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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br><b>The research program has demonstrated that thin films of <math>\text{MoS}_{2+y}</math> (<math>y = 0.3</math>) can be prepared by vapor deposition techniques. Electrical and optical properties of the films have been investigated as a function of sulfur content. The films can be electrochemically intercalated with 4 mol Li/mol <math>\text{MoS}_{2+y}</math>. Lithium diffusion coefficients were found to be in the range of <math>10^{-15} \text{ cm}^2/\text{s}</math> and are dependent</b> |                                    |                                                                                          |

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**SYNTHESIS AND PROPERTIES OF AMORPHOUS TRICALCOGENIDE  
CATHODE MATERIALS**

**FINAL REPORT**

Bruce Dunn and R. F. Bunshah  
Department of Materials Science and Engineering

August 1988

U.S. Army Research Office  
Contract/Grant Number: DAAG29-85-K-0170

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## FORWARD

This final report reviews the research accomplished over the period July 1, 1985 - June 30, 1988. The technical details mentioned in this report were more fully described in the five previous semi-annual progress reports submitted to ARO. The progress achieved over the final six months of the program (from January 1, 1988 to June 30, 1988) are contained in this report. The experimental work during this period involved the synthesis of Nb-doped films and additional electrochemical measurements.

At the time of this writing, the following items associated with the project were either in progress or were planned for the near future:

1. A small number of experimental studies are continuing. This work involves the synthesis and electrochemical properties of Nb - doped  $\text{MoS}_{2+y}$  films. This work is being used to complete the Ph.D. dissertation of Mr. H. Shin. The anticipated date of completion for his dissertation is December, 1988.
2. Technical publications based on the dissertation of Mr. Shin and the electrochemical characteristics of the  $\text{MoS}_{2+y}$  films are planned. The manuscripts should be completed during the Fall of 1988.

Upon completion, items 1 and 2 above will be sent to ARO in accordance with the usual procedures.

Finally, the authors of this report are very grateful to the U.S. Army Research Office for the support of this research project and for the support of our work on thin film cathode materials.

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## 1. STATEMENT OF THE PROBLEM

The objective of the research was to prepare thin films of amorphous chalcogenide cathode materials by vapor deposition techniques. These materials represent a novel class of electrochemical materials which possess interesting and useful properties that are not exhibited by their crystalline counterparts. The use of vapor deposition techniques is advantageous because it enabled us to modify the composition of the film while still retaining its amorphous structure. MoS<sub>3</sub> was the composition emphasized in this program because of its high capacity for lithium and its potential use as a cathode in secondary lithium batteries. One of the principal technical objectives was to use vapor deposition processes to modify the composition of the molybdenum sulfide in order to increase the electrical conductivity of the material. The breadth of the research program included the synthesis of doped and undoped sulfur-deficient MoS<sub>3</sub> films possessing various stoichiometries, the characterization of the chemical and structural nature of the films, and the investigation of their transport properties and electrochemical behavior.

## 2. SUMMARY OF RESULTS

### 2.1 Film Synthesis and Characterization

Molybdenum sulfide and niobium-doped molybdenum sulfide films were successfully synthesized by the activated reactive evaporation (ARE) process. Details of the technique have been presented in previous reports and the relationship between processing conditions and film stoichiometry is discussed in the following section. The technique involves evaporating 99.99% pure molybdenum rod or molybdenum-niobium alloys using an electron beam gun in an H<sub>2</sub>S plasma. The plasma was generated using a filament-anode geometry. The films were deposited onto soda-lime glass substrates placed beneath the water-cooled copper holder, which was designed to maintain low substrate temperature in order to develop non-crystalline films. Composition was varied by controlling the deposition variables such as deposition rate, anode current, working pressure and gas flow rate. High quality, pore free films at thicknesses up to 2 μm were produced by this method.

Sulfur content of the films was determined by an electron microprobe using wavelength dispersive analysis. The sulfur content varied from 61 a/o to 75 a/o by manipulating the above-mentioned deposition variables, producing the corresponding film composition of MoS<sub>x</sub> (1.6 < x < 3.0). Reproducible stoichiometries could be obtained by careful control of the H<sub>2</sub>S flow rate (10 to 20 SCCM) and deposition rate. (4-6 Å/s). The effect of sulfur content on film

resistivity (measured by four-point probe test) is shown in Fig. 1. The resistivity was found to increase with increasing sulfur content of the films.

