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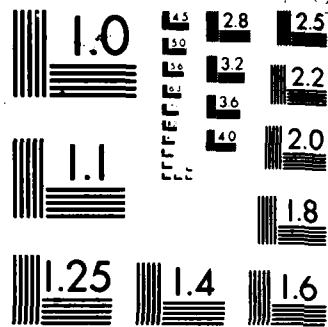
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Effects of Argon-Ion Bombardment on the Basal Plane Surface of MoS₂

JEFFREY R. LINCE, DAVID J. CARRÉ,
and PAUL D. FLEISCHAUER
Chemistry and Physics Laboratory
Laboratory Operations
The Aerospace Corporation
El Segundo, CA 90245

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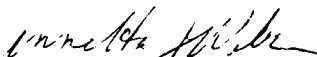
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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



ANNETTA WEBER, Capt, USAF
MOIE Project Officer
SD/YNSA



JOSEPH HESS, GM-15
Director, AFSTC West Coast Office
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The surface composition and structure of the basal plane surface of MoS ₂ was modified by 10 keV argon-ion bombardment and then studied with x-ray photoelectron spectroscopy. The rigid shift of both sulfur and molybdenum core levels is demonstrated to result from the formation of a small amount of Mo islands on the surface, although there is no a priori evidence for changes in oxidation state at the surface after bombardment. This conclusion is suggested by recent data on the deposition of metals on the		

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20. ABSTRACT (Continued)

MoS₂ basal plane that indicate a strong correlation between the Mo or (S) binding energy and the electronegativity of the metal. Our results are compared with those of previous studies and are explained in terms of recent theories on the effects of surface ion bombardment that suggest that chemical/thermal effects dominate over substrate mass effects for higher projectile-ion mass and energy.



PREFACE

The authors thank Jeffrey Childs for his assistance in taking the XPS spectra, and Dr. H. K. A. Kan for helpful discussions related to the original concept for the experiments.

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I. INTRODUCTION

The anisotropy of the MoS₂ crystal lattice is due to its layered, two-dimensional structure. Cleavage between layers produces a highly inert surface, while cutting along an edge plane causes the formation of dangling bonds that are considerably more reactive. Thus, the catalytic activity of MoS₂ has been ascribed mainly to adsorption on edge planes,^{1,2} whereas the excellent lubricating properties of MoS₂ have been attributed to the basal plane stability.³

The effects of noble-gas-ion bombardment on the basal plane of MoS₂ single crystals have been studied for over 15 years,^{1,4-6} with the goal of determining whether defects could be produced in the highly unreactive basal plane surface so that its catalytic activity might be increased.^{4,5} Such studies also have implications for improved solid lubricant performance, since ion bombardment (IB) might also be used to determine whether the reactivity of MoS₂ toward various metals could be increased with a corresponding increase in adhesion.

Despite the numerous studies, there is still extensive controversy about the effect of IB on the MoS₂ basal plane surface. Feng and Chen used Auger electron spectroscopy (AES) to characterize the effect of 300 eV Ar⁺ ions on the basal plane surface.⁵ After IB, the ratio of the S to Mo Auger signal decreased by a factor of ~ 5. They used this result, in addition to Auger peak shape analysis, to show that sulfur had been preferentially sputtered, and that islands of molybdenum metal had formed on the surface. They attributed the increase in chemisorption after IB, observed by Williams et al.,⁴ to the high reactivity of Mo metal.

In contrast, Davis and Carver reported that bombardment with 1 keV Ne⁺ ions produced no detectable metallic Mo. Using x-ray photoelectron spectroscopy (XPS) and ion scattering spectroscopy, they argued that the reduction in the S/Mo ratio was due mostly to the creation of sulfur surface vacancies, even for ion doses of $\sim 1 \times 10^{18}$ cm⁻². The only effect of ion bombardment on

the XPS spectra was to broaden the Mo peak slightly. There were no apparent Mo or S peak shifts.⁶

Matsunaga et al. have investigated the vapor adsorption and IB of MoS₂ edge and basal surfaces.¹ Their AES peak ratios implied that the basal plane surface was initially "enriched" (as compared to the bulk) with sulfur. After IB with 5 keV Ar⁺ ions, they observed a drop in the AES S/Mo ratio (by a factor of 2), which they interpreted to result from removal of an excess surface sulfur layer so that the new surface represented the bulk stoichiometry.

Clearly, the above studies lack consensus on the effect of IB on the basal plane surface of MoS₂. This report presents new ion-bombardment results, which will be compared with those of the previous studies and discussed in light of recent theories of IB effects and sputtering mechanisms.

