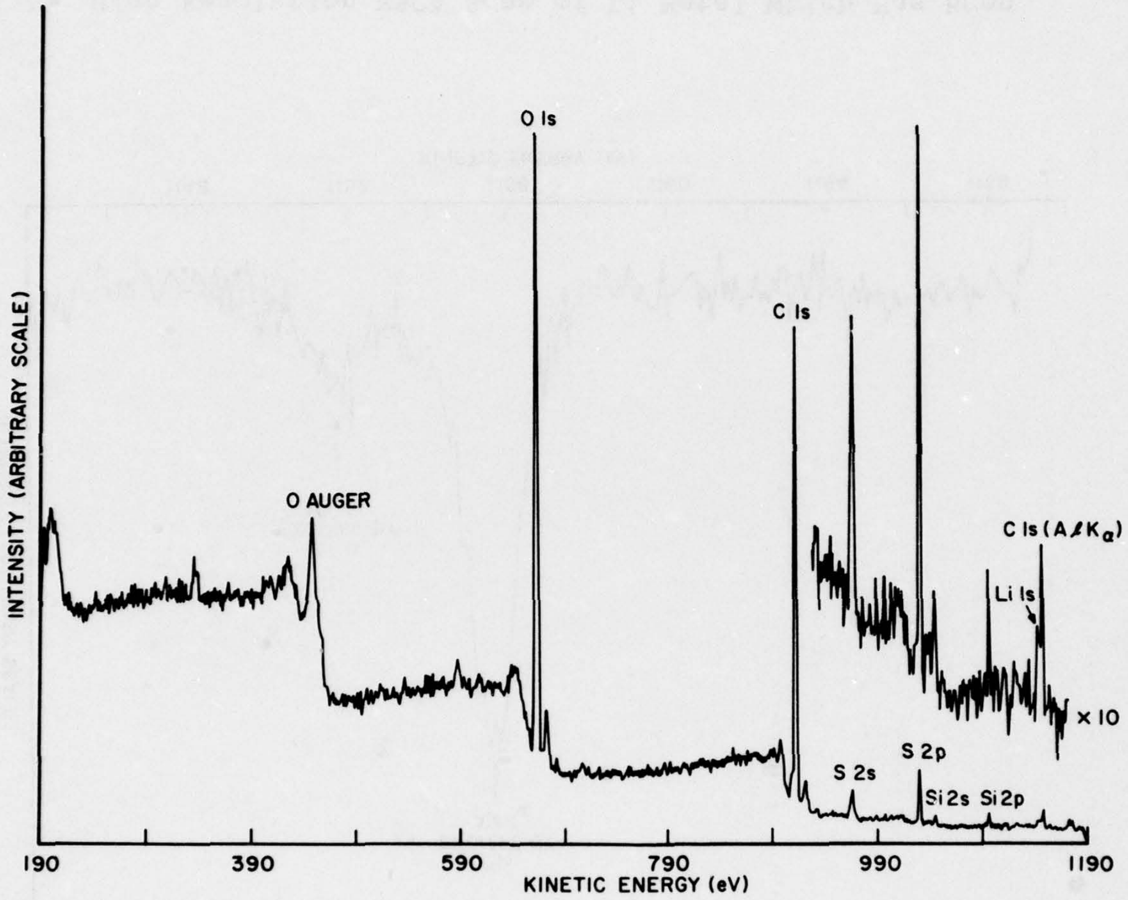


Figure 1. Survey ESCA Scan of Li Metal. All lines result from Mg Kα excitation unless otherwise indicated.

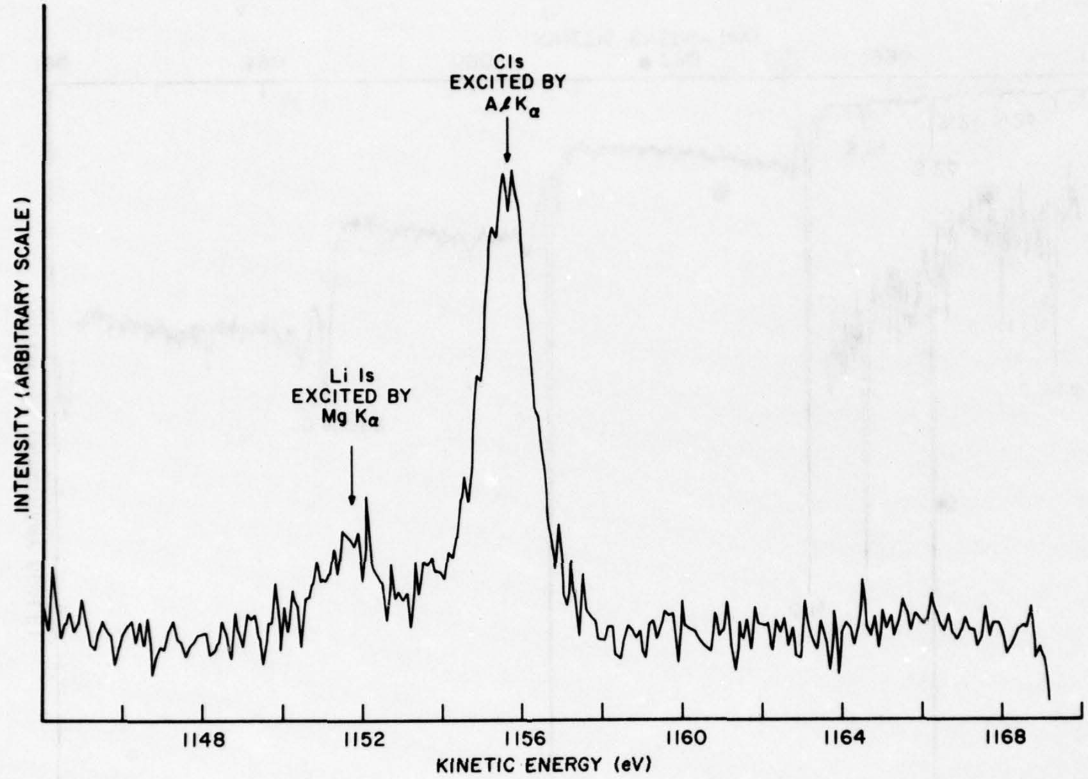


Figure 2. High Resolution ESCA Scan of Li Metal Which Has Been Previously Wiped Several Times with Methanol.

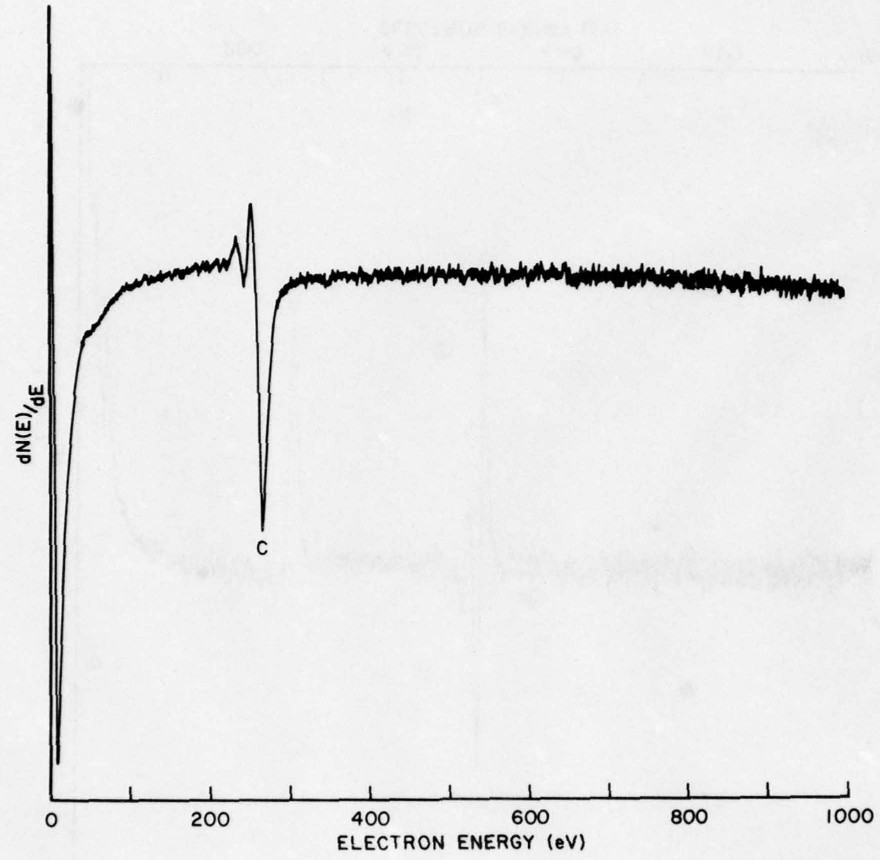


Figure 3. AES Spectrum of Li Metal Before Ion Etching.

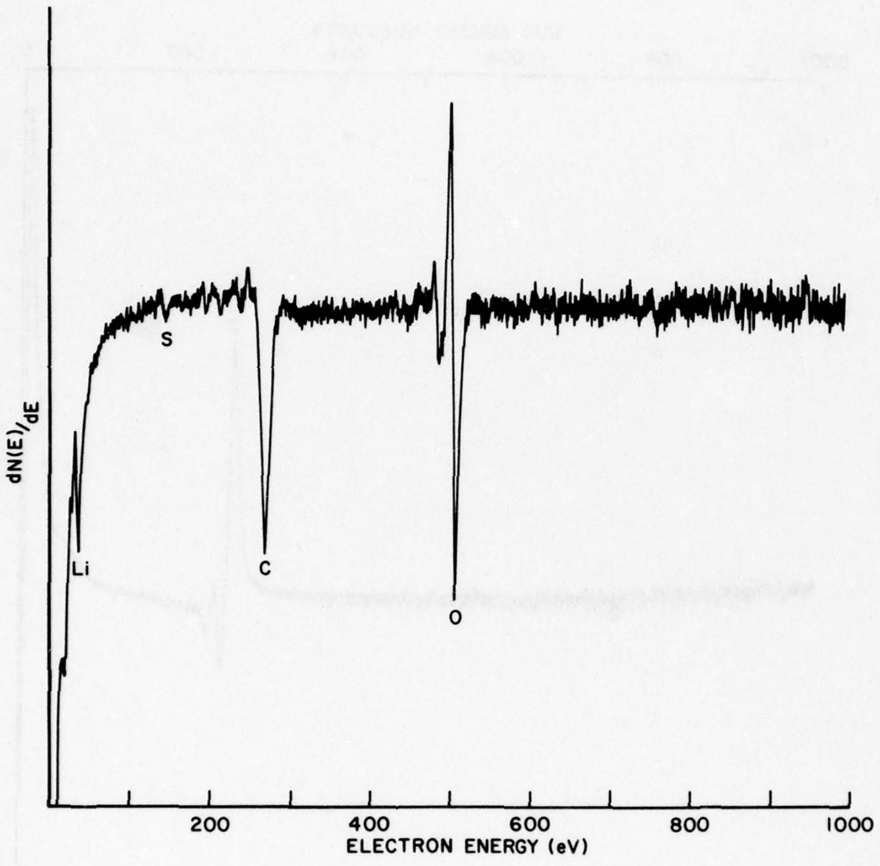


Figure 4. AES Spectrum of Li Metal After Ion Etching.

This indicates that there was probably oil present in pores in the lithium surface which was not removed by ion-etching.

Next, a piece of lithium was scraped with a razor blade under hexane in order to expose some of the bulk material. This surface was then cleaned with methanol and the sample inserted into the vacuum chamber. Figure 5 shows the AES spectrum of this sample. Note that the carbon level was substantially reduced compared to Figure 4. This sample did have a visible white film on the cleaned surface, which was probably lithium hydroxide and/or lithium oxide.