Deposition rate was the most dominant variable in the deposition process. As shown in Fig. 2, there was an optimum deposition rate ( $\sim 4$  A/sec) which produced films with the maximum resistivity ( $412 \Omega \text{ cm}$ ) and sulfur content (75 a/o). It is likely that reaction between molybdenum and sulfur atoms is the controlling factor regarding the sulfur content of the films.

The lowest substrate temperature obtained in this deposition system was approximately  $200^\circ\text{C}$ . Thus a completely amorphous structure was not obtained. Instead, the structure of most films, as determined by X-ray diffraction, was considered to be poorly-crystalline (pc) in that the diffraction peaks, although broad, were discernible. There was slight preferred orientation along the [100] direction. This pc-structure still shows good intercalation properties of lithium atoms as reported in a subsequent section.

Fig. 3 shows the Raman spectra for two films: a) a near stoichiometric  $\text{MoS}_2$  films and b) a film with about 75 a/o sulfur. Raman spectra of the two films are very similar except for one distinct peak in Fig. 3b at  $230 \text{ cm}^{-1}$ . Figs. 3a and 3b show four strong peaks (marked A) which belong to  $2\text{H-MoS}_2$  with other small broad peaks (marked C) which belong to amorphous  $\text{MoS}_3$  (a- $\text{MoS}_3$ ). The extra peak in Fig. 3b at  $230 \text{ cm}^{-1}$  corresponds to a- $\text{MoS}_3$ . The other C-peaks are, in general, more recognizable in Fig. 3b which has higher sulfur content. Raman spectra thus show that  $\text{MoS}_{2+y}$  films exhibit the dual structure characteristics of  $2\text{H-MoS}_2$  and a- $\text{MoS}_3$  and that the latter phase becomes more distinct with an increase in sulfur content of the film.

## 2.2 Electrical and Optical Properties

Electronic resistivity of the film varied from 0.06 to  $412 \Omega\text{-cm}$  depending on sulfur content. This behavior is shown in Fig. 1. This range of resistivities enabled us to study the influence of resistivity on electrochemical behavior. Niobium doping decreased the resistivity for a composition with maximum sulfur content by nearly three orders of magnitude, to  $0.1 \Omega\text{-cm}$ .

Fig. 4 displays the effect of temperature on the dc conductivity of a film with high sulfur content (70.4 a/o,  $327 \Omega\text{-cm}$ ). The dc conductivity is plotted against both  $T^{-1/4}$  and  $T^{-1/3}$  in order to identify the transport mechanism in the films. The fact that both curves are linear (within experimental error) suggests that transport involves a combination of mechanisms: two

dimensional electron hopping, as observed in crystalline MoS<sub>2</sub>, and electron hopping between localized states.

Optical transmission measurements were made over a range of wavelengths, from 0.2 to 2.6  $\mu\text{m}$ . For the all cases, a plot of  $(\alpha E)^{1/2}$  vs.  $E$  was nearly linear. The bandgaps of the films vary from 1.1 to 1.2 eV (Fig. 5), which is consistent with the bulk materials. Doping the film with niobium did not change the bandgap.

## 2.3 Electrochemical Properties

### Fabrication of Electrochemical Cell

Special electrochemical cells were constructed to solve the problem of maintaining electrical contact to the delicate cathode films while sustaining electrolyte exposure. The cell is shown in Fig. 6. First, gold was evaporated onto the edges of the surface of the MoS<sub>2+y</sub> films. This step provides reliable electrical contact with low contact resistance. The glass substrate rests on a Teflon stage while a Teflon reservoir ring rests upon the film itself. Steel electrodes are inserted through another Teflon block above the liquid electrolyte reservoir to make contact with the lithium foil anode. The liquid electrolyte was 1M LiAsF<sub>6</sub> in propylene carbonate. Four layers of electrolyte saturated fiberglass filter paper separates the lithium foil from the MoS<sub>2+y</sub> cathode. Steel electrodes hold the lithium foil in place. These components are placed between aluminum pressure plates which are then bolted together. Since Teflon deforms slightly with an applied force, a fairly good seal is achieved and very little electrolyte escapes from the reservoir.

The liquid electrolyte, LiAsF<sub>6</sub> in propylene carbonate, and the lithium anode are extremely water sensitive. Thus, all cell construction and testing were executed in a glove box with continuous argon flow.