II. EXPERIMENTAL SECTION

Clean basal plane surfaces were produced by cleaving natural molybdenite crystals in a N_2 -filled glove bag and then transferring them to the XPS sample chamber without exposure to atmosphere. The sample chamber was pumped to a base pressure of $\sim 3 \times 10^{-8}$ torr by both turbomolecular- and cryo-pumping. The samples were analyzed either immediately or after being subjected to IB from a GCA/McPherson model 36-K ion gun. Prior to IB, the highly unreactive basal plane surface remained clean at this pressure. Even in preliminary experiments, when the sample was cleaved in air, little oxygen (< 0.02 monolayer) was detected after pumpdown, and shifts in the Mo and S XPS peaks after IB were similar to those for N_2 -cleaved samples. Ar^+ ions were used with an energy of 10 keV and a current density of $\sim 20 \mu A cm^{-2}$. Bombardment time was 40 s; therefore, total exposure was $\sim 5 \times 10^{15} cm^{-2}$. Dependence on ion dose was not explicitly investigated, although preliminary results on higher doses showed effects similar to those reported here. XPS was performed with a GCA/McPherson ESCA-36 system with a hemispherical analyzer modified to include optical multichannel detection.⁷ The XPS analysis was completed < 10 min after IB. Excitation was by the Mg-K α line, and all spectra were referenced to the adventitious carbon peak set arbitrarily to 285.0 eV from a small amount of carbon (probably graphitic) that was present as an impurity in the natural molybdenite crystals. On different samples, this carbon peak shifted in a random manner after IB, and the shifts were considerably smaller than for those of molybdenum and sulfur. There was also no evidence of chemical alteration of the small amount (< 0.2 monolayer) of carbon, so the use of carbon as a reference was deemed appropriate. (There was shifting of some of the carbon peaks to higher binding energy after deposition of metals on the clean MoS_2 basal plane surface; the shifting of the carbon peak, however, was not always correlated with metal electronegativity, whereas the shifting of the corrected Mo and S peaks was highly consistent with metal electronegativity. The relation between peak shifting and metal electronegativity will be discussed in more detail in Ref. 9.) No impurity, other than carbon, was detected by XPS.

Because of possible broadening of the peaks during the experiment, and for ease of analysis, changes in the S/Mo ratio were calculated using a product of the full width at half-maximum of the peak and peak height (a "pseudo-area") rather than by the peak height only. This pseudo-area is used solely for elucidation of stoichiometry changes, rather than absolute stoichiometry.

III. RESULTS

XPS spectra of a cleaved MoS_2 surface, both before and after Ar^+ IB, are shown in Fig. 1, and are representative of 9 pairs of Mo/S spectra of cleaved surfaces and 20 pairs of spectra of bombarded surfaces (all at $\sim 5 \times 10^{15} \text{ cm}^{-2}$) that were analyzed in this study. These spectra qualitatively match previous spectra of MoS_2 .⁸ The Mo 3d peaks of the cleaved and ion-bombarded surfaces are shown in Fig. 1a. For the cleaved surface, the $3d_{3/2}$ and $3d_{5/2}$ peaks are virtually resolved, whereas for the bombarded surfaces, those peaks overlap. This is shown in Table I to be due primarily to an increase in peak width. The corresponding S 2p spectra are shown in Fig. 1b. For the cleaved surface, the $2p_{1/2}$ peak appears as a distinct shoulder on the left side of the $2p_{3/2}$ peak. After IB, however, the peaks also broaden and the S 2p doublet appears as a single asymmetric peak.

The broadening in both the Mo and S spectra after IB is not surprising, because IB of a solid causes defect formation, surface enrichment of a bulk component, or ion beam mixing, and, consequently, a loss of symmetry in the crystal lattice. Such a loss affects the local electronic environment of each atomic type in the lattice differently, and a greater binding-energy (BE) width is detected for each element. The bombardment also produces a drop of $\sim 15\%$ in the S/Mo peak area ratio (see Table I), indicating that there has been preferential sputtering of the sulfur.