In a further attempt to produce a "clean" lithium surface, the following experiment was devised: (1) a piece of lithium was cleaned in hexane to remove the mineral oil, (2) it was placed, while still under hexane, into a glove bag filled with N_2 attached to the AES vacuum chamber, and (3) it was scraped to expose bulk material and inserted into the system without exposure to the atmosphere. Figures 6 and 7 show the AES spectra of this sample before and after ion-etching. Note that it was possible to remove most of the surface oxygen by ion-etching for 30 minutes. Also note that the kinetic energy of the lithium Auger peak is different in these two scans, indicating that lithium existed as an oxide (or hydroxide) on the surface and free lithium metal was exposed during ion-etching.

Figures 8 and 9 are positive and negative SIMS spectra of lithium metal cleaned in hexane and methanol prior to analysis. The positive SIMS spectrum showed that potassium, calcium, and aluminum impurities were present in the lithium. The level at which these impurities were present could be estimated if standards with known impurity levels were available. The negative SIMS spectrum (Figure 9) showed large oxide and hydroxyl peaks.

These initial experiments indicated some difficulty in sample preparation of an active lithium metal surface. It

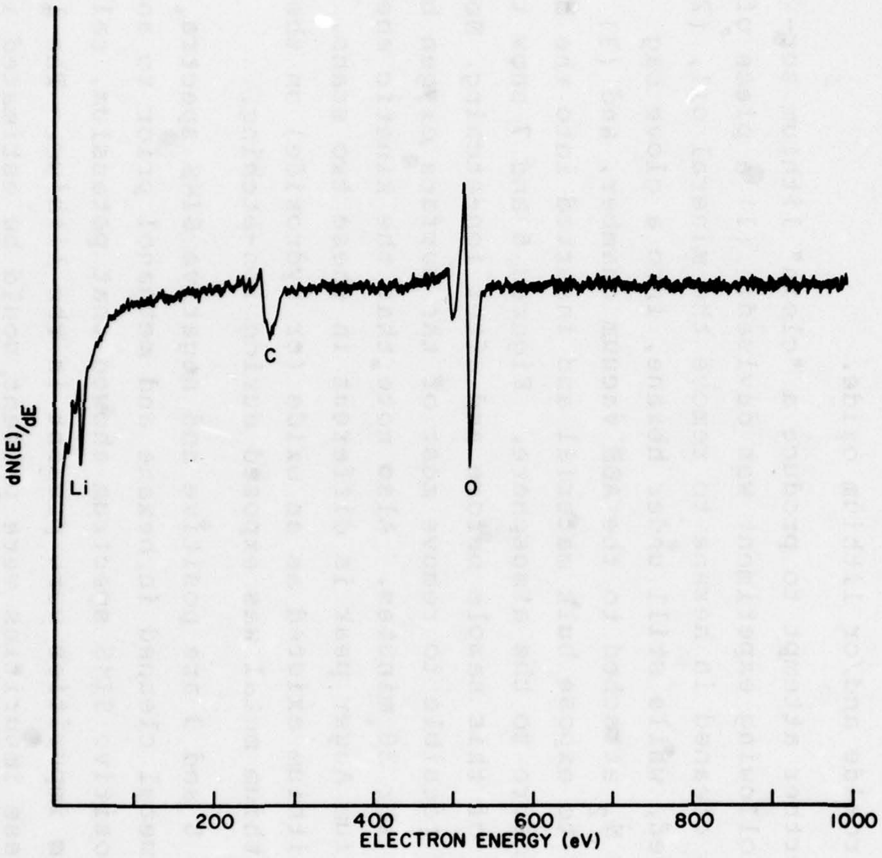


Figure 5. AES Spectrum of Li Metal Freshly Cut Under Hexane.

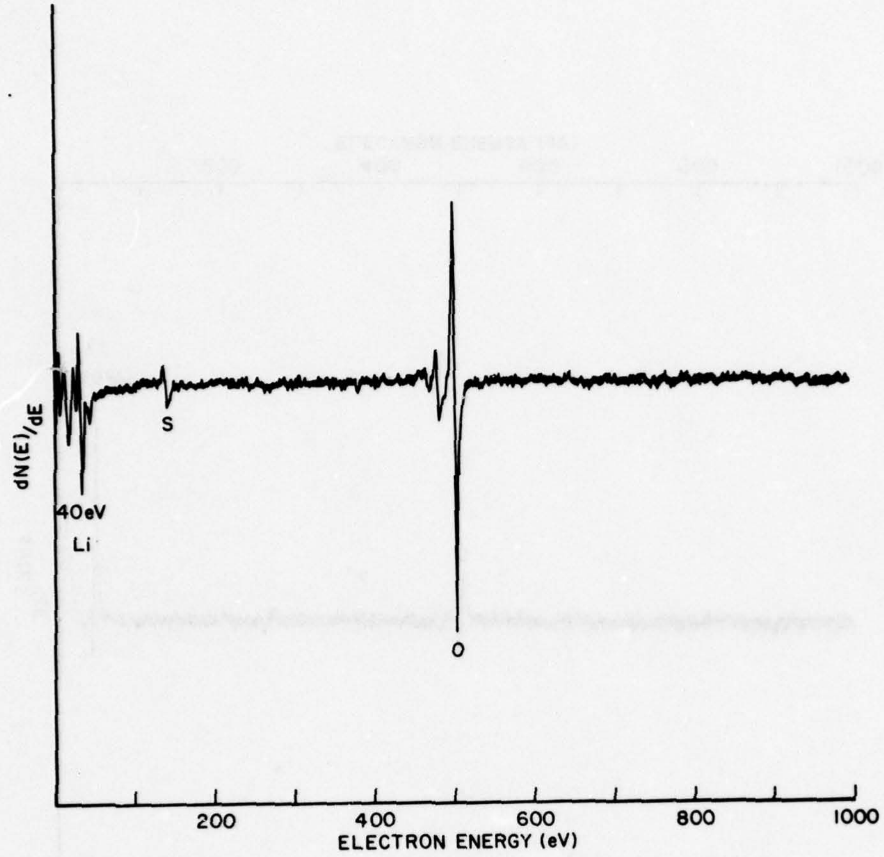


Figure 6. AES Spectrum of Li Metal Freshly Cut Under Hexane in an N_2 Glove Bag - Before Ion Etching.

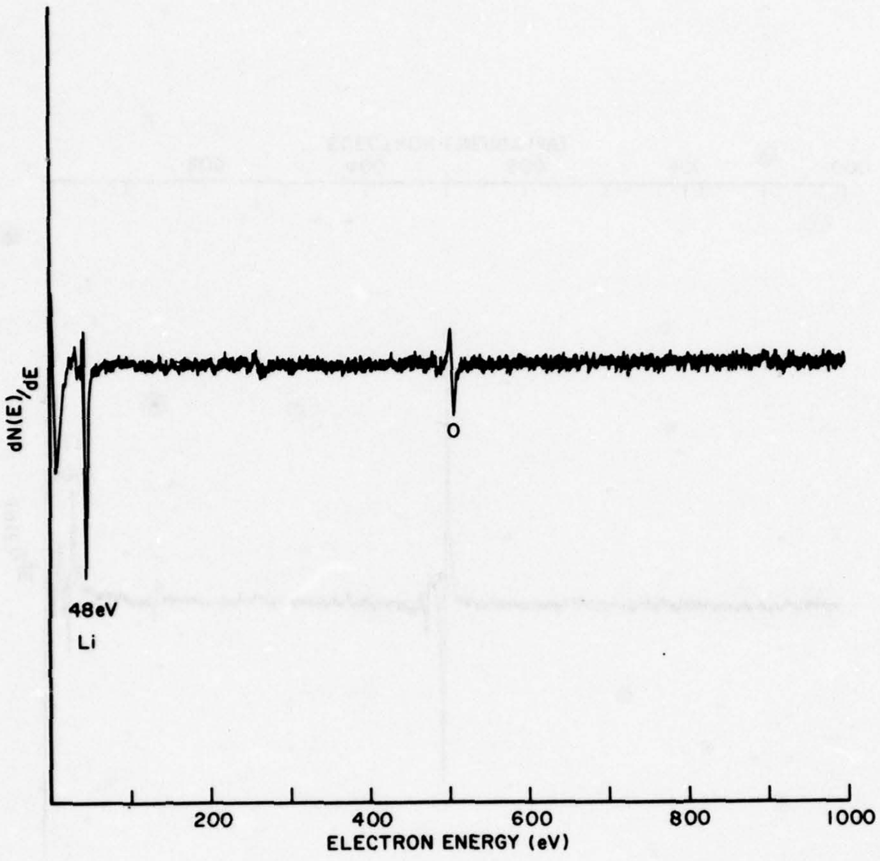


Figure 7. AES Spectrum of Li Metal Freshly Cut Under Hexane in an N_2 Glove Bag - After Ion Etching.

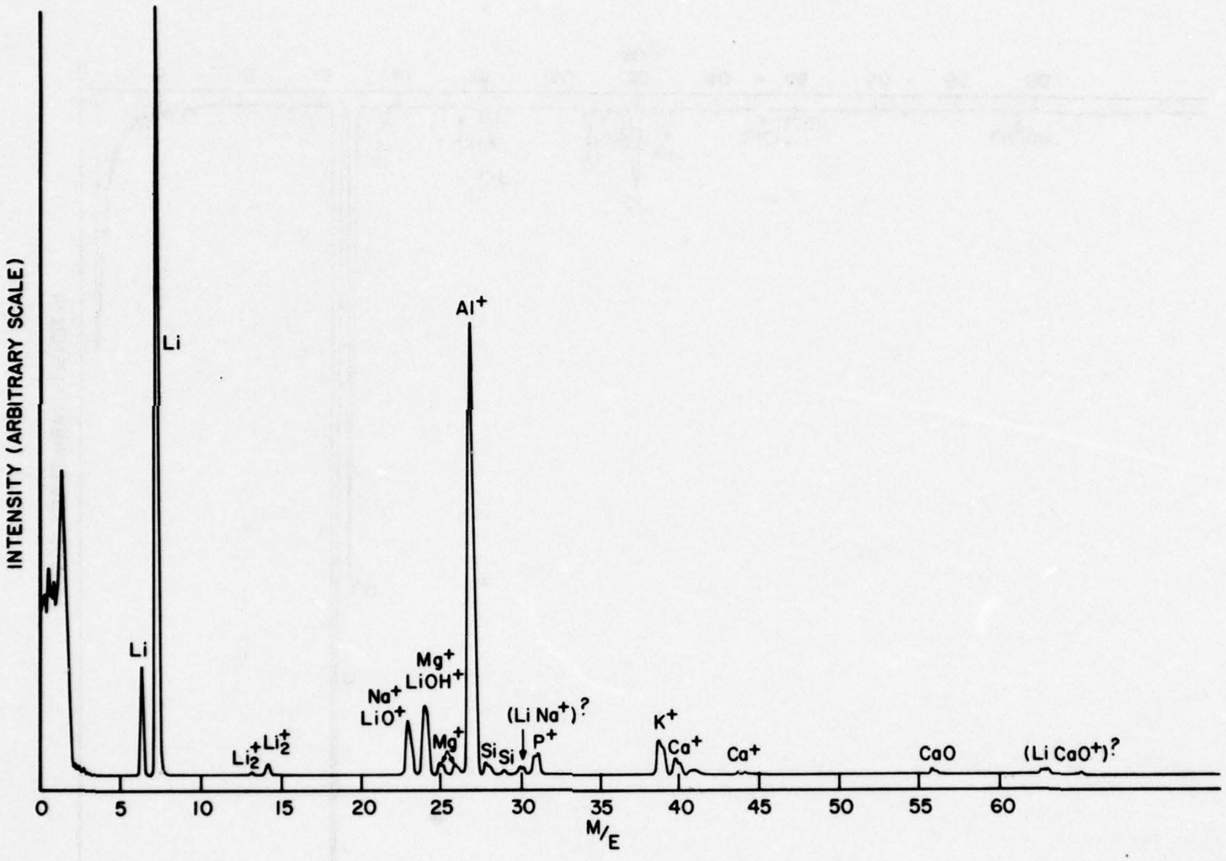


Figure 8. Positive SIMS Spectrum of Li Metal Freshly Cut Under Hexane.