### Cell Discharge

Several cells were assembled using films whose initial resistivities ranged from  $5 \times 10^{-2} \Omega\text{-cm}$  to nearly  $400 \Omega\text{-cm}$ . Cell characteristics are summarized in Table 1. The working voltage, open circuit voltage and the lithium capacity all compared favorably with bulk MoS<sub>3</sub>. In all cases the films exhibited rather good lithium intercalation properties; from 2.4 to 4.2 mol Li/mol MoS<sub>2+y</sub>. Film capacity was determined by dividing the quantity of lithium intercalated by the quantity of MoS<sub>2+y</sub> exposed to the electrolyte. To find the amount of lithium, it is assumed that

one Li atom is intercalated for every electron that passes through the discharge circuitry. The quantity of  $\text{MoS}_{2+y}$  was determined by multiplying the surface area of the reservoir by the film thickness (measured by Dektak profilometer) and dividing by the molar volume of the bulk material. The bulk value was used because the conventional methods for determining the density were far too inaccurate for thin films. The calculated capacity of the films is linearly dependant on film density. The lithium intercalation value of 4.9 for cell 163 is not accurate as the cell probably suffered from electrolyte leakage problems.

Typical cell discharge characteristics are shown in Fig. 7. After a relatively short time at a higher voltage, the working voltage dropped sharply to about 2.2V and then gently declined for the life of the cell. The cell was considered as fully lithiated when the working voltage reached approximately 1.2V. Initial studies indicated that the lithium intercalation was reversible although the extent of reversibility was not established. From the familiar relationship,  $\Delta G = -nFE$ , the Gibb's free energy was calculated to be approximately -280kJ/mole for the reaction between Li and the unlithiated  $\text{MoS}_{2+y}$  thin film. This value is larger than that for the Li -  $\text{TiS}_2$  reaction (213 kJ/mole).

One important result for the Li - thin film  $\text{MoS}_{2+y}$  cells was that the current density was substantially lower than that reported for the bulk material. Virtually all the results in the present investigation had values in the range of  $1 \mu\text{A}/\text{cm}^2$  as compared to bulk materials which generally have current densities around  $0.1\text{mA}/\text{cm}^2$ . The reason for this behavior is not understood but apparently thin film electrochemical cells seem to be characterized by low current densities in the  $\mu\text{A}/\text{cm}^2$  regime.<sup>2,3</sup>

## Lithium Diffusion

Lithium diffusion coefficients in  $\text{MoS}_{2+y}$  films were determined using the current-pulse relaxation technique.<sup>4</sup> Selected samples were interrupted during their discharge and allowed to equilibrate until their open circuit potentials stabilized. A large constant current pulse then creates an overpotential at the cell's terminals. By recording the overpotential as a function of time, one can derive the lithium chemical diffusion coefficient in  $\text{MoS}_{2+y}$  by solving the following equation.

$$\sqrt{D} = \left( \frac{mV}{FA\sqrt{\pi}} \right) (It) \left( \frac{t^{1/2}}{\Delta E} \right)$$

where:  $\Delta E$  = overpotential  
 $m$  = slope of voltage vs. composition ( assumed to be .25)  
 $V$  = molar volume of cathode ( assumed to be 40 cm<sup>3</sup>/mole )  
 $I$  = magnitude of the current pulse in amperes  
 $t$  = duration of the pulse in seconds  
 $F$  = 96,500 coulombs/mole  
 $A$  = cathode surface area

A computer program was written to control the galvanostat's pulse delivery and monitor the overvoltage as a function of time. The computer then does a linear regression to find the  $(t^{1/2}/\Delta E)$  coefficient and, subsequently, the desired diffusion coefficient. The diffusion coefficients for lithium in various cells are listed in Table 2 as a function of lithium intercalation. The data for two cells are plotted in Fig. 8.

The results of the lithium diffusion studies indicate that  $D_{Li}$  ranged from 10<sup>-14</sup> to 10<sup>-17</sup> cm<sup>2</sup>/sec during the life of the cell.  $D_{Li}$  decreased as more lithium was intercalated into the cathode film. These are the first measurements to be reported for the diffusion of lithium in MoS<sub>2</sub> or MoS<sub>3</sub> materials.