The most striking effect of ion bombardment is that both the Mo and S BEs shift downward by $\sim 0.85 \text{ eV}$ and $\sim 0.73 \text{ eV}$, respectively. These shifts cannot be explained by gross chemical changes in the surface region. Partial conversion of the Mo(IV) (in MoS_2) to metallic Mo would give rise to two Mo $3d_{5/2}$ peaks: the Mo(0) peak would appear $\sim 1.2 \text{ eV}$ below the Mo(IV) peak.⁸ However, only a single, slightly broadened Mo $3d_{5/2}$ peak is seen after IB. This might result from complete conversion of Mo(IV) to Mo(0) within the depth range of the detected photoelectrons ($\sim 30 \text{ \AA}$). But complete conversion does not occur: Only a small loss of sulfur ($<10\%$) was detected by XPS, and the Mo would exist in the elemental state only if no sulfur were present. Similarly,

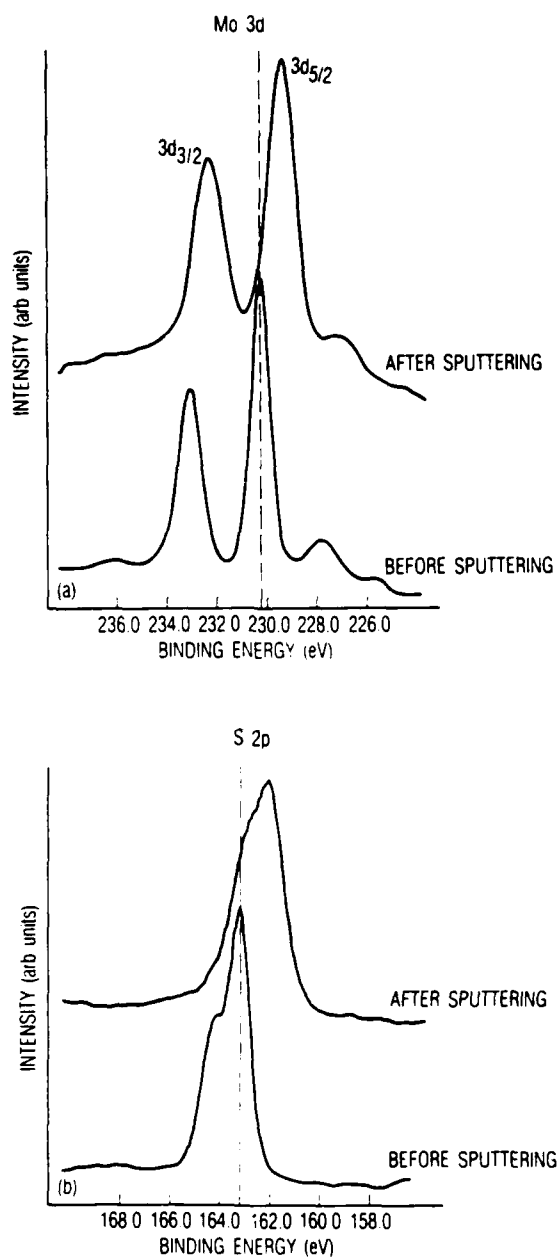


Figure 1. Examples of x-ray photoelectron spectra of the MoS₂ basal plane surface: (a) Mo 3d doublet before and after argon-ion bombardment; (b) S 2p doublet before and after argon-ion bombardment. All peaks were referenced to carbon at 285.0 eV. These spectra are representative of the many spectra taken in this study.

Table I. Changes in the S and Mo XPS Peaks as a Result of Argon-Ion Bombardment

	Cleaved surface	After ion bombardment	Change
S/Mo ("pseudo-area" ratio)	1.04 ± 0.07	0.88 ± 0.05	-15%
Mo 3d _{5/2} BE ^a	230.22 ± 0.06	229.37 ± 0.31	-0.85
S 2p _{3/2} BE ^a	163.26 ± 0.05	162.53 ± 0.23	-0.73
Mo 3d _{5/2} width ^b	0.90 ± 0.03	1.24 ± 0.06	+38%
S 2p _{3/2} width ^b	1.67 ± 0.03	2.04 ± 0.10	+22%

^aAll binding energies are in eV, including Mo and S BE changes.

^bAll peak widths are in eV and are measured as full widths at half-maximum (changes are in %).

there is no a priori way of explaining the shift of the S 2p peak to lower BE, since that would imply reduction of sulfur, which is unlikely because sulfur cannot be reduced any further than an oxidation state of -2.

IV. DISCUSSION

Our observations can be explained when correlated with previous results on the interaction of metals with the MoS₂ basal plane surface. Recently an XPS study of the interaction of small amounts of metals (<20 Å) with cleaved MoS₂ basal plane surfaces was conducted in our laboratory.⁹ Most of the metals tested did not exhibit detectable chemical reactivity with the MoS₂ basal plane. Instead, the interaction was simply electrostatic, i.e., caused by the differences in electronegativity/work function between the metal and the semiconductor. Such an electrostatic interaction results in "band-bending" as described by McGovern et al.,¹⁰ whereby electrons flow across the metal-semiconductor interface to equalize the Fermi level of the system, creating a Schottky barrier. This behavior is summarized in Figs. 2a and 2b, where the Mo 3d and S 2p BE shifts are shown to correlate linearly with the electronegativity of the deposited metallic elements.

