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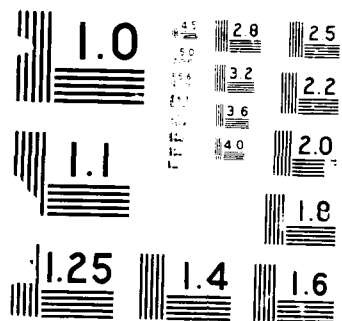
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CHEMISTRY AND PHYSICS LAB M R HILTON ET AL. 11 SEP 89
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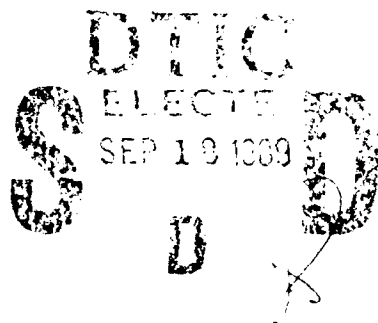


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Structural Studies of Sputter-Deposited MoS₂ Solid Lubricant Films

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11 September 1989

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89 9 18 047

This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, Report No. F04701-88-C-0089 with the Space Systems Group, P.O. Box 92960, Los Angeles, CA 90009-2960. It was reviewed and approved by The Aerospace Corporation by S. Feuerstein, Director, Chemistry and Physics Laboratory. Lt Carl Maes, SSD/CWDE, was the project officer.

This report has been reviewed by the Public Affairs Office (PAO) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION Unclassified			1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY			3. DISTRIBUTION AVAILABILITY OF REPORT	
2b. DECLASSIFICATION AND DOWNGRADING SCHEDULE			Approved for public release; distribution unlimited.	
4a. AUTHORING ORGANIZATION REPORT NUMBER(S) CR-0089-40641-2			5. MONITORING ORGANIZATION REPORT NUMBER(S) SP-IR-89-16	
6a. NAME OF REPORTING ORGANIZATION The Aerospace Corporation Laboratory Operations		6b. OFFICE SYMBOL (If applicable)		7a. NAME OF MONITORING ORGANIZATION Space Systems Division
6c. ADDRESS (Include City, State, and ZIP Code) El Segundo, CA 90245		7b. ADDRESS (Include City, State, and ZIP Code) Los Angeles Air Force Base Los Angeles, CA 90009-2950		
8a. FUNDING SPONSORING AGENCY		8b. OFFICE SYMBOL (If applicable)		9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F04901-88-C-0089
9a. ADDRESS (Include City, State, and ZIP Code)			10. SOURCE OF FUNDING NUMBERS	
			PROGRAM ELEMENT NO	PROJECT NO
			TASK NO	WORK UNIT ACCESSION NO
11. TITLE (Include Security Classification) Structural Studies of Sputter-Deposited MoS ₂ Solid Lubricant Films				
12. PERSONAL AUTHOR(S) Hilton, Michael R.; Fleischauer, Paul D.				
13a. TYPE OF REPORT		13b. TIME COVERED FROM TO		14. DATE OF REPORT (Year, Month, Day) 1989 September 11
				15. PAGE COUNT 24
16. SUPPLEMENTARY NOTATION				
17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	Lubricant film(s) Solid lubricant(s)	
			Molybdenum disulfide Tribology	
			MoS ₂	
19. ABSTRACT (Continue on reverse if necessary and identify by block number)				
The relationship between the morphologies and crystal structures of sputter-deposited MoS₂ films and their lubricating capabilities is discussed. In particular, aspects of plastic deformation processes are presented. Scanning electron microscopy (top surface and cross sectional), transmission electron microscopy (lattice imaging and dark field), and x-ray diffraction techniques were used to characterize film structure. The as-deposited morphology, which can be described in terms of zone models, was found to influence the initial nature of the wear debris and the load-bearing capabilities of the films. In many cases a highly deformed region confined to the surface of the films was found. Applied stress was found to reorient crystallites and to induce crystallization, with the degree of both processes being related to the initial structure of the film. Criteria are presented for selecting the film type and preparation conditions to fit various applications.				
20. DISTRIBUTION AVAILABILITY OF ABSTRACT			21. ABSTRACT SECURITY CLASSIFICATION	
☒ UNCLASSIFIED ☐ SAME AS RPT ☐ DTIC USERS			Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL			22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL

PREFACE

The authors thank B. D. McConnell and L. L. Fahrenbacher for helpful discussions, and R. Bauer (The Aerospace Corporation), B. C. Stupp (Hohman Plating, Inc.), and E. W. Roberts (National Centre of Tribology, U.K.) for providing the RF, DC, and RFM films examined in this investigation. The assistance of P. Adams with the XRD experiments is also acknowledged.



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I. INTRODUCTION

Molybdenum disulfide (MoS_2) is a useful solid lubricant because it deforms plastically more readily than the solid surfaces between which it is placed. On the macroscopic scale, the low shear strength of MoS_2 reduces the friction between sliding surfaces. On the atomic scale, the low shear strength (low friction) of MoS_2 is explained by its anisotropic crystal structure: the material is comprised of hexagonally packed planes consisting of a layer of Mo bounded on each side by a layer of S. All effective strong bonding is within the resulting "sandwich" planes, not between adjacent sandwiches. MoS_2 is strong in two dimensions and weak in the third, making the material a two-dimensional mechanical analog to one-dimensional linear polymers. The low-shear-strength basal planes provide an atomic mechanism for single-crystal plastic deformation; this mechanism plays a role similar to that of dislocations in close-packed metals. However, on the microstructural scale, which relates atomistic and macroscopic phenomena, the mechanisms of MoS_2 plastic deformation have not been fully explained. For example, MoS_2 lubricants are polycrystalline, and while the basal-plane slip mechanism explains deformation within a single crystal, the nature of the intercrystalline slip and its contribution to overall deformation are not understood at this time.

Sputtering provides a method of applying MoS_2 as a lubricant in thin-film form; it avoids the use of the organic binders used in powder applications, which can outgas in the vacuum of space. Studies have been reported that attempt to relate MoS_2 sputter-deposition conditions to its mechanical properties, such as friction and wear, and oxidation resistance.¹⁻⁵ An increasing emphasis has been placed on elucidating the physical properties of films, such as composition, crystallinity, crystal orientation, and adhesion, in order to explain the effects of deposition conditions on film performance.⁶⁻²² These microstructural studies have not generally incorporated zone-model nomenclature in their reports.²³ Such zone models provide a conceptual framework for relating growth conditions to the physical properties of films.

The zone models use the dominant diffusion mechanism operating during growth as a classification criterion. The zone 1 structure is the result of a low (or absent) adatom mobility that is insufficient to overcome the effect of the shadowing favored at low T/T_m (deposition and melting temperatures, respectively) and high gas pressures. (In general, increasing gas pressure enhances the shadowing of the impinging flux, as well as decreasing the kinetic energy of ion bombardment in plasma deposition processes. Gas pressure was added by Thornton²⁴⁻²⁵ to the original model of film formation via evaporation developed by Movchan and Demchisin²⁶ as an additional measure, along with homologous temperature, of the degree to which adatom mobility is affected by deposition conditions.) The morphology of zone 1 films consists of large dome-capped grains that have poorly defined fibrous interiors. The films can be amorphous or crystalline, they often contain voids, and they generally have poor mechanical properties. The zone 2 structure results when surface diffusion dominates; this structure consists of well-defined columnar grains that have faceted or flat tops. The zone 3 structure occurs when lattice diffusion dominates. Grain growth or recrystallization can occur, promoting large columnar grains or equiaxed grains, respectively. The mechanical properties of zone 2 and zone 3 structures are better than those of zone 1 structures. Thornton also defined a zone T (transition) morphology, favored by lower gas pressures and a higher T/T_m , that was between the zone 1 and 2 structures. The zone T material has the zone 1 fibrous interior, but with flat tops; it is not porous and its mechanical properties can be good.