### Film Impedance

Film impedance measurements were taken several times as the cell was discharging. The resistance across the length of the film could be measured while in the cell by using a lock-in amplifier to determine the magnitude and phase of an a.c. signal across the film. By making these measurements over a reasonable frequency range (10Hz -100kHz ), it is possible to graph the results in a complex plane representation. The data in Fig. 9 indicate that the partially lithiated MoS<sub>2+y</sub> film is a mixed conductor (i.e., both electronic and ionic conduction) consisting of both film and interfacial resistances. In the present study the film impedance is of primary importance and this value is taken from the intersection of the curve with the real axis. Fig. 9 shows very clearly that the real impedance in cell 153 increased continuously with the addition of lithium. The fact that the shape of the complex impedance curves do not change very much with intercalation suggests that electrical contact to the film remains consistent for the lifetime of the cell.

## **Cell Impedance**

Cell impedance measurements were made in a similar fashion to those of film impedance. A small a.c. signal is input at the anode and the lock-in amplifier measures the a.c. voltage drop across the entire cell. In this way cell impedance can be measured while the cell is discharging under d.c. conditions. The data is shown as a function of lithium content in Fig. 10 where film impedance is divided by cell impedance. A linear correlation is observed indicating that the increase which occurs in cell impedance can be largely attributed to the increase observed in film resistance.

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3. K. Kanehori, Y. Ito, F. Kirino, K. Miyauchi and T. Kudo, Solid State Ionics 18/19, 818 (1986)
4. S. Basu and W. L. Worrell, in Fast Ion Transport in Solids, eds. P. Vashishta, J. N. Mundy and G. K. Shenoy (North-Holland, 9179) 149-152.

## **3. PARTICIPATING SCIENTIFIC PERSONNEL AND ADVANCED DEGREES AWARDED**

Dr. R. F. Bunshah

Mr. C. T. Chu

Dr. C. V. Deshpandey

Dr. B. Dunn

Mr. P. Fuqua

Mr. H. Shin (Ph.D. degree to be awarded December, 1988).

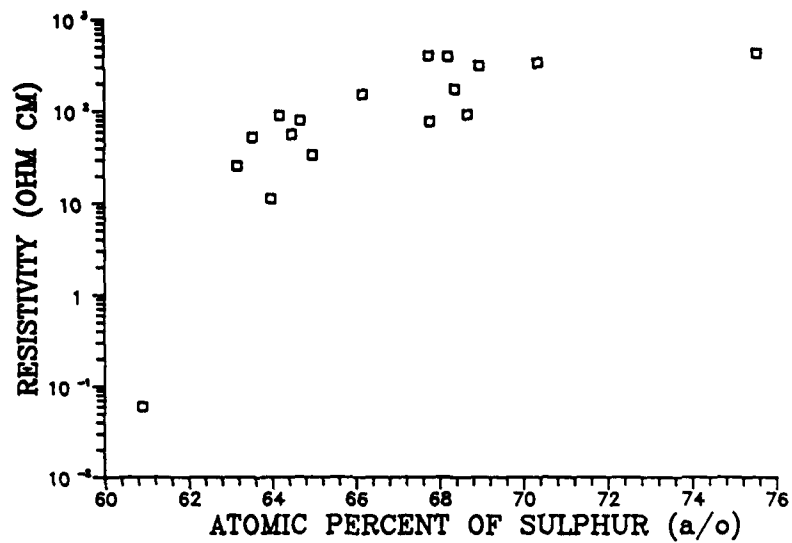
**TABLE 1 CELL CHARACTERISTICS**

| film # | resistivity | ave. current density           | capacity    | comments        |
|--------|-------------|--------------------------------|-------------|-----------------|
| 130    | 4.7 ohm-cm  | 0.77 $\mu\text{A}/\text{cm}^2$ | 2.4 mol/mol | chem. initiated |
| 132    | 149.7       | 1.23                           | 2.4         | pass. discharge |
| 145    | 386.0       | 0.77                           | 4.1         |                 |
| 151    | 1.57        | 0                              | -           | damaged         |
| 152    | 3.16        | 0.92                           | 3.9         |                 |
| 153    | 0.059       | 0.92                           | 4.2         |                 |
| 156    | 309.0       | 0                              | -           | damaged         |
| 163    | 0.063       | 0.77                           | 4.9         |                 |