When the mean Mo 3d and S 2p BEs for the ion-bombarded basal plane surface are plotted on Figs. 2a and 2b, respectively, the result is striking. In both figures the point on the curve that corresponds to the BEs of Mo and S after ion bombardment also appears to correspond closely to the Pauling electronegativity of Mo metal (=2.16)¹¹ to within experimental error. Bombardment with 10 keV Ar⁺ ions, therefore, produces a situation that is similar to the deposition of Mo metal on an undisturbed MoS₂ basal plane surface. The Mo metal is probably in the form of small islands on the surface, since small amounts of deposited metals tend to form disconnected islands on the MoS₂ basal plane rather than forming a continuous layer.¹² Since the Mo islands probably cover very little of the MoS₂ surface, the metallic Mo(0) 3d peak may not be intense enough to be observed. Although there is little direct evidence of a Mo(0) 3d peak in the XPS spectra, the effect of this metallic Mo on the BEs of the Mo(IV) 3d and S 2p peaks is dramatic.

It might be argued that the binding-energy shifts are caused by the appearance of defects at the surface that pin the Fermi level at the top of

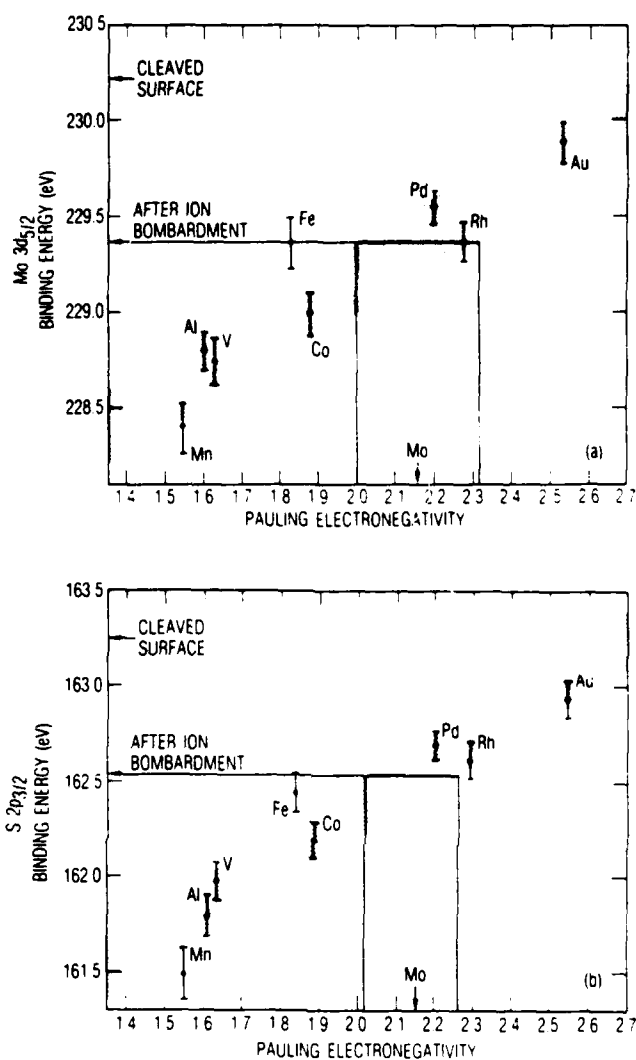


Figure 2. Plots showing the relationship between the XPS binding energy and the Pauling electronegativity of various metals deposited on the MoS₂ basal plane surface for (a) the Mo 3d_{5/2} peaks and (b) the S 2p_{3/2} peaks. The shaded regions represent the approximate uncertainty in the least-squares fit to the metal-MoS₂ data. The binding energy for the freshly cleaved surface is shown on the ordinate. The XPS binding energy of the ion-bombarded surface is represented as a line drawn from the ordinate to the curve in both (a) and (b), and the range of electronegativities that correlate with this binding energy is shown on the abscissa. The electronegativity of Mo is marked on the abscissa, and is shown to lie within this range in both (a) and (b), suggesting the formation of Mo metal islands on the MoS₂ surface. [Note: In (a), it is coincidental that the after-IB binding-energy line passes through the points for both Fe and Rh.]

the MoS₂ valence band. This is not the case, however, since Davis and Carver⁶ showed that when defect formation occurred without Mo metal formation, there was no apparent change in either the Mo or S BEs when bombarding with Ne⁺.