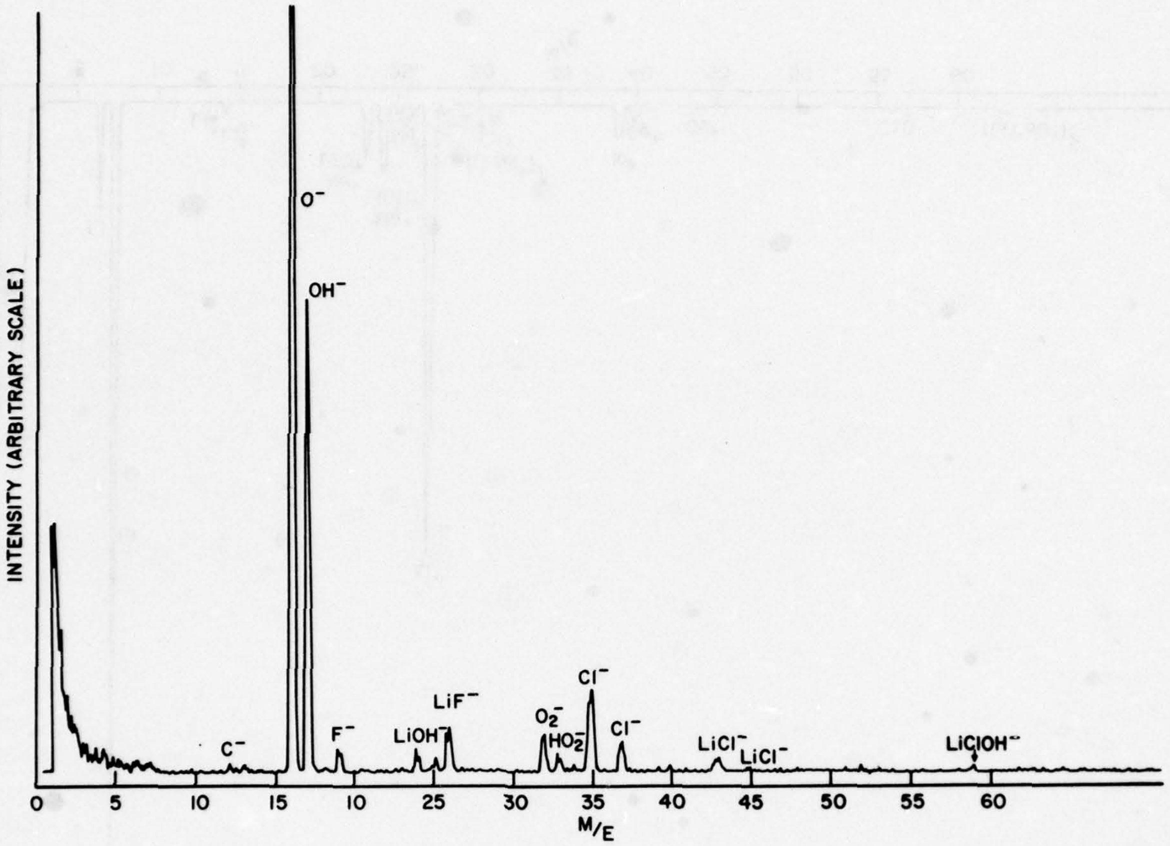


Figure 9. Negative SIMS Spectrum of Li Metal Freshly Cut Under Hexane.

seemed imperative from these studies that fresh lithium metal must be prepared in an inert atmosphere and mounted in a sample insertion vacuum interlock to insure minimum surface contamination. Such an insertion system does not, at present, exist on any of the surface instrumentation available at the University.

SECTION III

VACUUM INTERLOCK SYSTEM

Initial contract specifications called for preparation of samples at Wright-Patterson Air Force Base with subsequent analyses to be performed at the University campus. The work statement was later modified to provide for partial sample preparation at the University with supplemental samples supplied by GTE Sylvania Laboratories Waltham, Massachusetts. The contract changes further indicated the need for a vacuum transfer chamber. Such a system was thus designed and is now nearing completion. The system was sized to fit within a standard dry box. The chamber is capable of withstanding gas pressures >1 atmosphere; however, it is also leak tight and compatible with the ultra-high vacuum systems of the ESCA and AES spectrometers. This system will be extremely important in insuring the integrity of lithium anode samples exposed to $SOCl_2$.

Samples which have been immersed in $SOCl_2$ liquid would likely adsorb and/or absorb $SOCl_2$ gas molecules. The majority of these gas molecules would be liberated when the samples are submitted into the surface analyzers. One of the major residual gas components in the ESCA/AES stainless steel chamber is water. Thionyl chloride reacts with water vapor producing HCl which is known to react with stainless steel. A cold trap was thus constructed for use in condensing the $SOCl_2$ vapors, thus, protecting the expensive ESCA/AES instrumentation.

SECTION IV

REVISION OF THE TEST PLAN AND MODIFICATION OF THE HELIUM DRY BOX

Due to personnel changes at the Air Force Aero Propulsion Laboratory (AFAPL) it became apparent that adequate technical support would not be available for preparation of lithium samples. This was aggravated by a limited access to the AFAPL Laboratories. University employees and their students working at Wright-Patterson Air Force Base were limited to daytime hours, five days per week. Therefore, it was necessary to transfer routine Li sample preparation to the University of Dayton campus. In order to facilitate this change a Fisher Scientific Isolator/Lab Dry Box was transferred from the AFAPL to the University for use in exposure of Li samples to $SOCl_2$. At the time of transfer, the dry box was operating at minimal efficiency. There were also several inherent potential safety hazards due to the design of the gas purification train. Extensive modifications were made to the system in order to correct the problem areas.

Initially, the dry box consumed more than two tanks of helium gas per week. In addition, the molecular sieve (gas drying) recirculation system required daily regeneration. As a result, projected maintenance costs would have far exceeded the proposed two year budget. The following modifications were made to correct these deficiencies. First, all rubber gas supply lines were replaced by copper tubing. This along with replacement of a faulty gas pressure regulator made a significant improvement. Second, a crack was found in the case of the gas recirculation pump. Checking with the manufacturer, the pump contained no shaft seals making it a poor choice for a helium system. The helium leaked out via the cracked pump housing and quantities of air were ingested around the shaft. Replacement of the old pump with a new rubber diaphragm pump

The vacuum access chamber would not hold a reasonable vacuum making it impossible to insert samples to the dry box without introducing atmospheric moisture and oxygen. Three steps were taken to correct the problem. A new outer door was constructed to replace the old rubber gasketed assembly. An O-ring was incorporated into the new door to assure proper sealing. A second vacuum pump was also installed expressly for use in pumping down the access chamber. Third, the plumbing was altered to make pumping of the chamber more efficient. These modifications proved successful in making the access chamber vacuum tight.

The vacuum pump used to pump both the main chamber and the molecular sieves were found to be defective. Disassembly revealed extensive corrosion due to ingestion of $SOCl_2$ vapors. The problem was traced to the lack of a suitable $SOCl_2$ neutralization trap between the molecular sieves and the pump. A replacement pump was obtained from the AFML and the vacuum lines connecting the molecular sieves to the pumps were rerouted through existing traps of liquid nitrogen and CaO.

The CaO and liquid nitrogen traps proved ineffective in removing and/or neutralizing $SOCl_2$. The CaO was incapable of completely neutralizing the $SOCl_2$. As a result, chlorine gas was detected within the laboratory. The liquid nitrogen, while effective in removing $SOCl_2$, also had two serious drawbacks. First, the liquid nitrogen trap was constructed of glass making it fragile and difficult to clean. Second, the liquid nitrogen evaporated quickly making it impossible to maintain the trap overnight. A new trap system was constructed consisting of an improved liquid nitrogen trap and an activated charcoal bed.

The automatic pressure regulation system was improved. There was no easy means for disengaging the automatic system while working inside the box. Also, the pressure reduction required to allow easy entry into the box was awkward, requiring resetting of the pressure limit switches. An on/off and a manual override switch to reduce the box pressure were installed. The changes made the dry box efficient, safe, and convenient to operate.

SECTION V

LITHIUM/THIONYL CHLORIDE EXPOSURE TESTS

1. PREPARATION AND CHARACTERIZATION OF FRESH THIONYL CHLORIDE

An inert distillation apparatus was constructed of standard glassware and standard 24/40 ground glass joints. Each glass fitting was assembled to the main distillation apparatus with a teflon sleeve. A piece of freshly cut lithium metal was placed in the boiling flask which contained approximately 1000 ml of $SOCl_2$. The $SOCl_2$ was purchased as reagent grade from Baker Chemicals. Helium was continuously purged into the distillation apparatus prior to and during distillation of the $SOCl_2$. The temperature of the distillation apparatus was kept at 77.9°C. This temperature was monitored with a platinum resistance sensor and digital readout. The first fraction of the distillate was discarded. The next 200 ml was captured into a ground glass flask for storage in a dry box.

Aliquots of $SOCl_2$ were taken from the freshly distilled $SOCl_2$ and analyzed with gas chromatograph (GC) Model No. 5730A. Some characteristics of the GC were: Poropak Q column, a detector temperature of 200°C, injection port temperature of 100 C, an initial column temperature of 40 C, and a final column temperature of 140°C. The column programming rate used was 8 C/min. Figure 10 illustrates the GC trace taken of the freshly distilled $SOCl_2$ sample. The ordinate is given in the log of signal current and the abscissa in retention time (RT) in minutes. There are four principal regions (A, B, C, and D) of interest in the Figure. The depression noted at A is simply due to the injection of the sample into the column; the peak at B, RT = 5.3 min, is characteristic of pure $SOCl_2$; the region C, RT = 12.4 minutes, is the RT where HCl would appear; and the broad band at D is characteristic of decomposition of $SOCl_2$ in the detector. Measuring the area of C to B, one can roughly calculate that there is 4% HCl remaining in SOCl.