**TABLE 2 DIFFUSION OF LITHIUM IN  $\text{MoS}_{2+y}$  CELLS (D in  $\text{cm}^2/\text{sec}$ )**

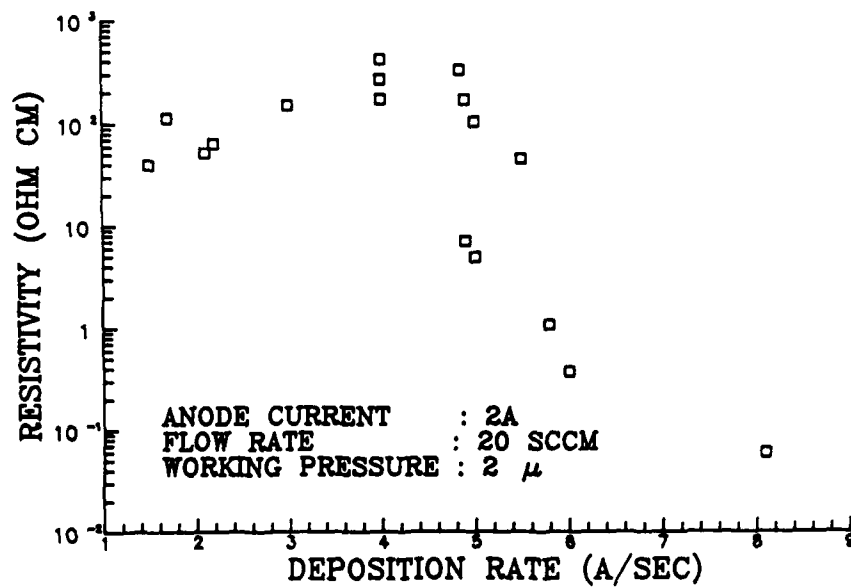
| x   | $D_{\text{Li}}$ cell 145 | $D_{\text{Li}}$ cell 152 | $D_{\text{Li}}$ cell 153 |
|-----|--------------------------|--------------------------|--------------------------|
| 0.5 | $3.5 \times 10^{-16}$    | $1.2 \times 10^{-14}$    | $2.1 \times 10^{-15}$    |
| 1.0 | $4.5 \times 10^{-16}$    | $9.8 \times 10^{-15}$    | $2.0 \times 10^{-15}$    |
| 1.5 | $7.2 \times 10^{-16}$    | $8.7 \times 10^{-15}$    | $1.1 \times 10^{-15}$    |
| 2.0 | $5.0 \times 10^{-16}$    | $3.5 \times 10^{-15}$    | $3.9 \times 10^{-16}$    |
| 2.5 | $1.8 \times 10^{-16}$    | $2.8 \times 10^{-16}$    | $2.0 \times 10^{-16}$    |
| 3.0 | $2.0 \times 10^{-17}$    | $2.5 \times 10^{-17}$    | $6.5 \times 10^{-17}$    |