In a recent study by Kamaratos and Papageorgopoulos, a work function (WF) lowering of ~ 1.0 eV resulted when Fe was deposited (past a certain threshold) on the MoS₂ basal plane.¹³ They attributed the lowering to formation of metallic Mo islands at the surface. However, that change is ~ 0.5 eV greater than what would be expected from the differences in the WFs of Mo and MoS₂ (4.3 and 4.8 eV, respectively), a discrepancy they ascribed to the termination of the basal plane with sulfur atoms: The interaction of this electronegative sulfur layer with the Mo metal results in a surface dipole moment that causes the additional WF lowering. Similar results between their study and this one are not surprising, since the WF and the electronegativity are known to be at least semiquantitatively related.¹⁴ Although they have qualitatively explained the presence of Mo metal with WF arguments, our determination based on electronegativity gives a more quantitative correlation.

We can now correlate the above-described results on the ion bombardment of the MoS₂ basal plane with those of the present study. The conclusion of Matsunaga et al., that an enriched sulfur surface is removed during IB, uncovering the stoichiometric bulk underneath, is probably incorrect. There is no reason to assume that a random cleave through the crystal produces a surface region with an excess of sulfur, since decomposition of the highly stable MoS₂ and mass flow of sulfur to the surface region would have to be involved. Their initially high Auger S/Mo ratio resulted from the fact that the MoS₂ basal plane surface is always terminated by a single atomic layer of sulfur. Because of the small escape depth of Auger electrons, a higher than stoichiometric S/Mo ratio is detected. After IB, the drop in the S/Mo ratio is probably caused by Mo metal forming on the surface.

Our observations appear to agree with the study by Feng and Chen (FC),⁵ but appear to disagree with the results of Davis and Carver (DC).⁶ However, the three studies used different parameters, so this apparent contradiction

does not nullify the results of DC. Ion beam energy does not appear to explain the results since DC used an ion energy (1 keV) that was intermediate between that of FC (300 eV) and the present study (10 keV). Similarly, total ion dosage does not explain the results since FC used a higher dosage (10^{17} cm⁻², or ~20 ions/surface site) than the present study ($\sim 5 \times 10^{15}$ cm⁻²), and DC reported no metal formation as they varied the dosage over three orders of magnitude (10^{15} - 10^{18} cm⁻²).

The parameter that seems to correlate well with the observed results is the ion mass of the projectile. In both FC and the present study, Ar⁺ ions were used; DC used the lighter Ne⁺ ions. This observation can be correlated with recent theories on surface compositional changes of binary solids during ion bombardment.^{15,16}

Sulfur vacancies were produced in the study by Davis and Carver because of the relatively light mass of the Ne⁺ ion, which preferentially removed the lighter sulfur atoms over the molybdenum. Ion bombardment with the heavier Ar⁺ ion, however, results in a higher-energy accommodation (energy transfer) by the lattice atoms.¹⁷ For a binary compound in which one element is considerably more volatile than the other, this higher-energy accommodation can cause high local temperatures to be generated during the collision cascade, inducing thermal sputtering of the element with the higher vapor pressure.¹⁵ For Ar⁺ on MoS₂, Mo and S having considerably different volatilities, gaseous sulfur is probably generated locally and leaves behind Mo-rich areas that nucleate into islands. It is coincidental, however, that the element with the higher volatility, sulfur, also has the lighter mass, and thus is sputtered preferentially in all studies mentioned, albeit by different mechanisms.

Preliminary experiments involving Auger peak shape analysis before and after ion bombardment have confirmed the results of Davis and Carver. IB with 1 keV Ne⁺ did not appear to change the shape of the Mo Auger peak as was observed by Feng and Chen for 300 eV Ar⁺. Interestingly, higher-energy IB (~ 5 keV Ne⁺) did cause the Mo peak shape to appear similar to that for Mo metal. Therefore, it appears that there is an energy threshold for Mo metal formation, in addition to the ion mass effect mentioned above.

There are issues in the study of noble-gas-ion bombardment of MoS₂ basal plane surfaces that must still be resolved. The apparent nondegradation of the surface region (other than Mo island production) must be explored. Also, more results are needed to confirm the effect of ion mass, energy, and dosage so that a cohesive theory on the mechanism of ion bombardment of MoS₂ surfaces can be developed and, in turn, one of ion bombardment of solids in general. These studies are continuing and will be published at a later time.

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