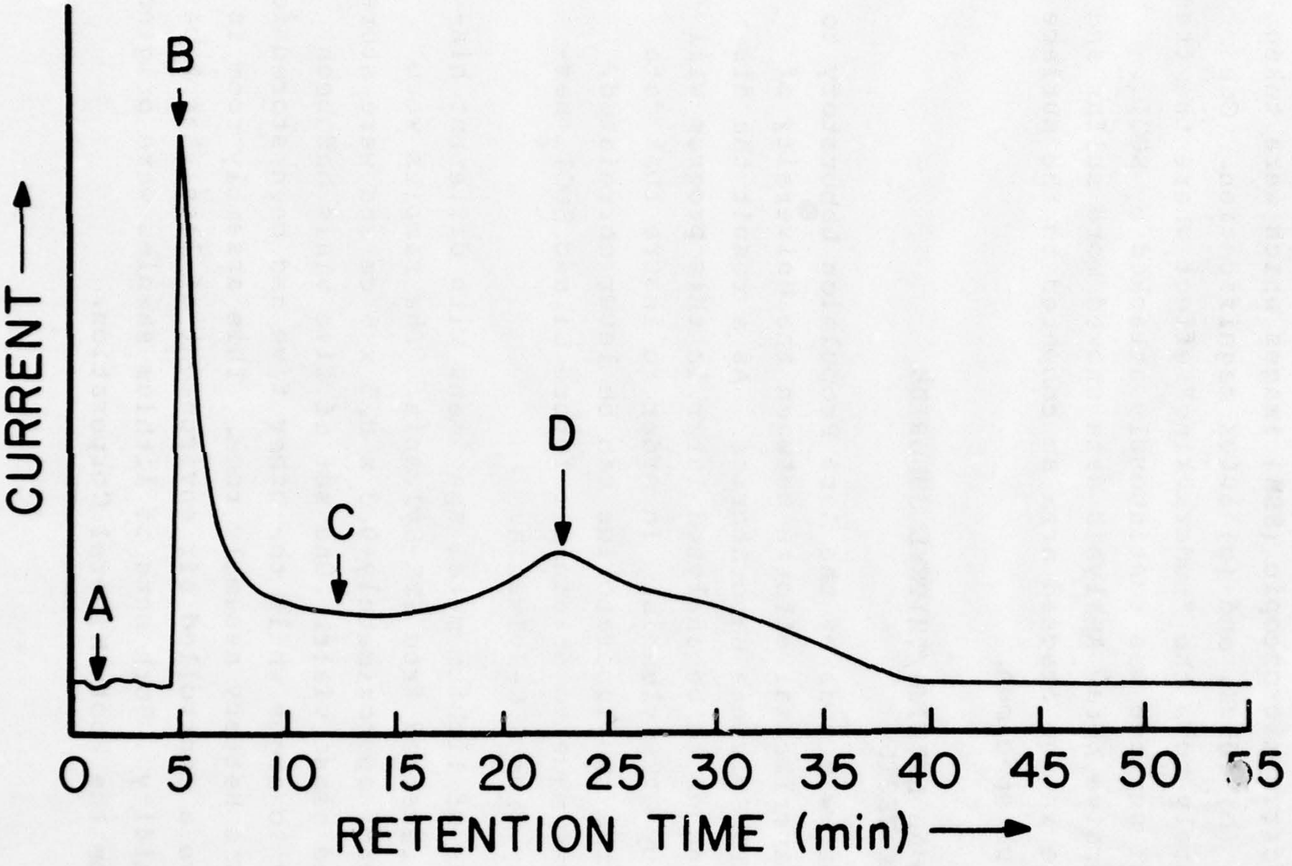


Figure 10. The Gas Chromatographic Trace of Freshly Distilled $SOCl_2$.

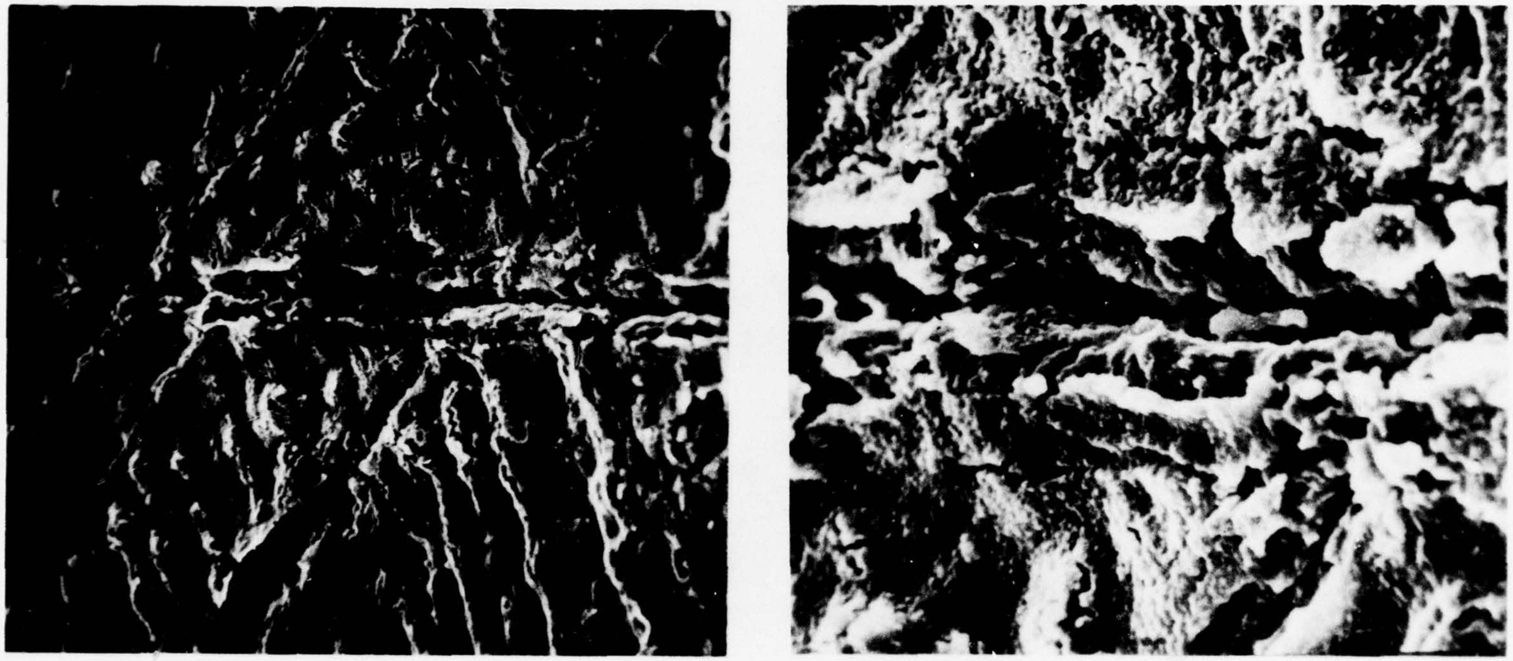
2. INITIAL Li/$SOCl_2$ EXPOSURE TESTS

Freshly cut lithium specimens were immersed in liquid $SOCl_2$ and left for 2 weeks. Figure 11 illustrates the scanning electron microscopic (SEM) images which were taken at (a) 300X, (b) 1000X, and (c) 3000X magnification. One will immediately note the "mudcracking" effect where the fresh lithium metal surface was continuously attacked by $SOCl_2$. Energy Dispersive X-ray Analysis data showed more sulfur and less chlorine in the cracked area as compared to the surface of the lithium specimen.

3. ADDITIONAL LITHIUM/THIONYL CHLORIDE EXPOSURE TESTS

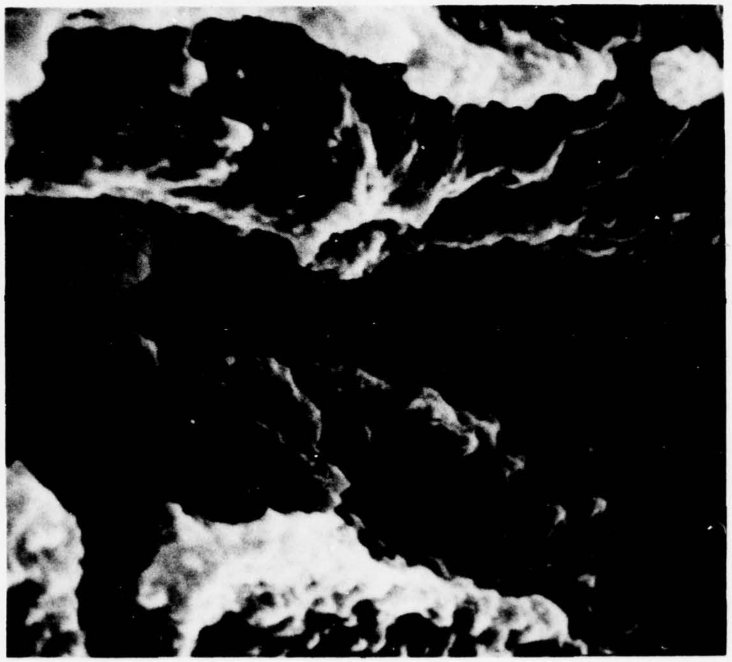
An effort was made by the Aero Propulsion Laboratory to coordinate experimental efforts between the University of Dayton and GTE-Sylvania Laboratories. As a result the discharged Li anodes to be analyzed later in this program will be supplied by GTE-Sylvania. In order to insure that data obtained by the two laboratories can be later correlated, a decision was made to obtain all future Li and $SOCl_2$ samples directly from GTE-Sylvania.

Two sets of lithium metal specimens with different histories were received from GTE-Sylvania. The samples were in ribbon form, approximately 0.3 x 0.5 x 6 cm and were stored in wax sealed glass vials. One set of five vials had been exposed only to argon while the other five had been stored for 88 hours in GTE's battery assembly room. This assembly room is known to have a controlled air environment of less than 2 percent relative humidity. Both sets of lithium samples were originally obtained from the Foote Mineral Corporation.



a) 300X b) 1000X

Figure 11. SEM Photomicrographs of the "Mud Cracked" Surface on Li Metal Sample Exposed to $SOCl_2$.



c) 3000X

Figure 11. (Concluded)

Freshly distilled $SOCl_2$ was also received from GTE-Sylvania. The $SOCl_2$ was originally purchased from Mobay Chemicals. Analysis by GTE had shown the $SOCl_2$ to contain less than 30 ppm of water.

Samples from both sets of Li as well as the GTE $SOCl_2$ were introduced to the helium dry box. A new, stainless steel, single edge razor blade was used to cut several small (0.5 x 1 cm) Li samples from the as-received ribbons. These were then placed three each, in a series of four Mobay glass, teflon lined screw cap septum vials. Approximately 20 ml of $SOCl_2$ were introduced into two of the vials and all four vials were then capped. The vials were stored in the dry box for 70 hours and then one Li sample was removed from each for SEM analysis.

Li samples were prepared for SEM examination as follows: Individual samples were mounted on one inch diameter aluminum sample mounts using a silver-containing paint as a glue. Samples were positioned so that both a cut and an as-received surface could be observed. Samples were then transferred from the dry box to the SEM room in zip-lock polyethylene bags so as to minimize ambient atmosphere induced sample degradation.

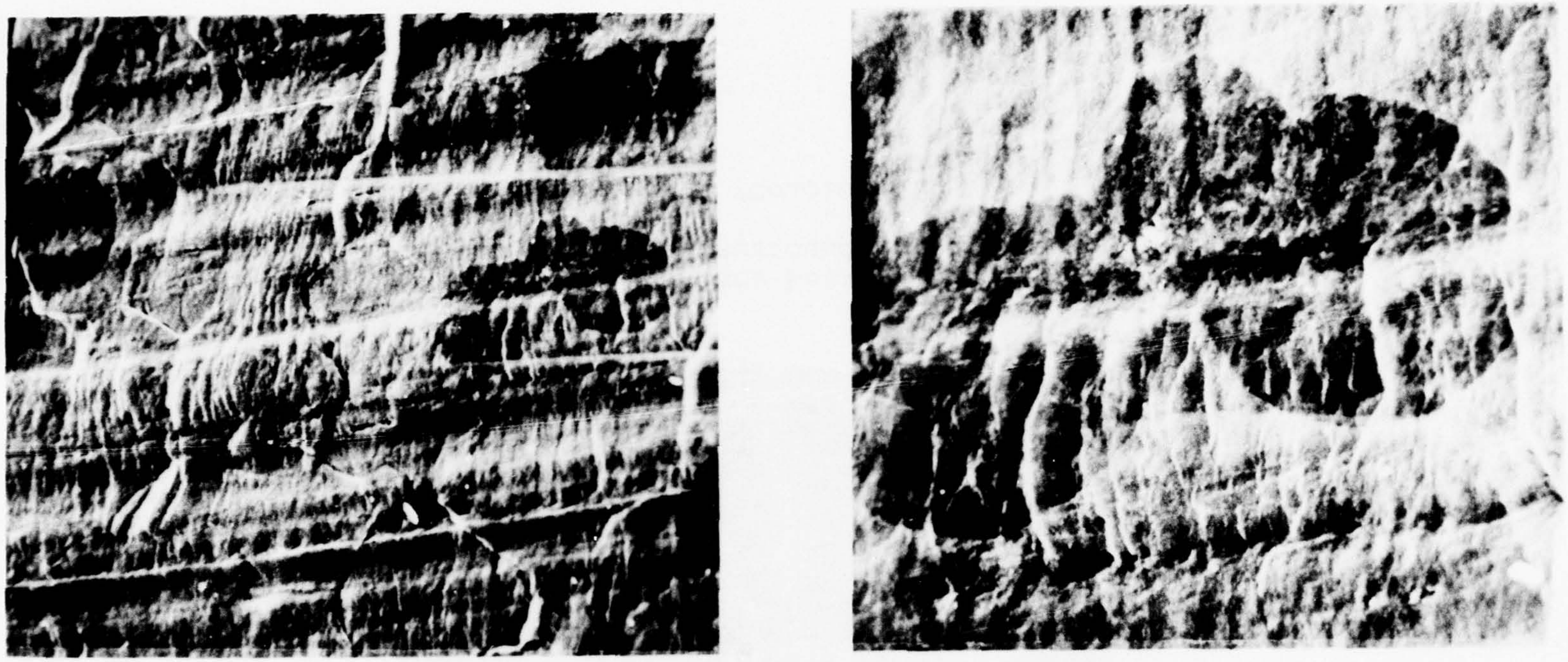
GTE-Sylvania Laboratories informed UDRI of the appearance of nodular black spots on certain Li surfaces. Subsequent analysis by GTE indicated the black nodules contained lithium nitride. The presence of black nodules was confirmed during subsequent analysis performed at UDRI on the $SOCl_2$ exposed samples.