FIGURE 1



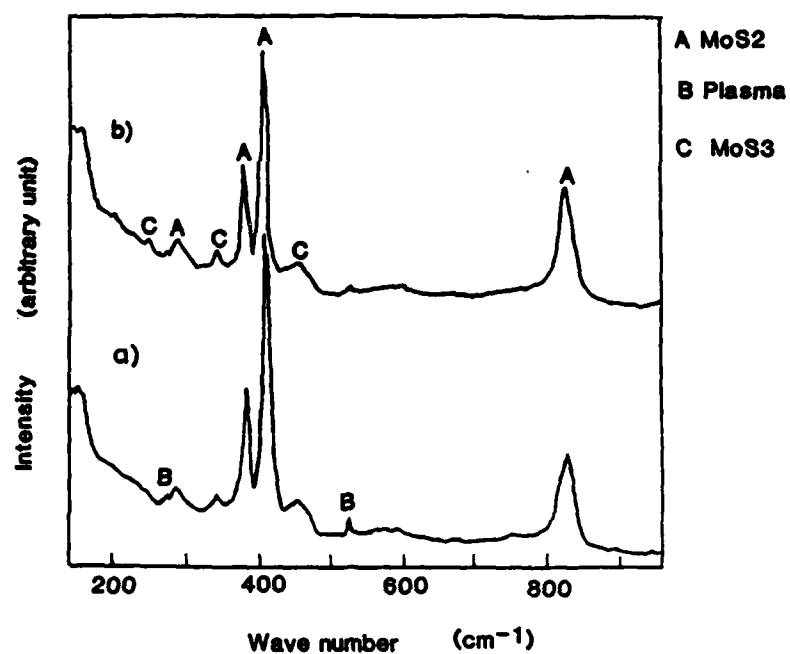
RESISTIVITY VS. SULFUR CONTENT

FIGURE 2



RESISTIVITY VS. DEPOSITION RATE

FIGURE 3



RAMAN SPECTRA

FIGURE 4

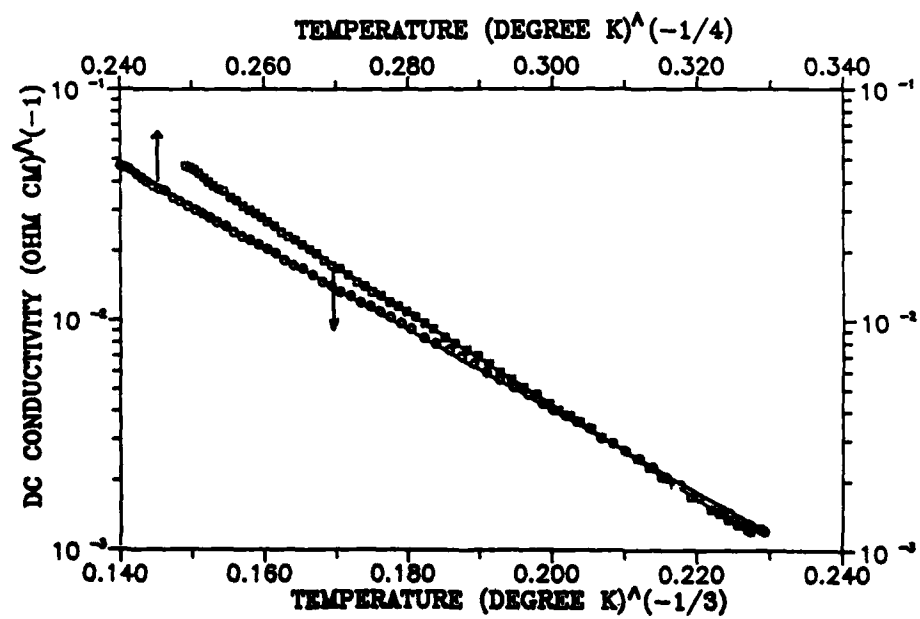
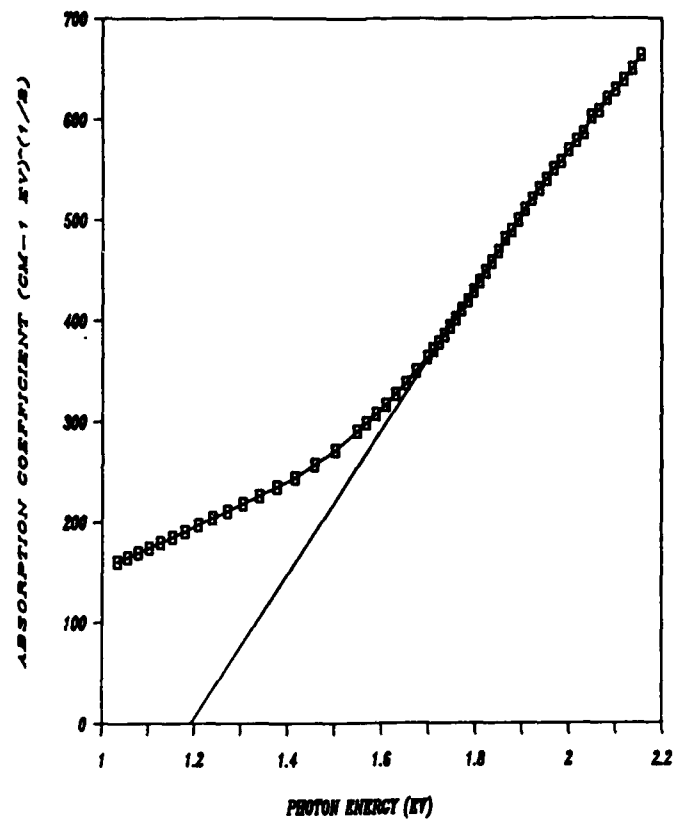