Figure 12 illustrates the SEM photographs of an as-received Li surface which was exposed to the GTE battery assembly room. Figures 12a, 12b, and 12c were taken at magnifications of 100X, 300X, and 3,000X respectively. Note the presence of dark spots. The spots were approximately 200 μm in diameter. Of particular interest was the difference in the surface appearance across the interface between the black spot and surrounding surface (Figure 12c). The spotted area was much smoother. Also noted was the presence of striations (12a and 12b) which were likely formed during initial sample cutting at GTE.

Figure 13 shows the SEM photomicrographs on an as-received Li sample that had been exposed only to the argon dry box at GTE-Sylvania. Dark spots were still observable but were reduced in both size and apparent number by a factor of approximately four. The surface textures were similar to those observed in Figure 12.

Figures 14 and 15 show SEM scans of "freshly" cut Li surfaces from GTE "assembly room" exposed and argon dry box exposed samples respectively. Although very similar in appearance, the argon dry box exposed sample had a slightly rougher texture than the assembly room sample. As expected, both fresh-cut samples had smoother surface textures than as-received surfaces. Of significance was the absence of any dark spots on the fresh-cut surfaces.

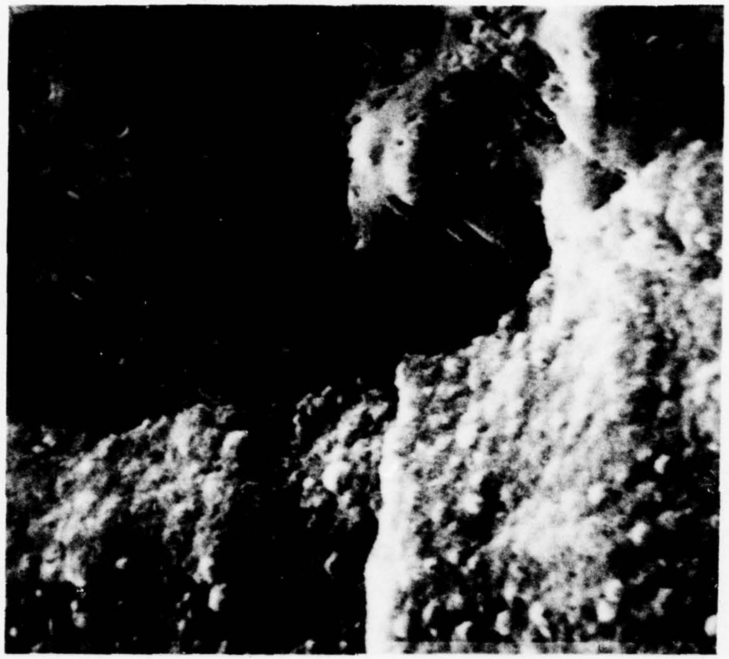
Figures 16 and 17 show SEM photomicrographs of freshly cut Li surfaces exposed to $SOCl_2$ for 70 hours. Figure 16 shows the argon glove box sample and Figure 17 the assembly room exposed sample. Differences are apparent including the presence of black spots on the assembly room exposed sample Figure 17c). Explanations of these figures, especially for the black spots on a fresh-cut surface are not possible at this time.



a) 100X

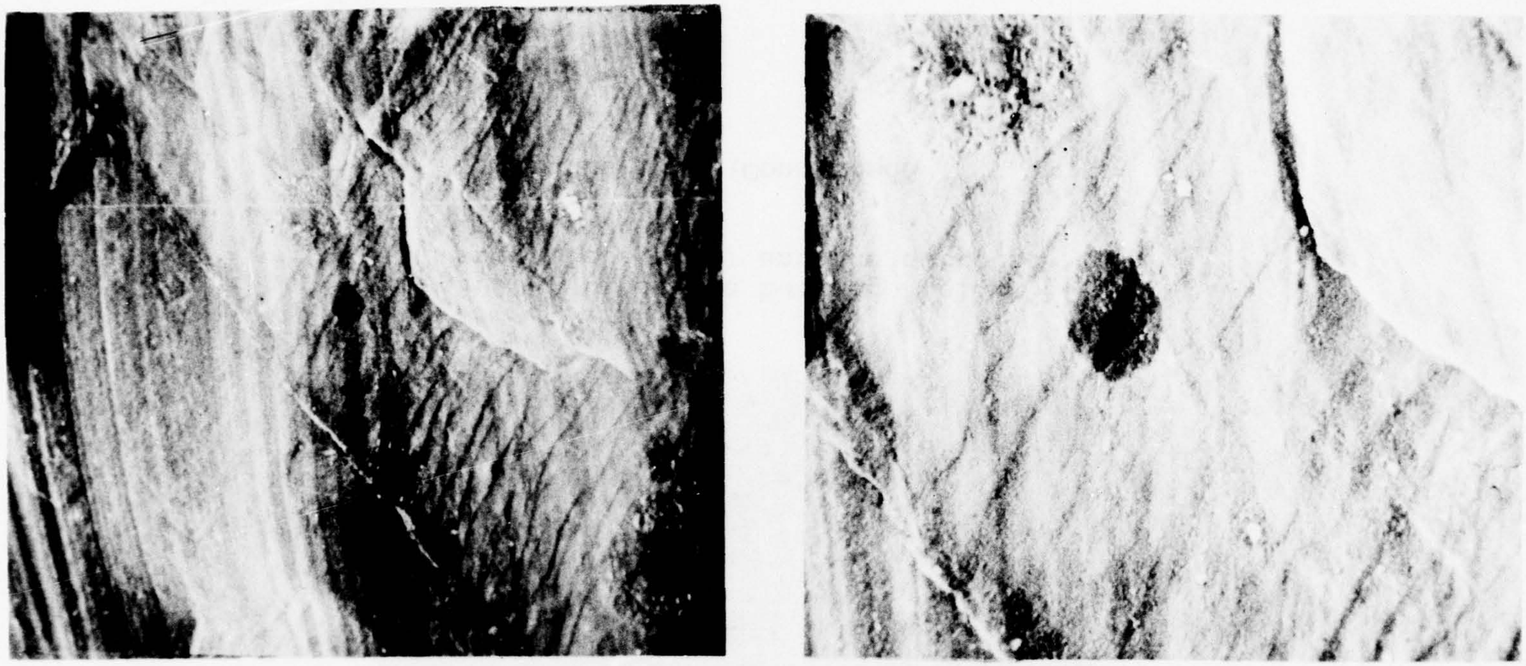
b) 300X

Figure 12. SEM Photomicrographs of an As-Received Li Metal Surface that was Exposed to the GTE-Sylvania Battery Assembly Room.



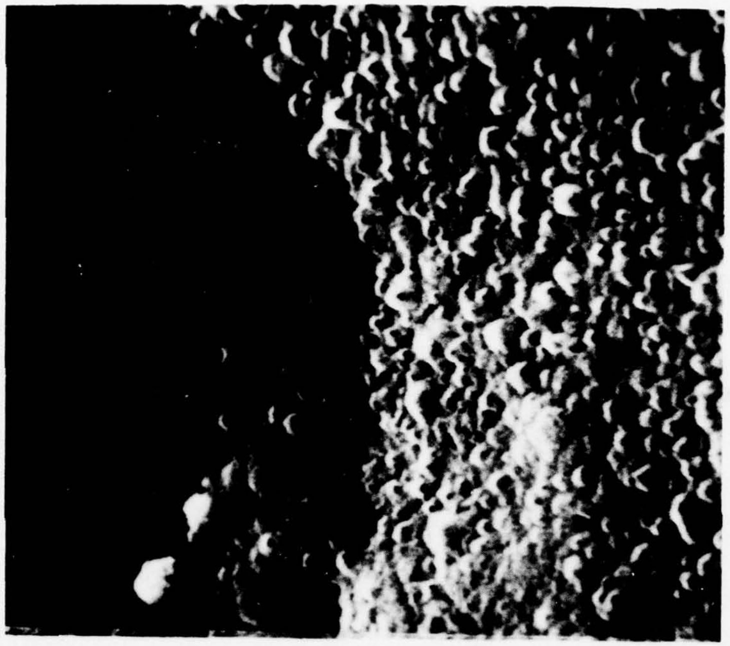
c) 3000X, interface between a black spot and the surrounding surface.

Figure 12. (Concluded)



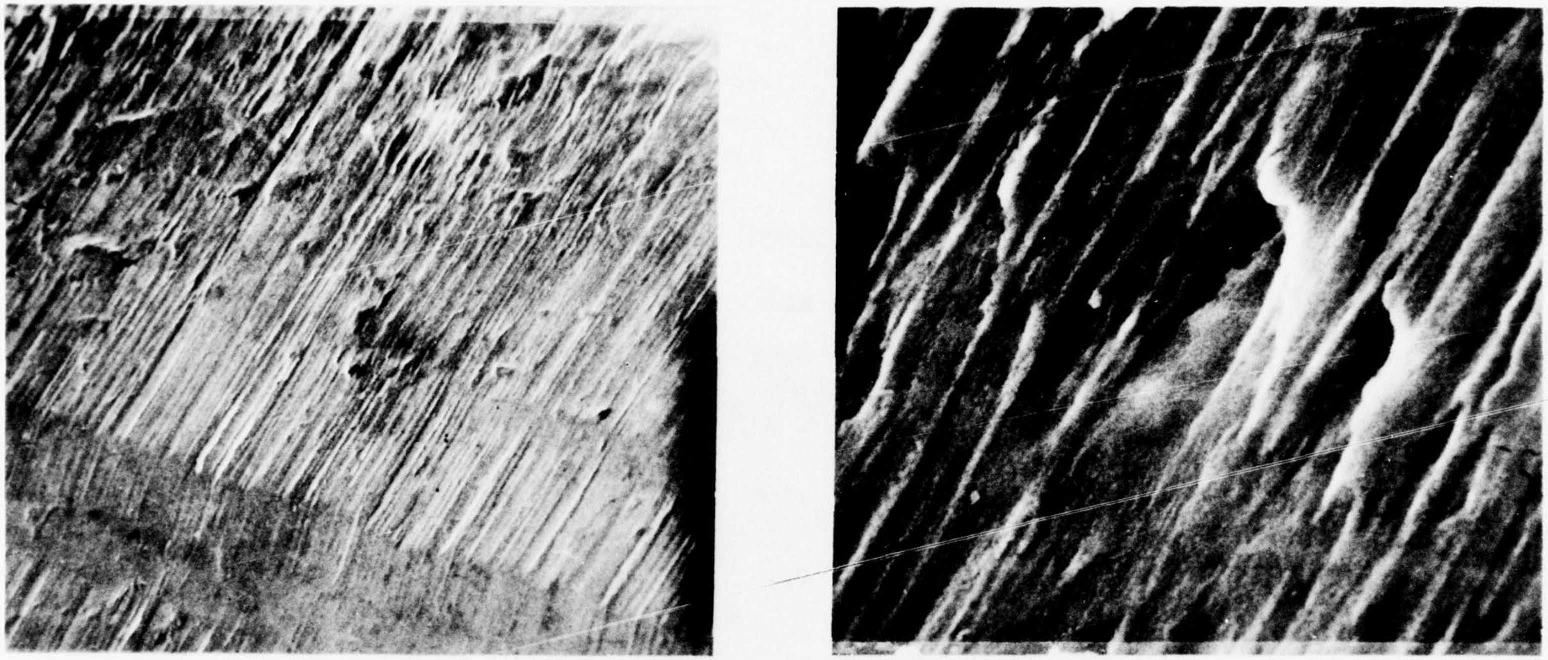
a) 100X b) 300X

Figure 13. SEM Photomicrographs of an As-Received Li Metal Surface that was Exposed to the GTE-Sylvania Laboratory Argon Dry Box.



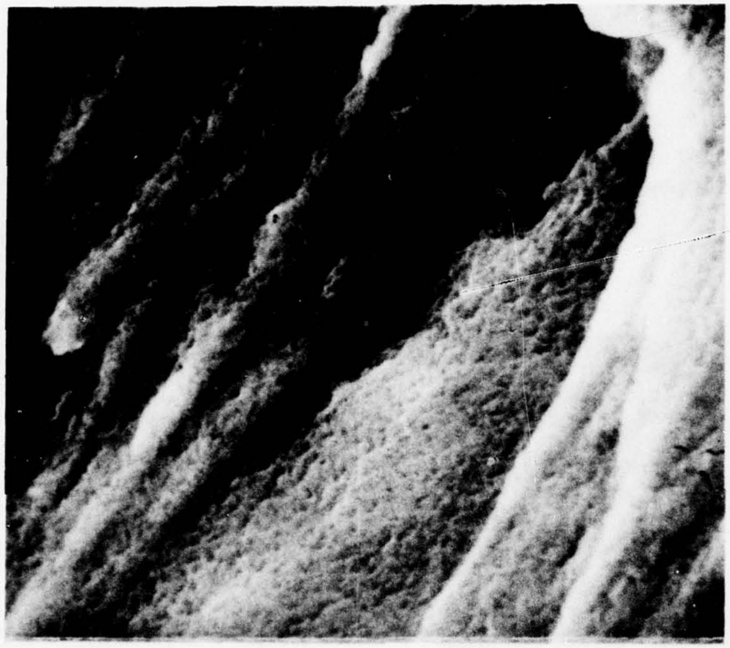
c) 3000X, interface between a black spot (left side) and the surrounding surface

Figure 13. (Concluded)



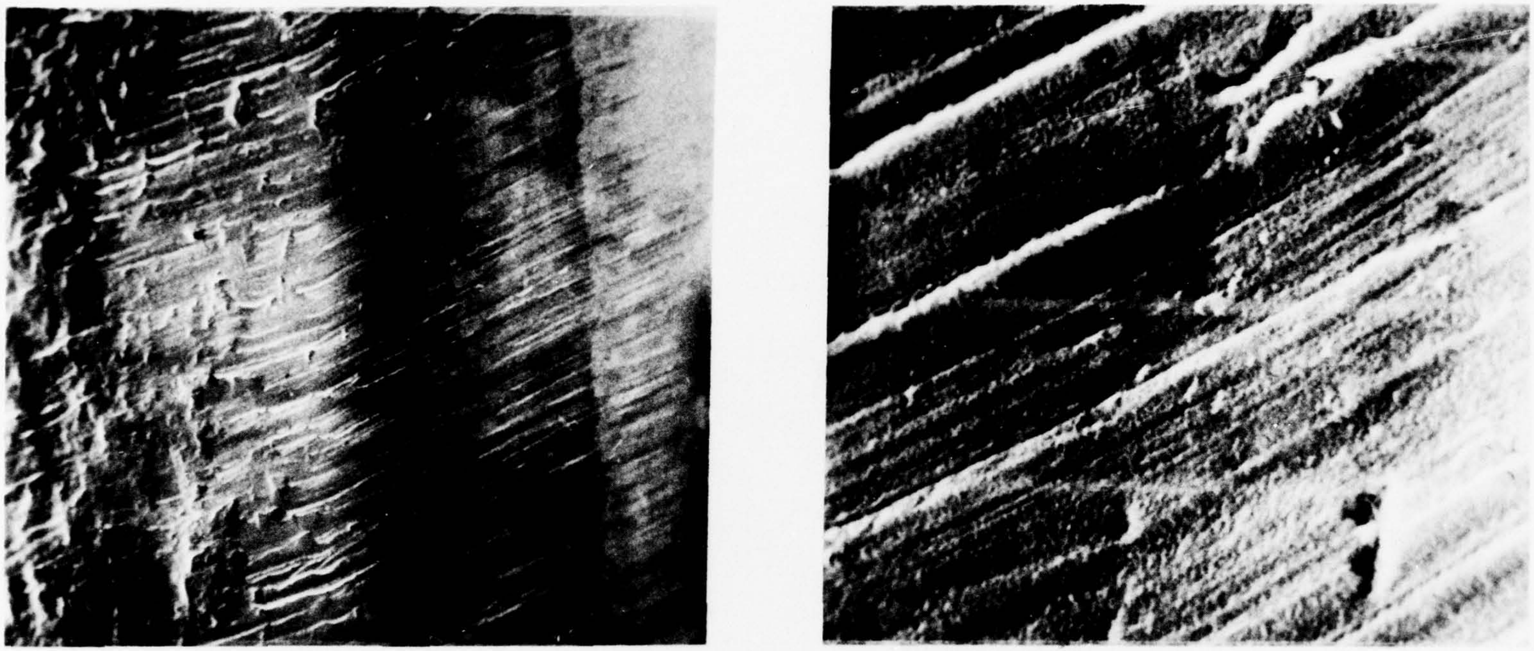
a) 100X b) 1000X

Figure 14. SEM Photomicrographs of a Freshly Cut Li Metal Surface. The Li metal had been exposed to the GTE-Sylvania battery assembly room.



c) 3000X

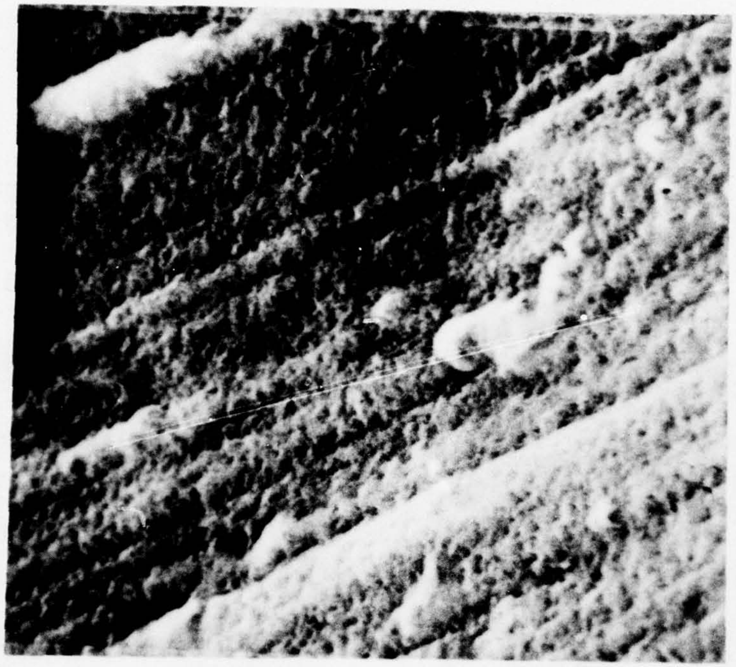
Figure 14. (Concluded)



a) 100X

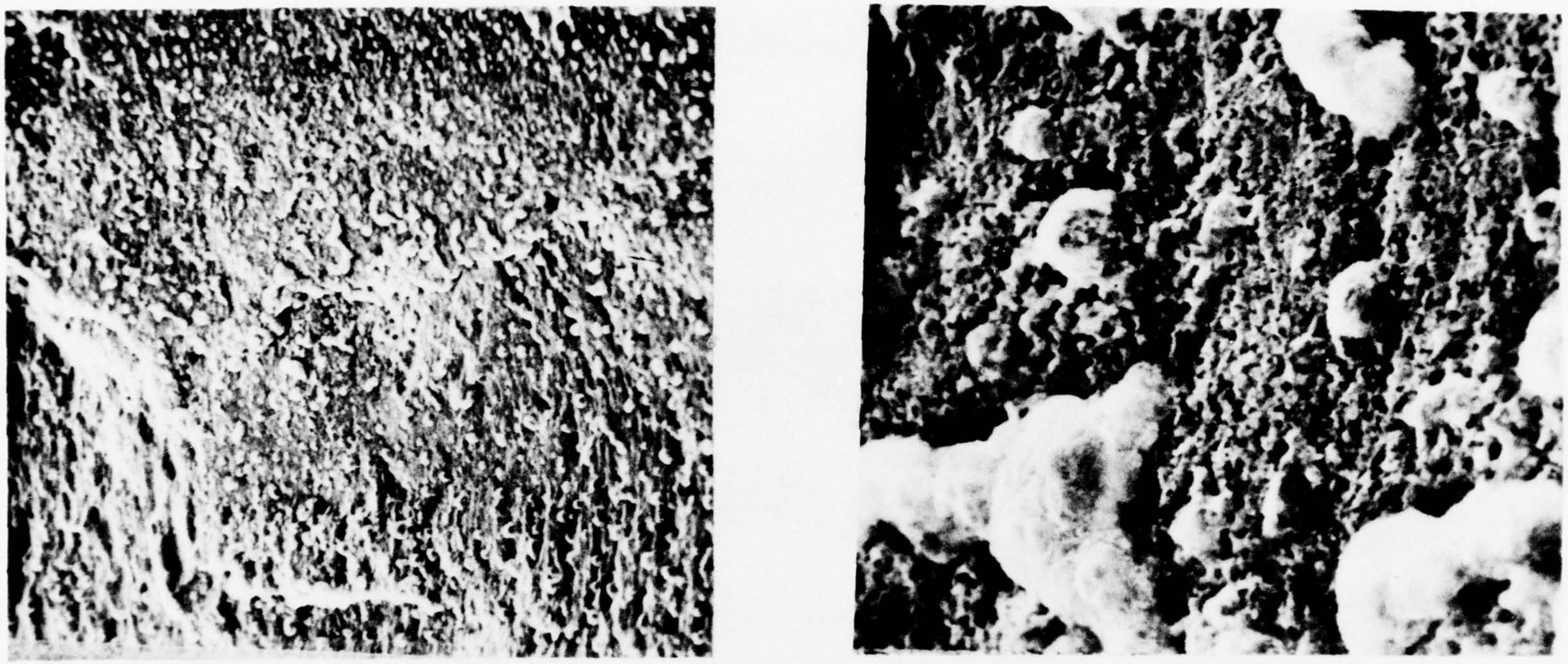
b) 1000X

Figure 15. SEM Photomicrographs of a Freshly Cut Li Metal Surface. The Li metal had been exposed to the GTE-Sylvania Laboratory argon dry box.



c) 3000X

Figure 15. (Concluded)