FIGURE 5



OPTICAL BANDGAP OF  $\text{MoS}_{2+y}$  FILM

FIGURE 6

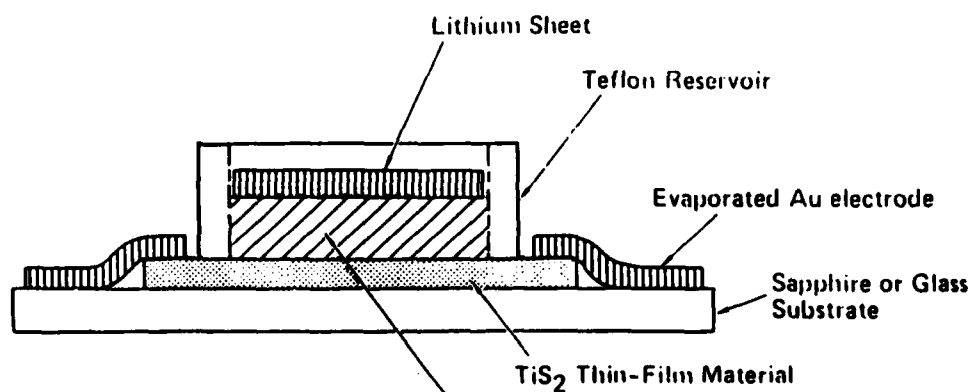


FIG. 6. Glass Filter Pump with Li-O-E in PC

FIGURE 7