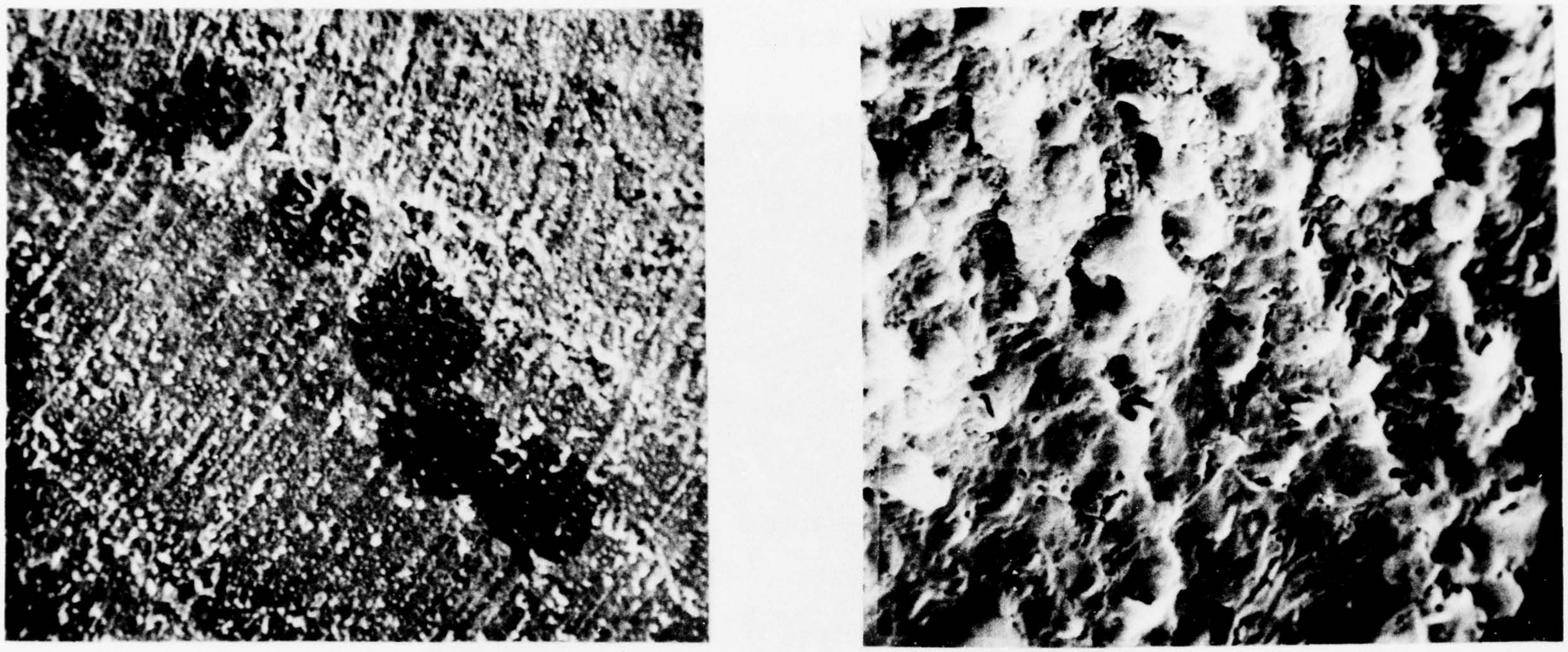


a) 100X b) 1000X

Figure 16. SEM Photomicrographs of a Freshly Cut Li Metal Surface After 70 Hours Exposure to $SOCl_2$. The metal had originally been exposed to the GTE-Sylvania Laboratory argon dry box.

c) 3000X

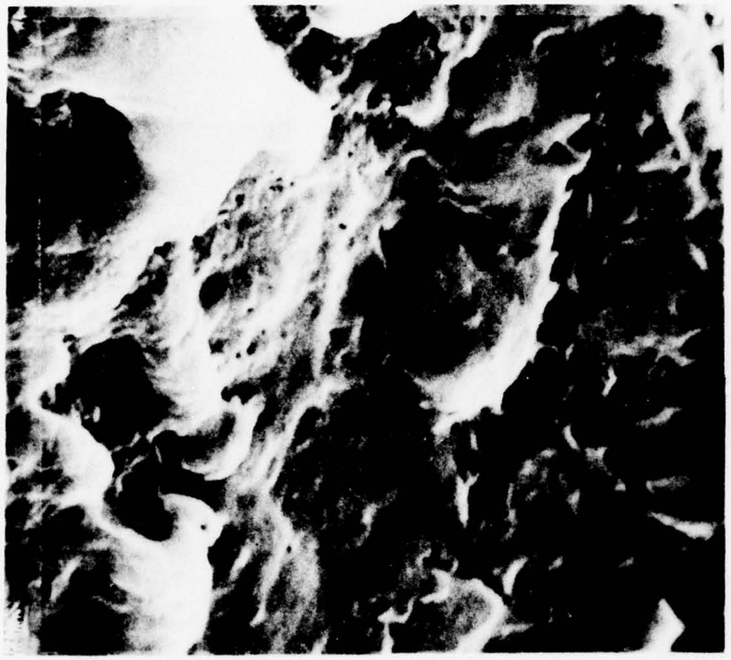
Figure 16. (Concluded)



a) 100X

b) 1000X

Figure 17. SEM Photomicrographs of a "Freshly Cut" Li Metal Surface After 70 Hours Exposure to $SOCl_2$. The metal had originally been exposed to the GTE-Sylvania battery assembly room.



c) 3000X

Figure 17. (Concluded) SEM Photomicrographs of a "Freshly Cut" Li Metal Surface After 70 Hours Exposure to $SOCl_2$. The metal had originally been exposed to the GTE-Sylvania battery assembly room.

4. AES ANALYSIS OF BLACK SPOTS

An intensive effort was directed toward characterization of several small black spots found on the GTE-supplied Li samples. Initial studies were concentrated on discovering differences in composition between the spotted and nonspotted Li surfaces.

Li samples were initially prepared by cutting with a razor blade inside a plastic glove bag attached to the side of the AES vacuum chamber. During the sample cutting and loading process, both the glove bag and the vacuum system were continually flushed with a fast flowing dry nitrogen gas. The chamber was then sealed and immediately evacuated. A whole series of samples were evaluated including freshcut and as-received surfaces, $SOCl_2$ exposed surfaces, and spotted versus nonspotted areas.

The AES spectra were difficult to interpret due to the high oxygen levels. Ion sputtering of the surfaces did not significantly alter the oxygen levels indicating the presence of a thick oxide. As a result, sample charging was a continual problem.

AES spectra for as-received non-$SOCl_2$ exposed Li samples are shown in Figures 18 and 19. Consistent chemical differences between the spotted and non-spotted areas were unobservable. Although not immersed in liquid $SOCl_2$, several of the as-received Li samples showed significant amounts of Cl on the surface (Figure 18). This was probably due to a reaction between the Li and $SOCl_2$ vapors present in the dry box during sample cutting. As a result, it was impossible to properly characterize the black spots on either the as-received or the liquid $SOCl_2$ exposed Li samples.

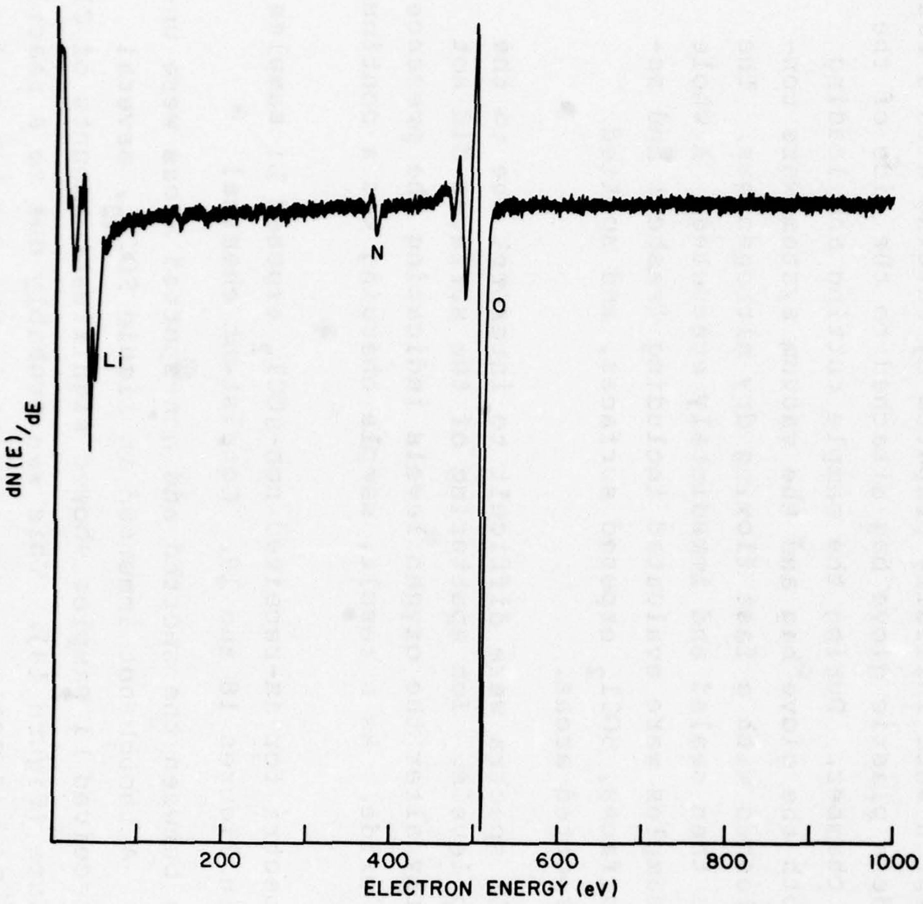


Figure 18. Typical Auger Spectrum for an As-Received, Non-$SOCl_2$ Exposed Li Metal Surface. Spectrum was taken on a black spot.

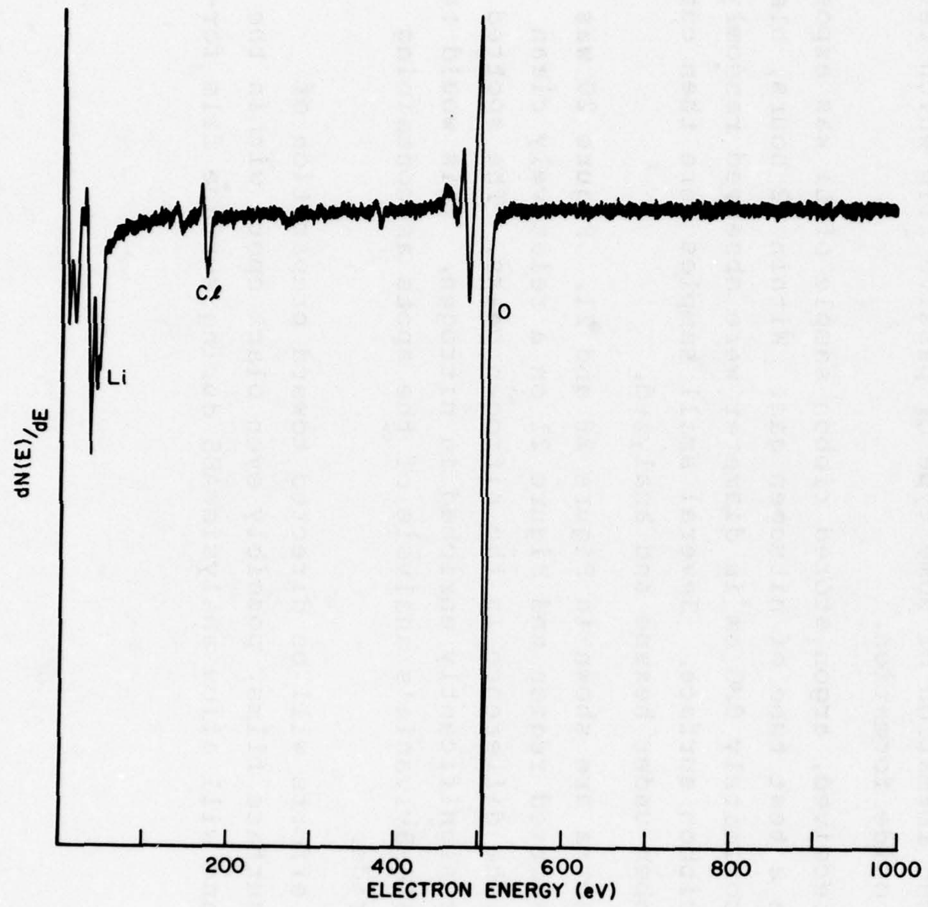


Figure 19. Typical Auger Spectrum for an As-Received, Non-$SOCl_2$ Exposed Li Metal Surface. Spectrum was taken on a non-spotted area.