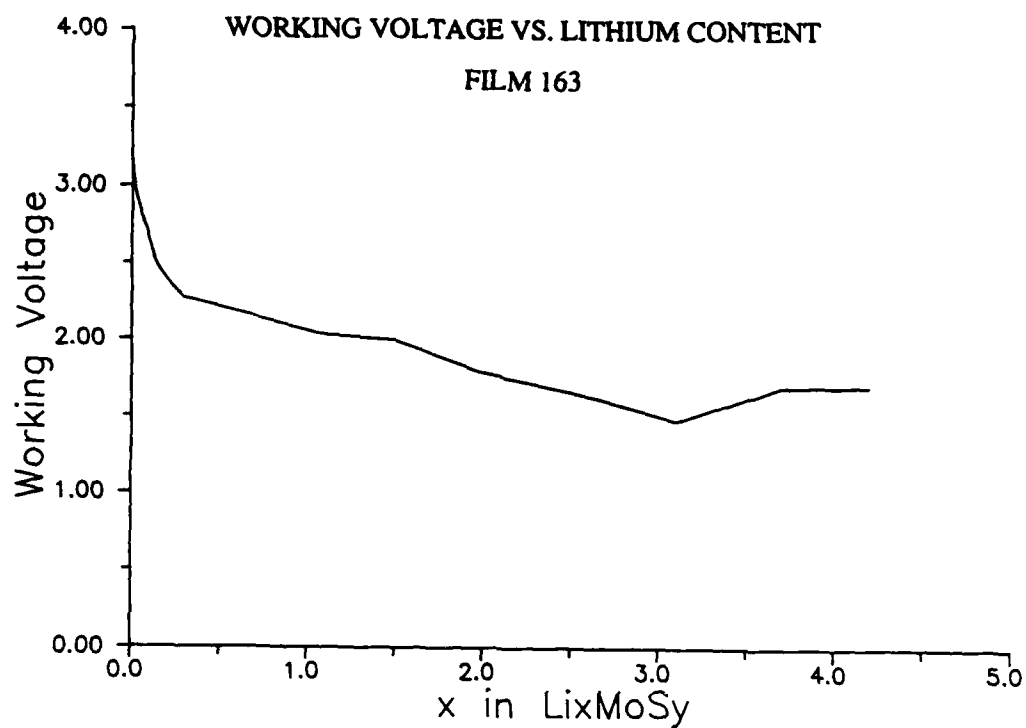


FIGURE 8

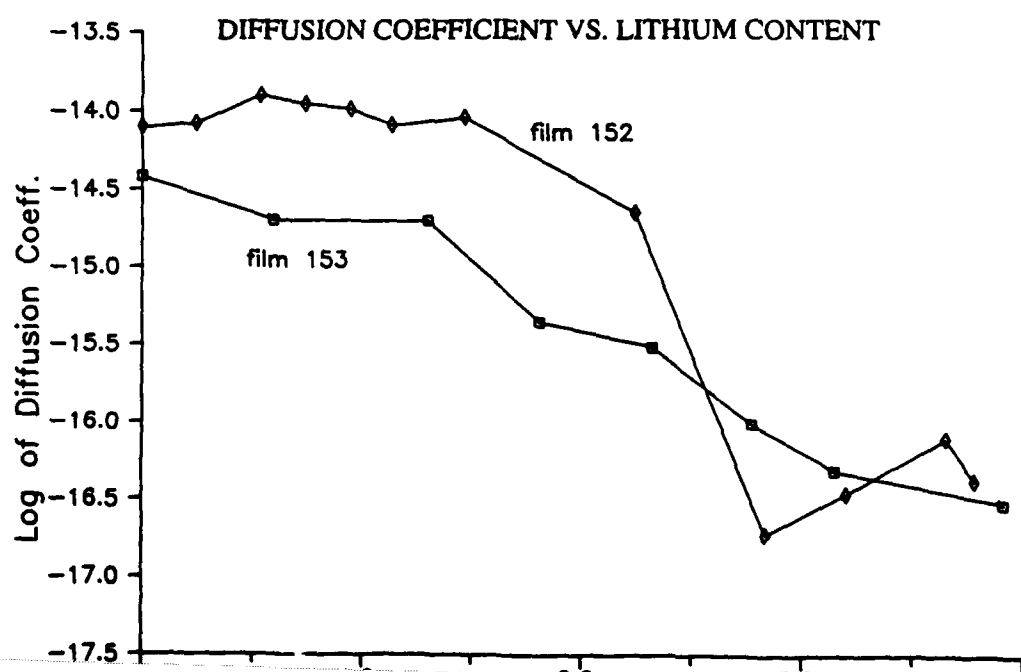


FIGURE 9

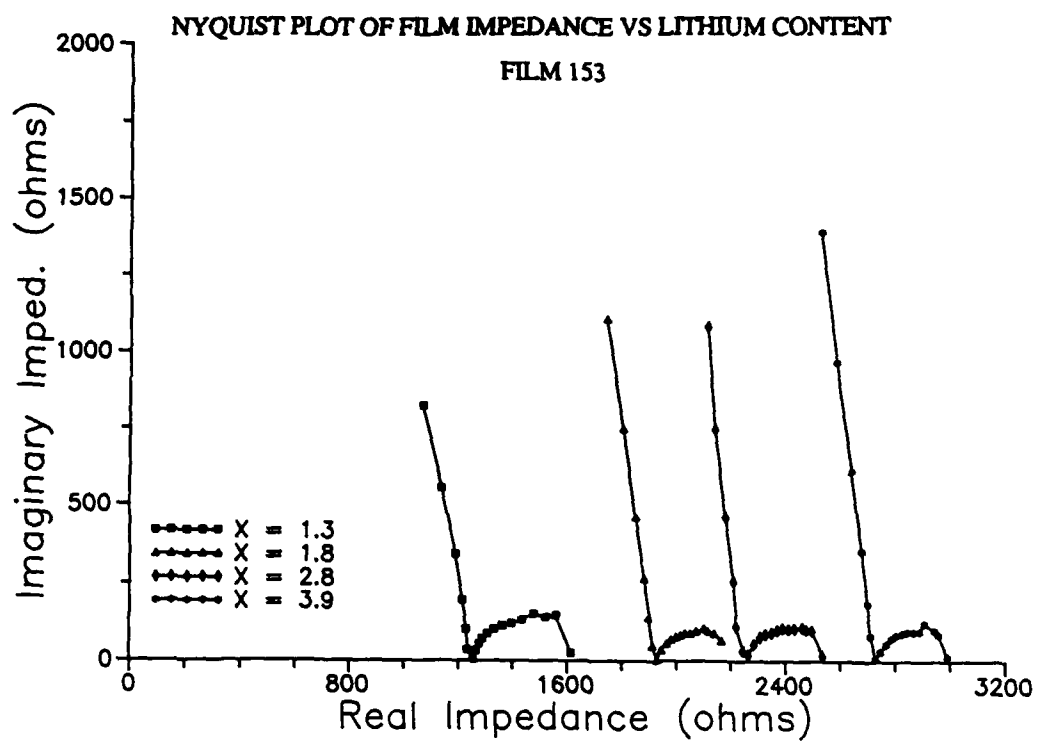


FIGURE 10