Since the earlier AES work on mineral-oil-stored Li had resulted in good Auger spectra with low oxygen peaks, a decision was made to dip subsequent Li samples in hexane prior to loading into the AES vacuum chamber. As before, samples were handled in a nitrogen gas glove bag attached to the chamber. The technique proved successful in that oxygen peaks, although initially strong, were easily reduced by ion sputtering. It would appear the hydrocarbon induced formation of some type of passive film which retarded further oxide formation.

An as-received, argon stored ribbon sample of Li was exposed overnight in a test tube of nitrogen gas. Within 12 hours, black nodules approximately 0.3 cm in diameter were observed randomly across the ribbon surface. Several small samples were then cut from the ribbon under hexane and analyzed.

AES spectra are shown in Figure 20 and 21. Figure 20 was taken on a spotted region and Figure 21 on a relatively clean area. Note the difference in the nitrogen peaks. The spotted area appears significantly enriched in nitrogen. This would tend to confirm GTE-Sylvania's analysis of the spots as containing lithium nitride.

Future efforts will be directed toward preparation of various Li surface films, possibly even black spots within the AES vacuum and will allow analysis AES during surface film formation.

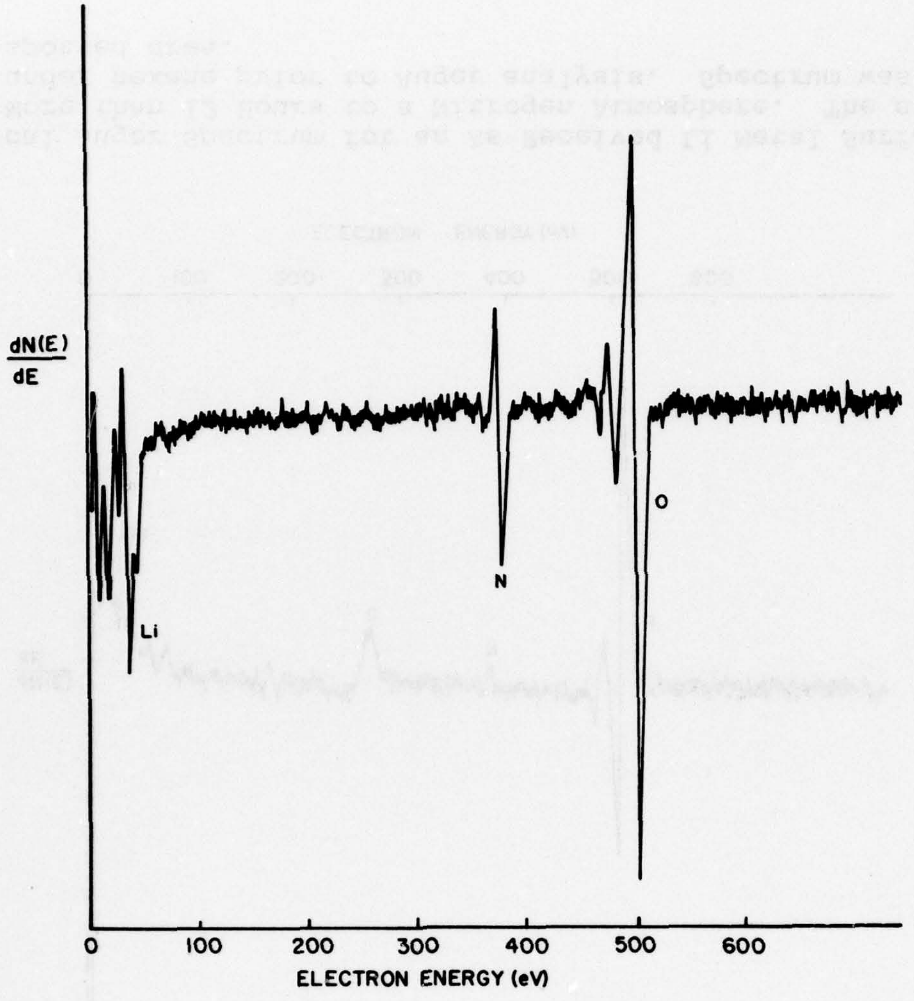


Figure 20. Typical Auger Spectrum for an As-Received Li Metal Surface Exposed for More than 12 Hours to a Nitrogen Atmosphere. The sample was cut under hexane prior to Auger analysis. Spectrum was taken on a black spot.

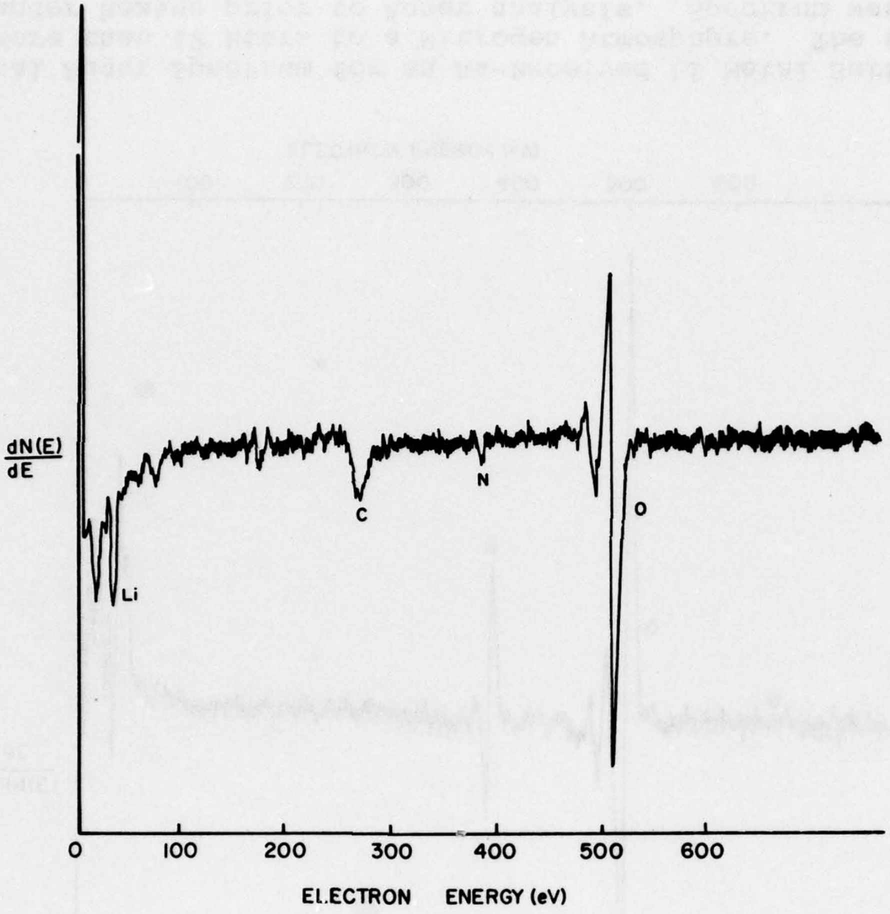


Figure 21. Typical Auger Spectrum for an As-Received Li Metal Surface Exposed for More than 12 Hours to a Nitrogen Atmosphere. The sample was cut under hexane prior to Auger analysis. Spectrum was taken on a non-spotted area.

SECTION VI

GAS MANIFOLD SYSTEM

The gas manifold inlet system is illustrated in Figure 22. The manifold contains four stainless steel cylinders, connected in parallel, capable of storing four gases. This gas handling system will allow for improved efficiency of ESCA/AES analysis in the study of "fresh" lithium surfaces exposed to dry oxygen, dry nitrogen, dry carbon dioxide, and/or water vapor. This system will also allow for lithium exposure studies to both mixtures and sequences of gases, such as a mixture of water and oxygen, or exposure to dry oxygen followed by water vapor.

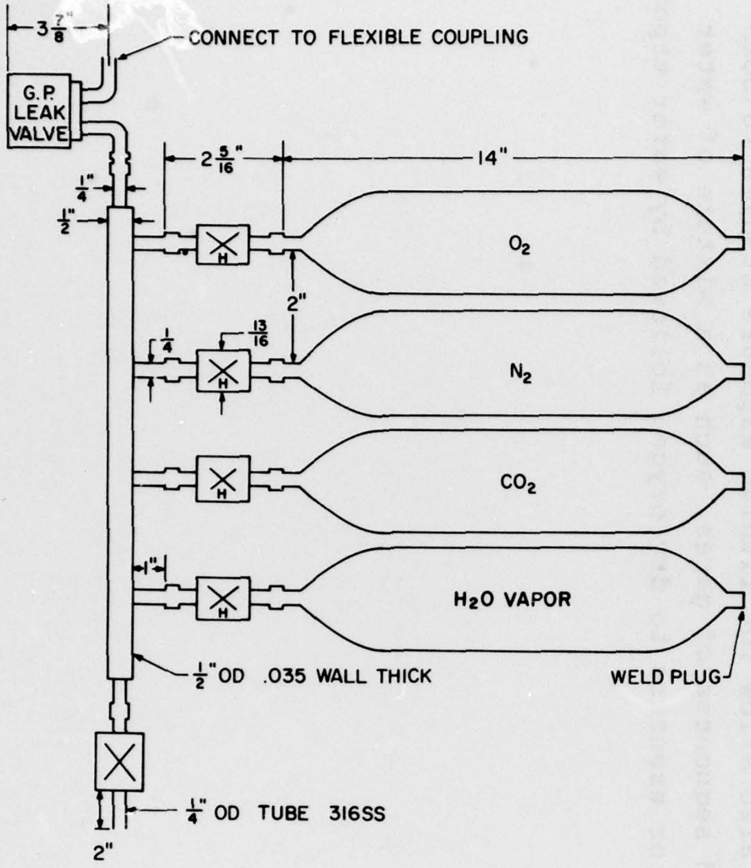


Figure 22. Gas Manifold for Vapor Exposure Studies on Fresh Lithium Metal Surfaces.

SECTION VII

FUTURE STUDIES

Next year's studies will involve the interaction of "fresh" lithium surfaces with the components of the environment, O_2, N_2, CO_2, and H_2O vapor. Freshly cut lithium specimens will be prepared and submitted into the AES vacuum chamber as described in Section 2 of this report. Ion-etching will be performed, in situ, in the AES system, in order to create a clean lithium surface. This freshly prepared surface will then be exposed to known doses of the environmental gases, both singularly and mixed. AES data will be obtained under equilibrium conditions, in order to ascertain which gas(es) chemisorb to lithium. It is anticipated that an understanding of the mechanism of atmospheric/lithium passive film formation will be determined from the above information. Studies on the interaction of this environmental film with liquid and gaseous thionyl chloride will also be examined. It will be of interest to note the status of the environmental film following $SOCl_2$ exposure. Is the film still present or has it been dissolved? It is anticipated that studies will be initiated on the nature of films formed on lithium as an anode material in open cells, with and without the presence of electrolyte.

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