In this report we discuss the microstructures of sputter-deposited MoS_2 films, prepared in three different laboratories, in terms of zone-model nomenclature and the relationship between the as-deposited microstructure and the microstructural deformation that results from sliding contact. Sliding-wear test results are reported in detail elsewhere;²¹ from correlations between our observations and results reported in the literature, we show that sliding wear can induce crystallization, a heretofore unreported result.

II. EXPERIMENTAL

The MoS₂ films on 440C bearing steel were prepared by three sputtering techniques in different laboratories using (1) an rf target,²¹ (2) a dc target,^{3,5} and (3) an rf magnetron target.¹⁷⁻¹⁸ For convenience, the films will be referred to as RF, DC, and RFM, respectively. Typical deposition temperatures, pressures, and growth rates were as follows: RF: 70-220°C, 20 mT, 245-345 Å/min; DC: 135-190°C, 23 mT, 600 Å/min; RFM: 25-70°C, 20 mT, 400-600 Å/min. Some of the DC films contained codeposited nickel.^{3,5} It is worth emphasizing that the RF and DC films were formed in contact with the plasma during growth, while the RFM films were deposited with the plasma magnetically confined away from the surface of the film. Surface adatom mobility, which is influenced by substrate temperature (which in turn is affected by secondary-electron bombardment), should be lower in the RFM case. The substrates were sputter-precleaned in the DC and RFM experiments, which was not done in the RF experiments.

The sliding-wear deformation was produced by a thrust washer apparatus under conditions described previously.²¹ Briefly, the machine consists of a disk that slides against a coated flat under low loads (3.18 kg dead-weight load) at a mean sliding velocity of 33 mm/s. The apparent contact area between the rider and the stationary member was approximately 45.2 mm². In some tests films were run until they failed (arbitrarily defined as that point where the reaction torque of the stationary member exceeded 0.07 Nm); other tests were terminated after a fixed number of revolutions, then characterization measurements were made.

Film morphology was investigated by means of scanning electron microscopy (SEM). Both top-surface and cross-sectional morphologies (generally viewed at a 30° tilt) were investigated. Film cross sections on the tough steel substrate were prepared by impacting the sample with a diamond brale indenter in a Rockwell C hardness tester at a static load of 150 kg. The radial compressive load generated at the indentation rim caused the film to

sample, which reproducibly exposed a cross section.²⁷ Previous investigations of MoS₂ cross-sectional morphology have generally been limited to films on brittle substrates that could be fractured^{7,8,10,14,16,19} except for cross sections exposed adjacent to sliding-wear tracks on steel.^{9,13} Indentations were made in unworn and worn regions. Prior to SEM observation, the MoS₂ surfaces were coated with gold to improve the conductivity of the sample.

Film crystallinity and orientation were studied by means of x-ray diffraction (XRD) using diffractometer and film techniques. A Philips Electronics model APD-3720 vertical diffractometer equipped for normal x-rays using Cu-K α x rays (1.54 Å) was used. X rays, which resulted from the fluorescence of iron in the steel because of Cu-K α irradiation, were filtered out with a monochromator. The films were oriented so that the scattering vector $\bar{G}$ (the vector subtraction of the incident x-ray vector from the outgoing x-ray vector) was always normal to the surface. The area of irradiation was 0.9 $\times$ 1.0 cm. To analyze some of the samples, the resulting XRD pattern was photographed with a Read thin-film camera, for which the angle of incidence of the x rays with respect to the substrate surface ranges from 5 through 15° (this angle serves to emphasize near-surface species in the XRD pattern²⁰). The use of the Read technique provides two other advantages: (1) the scattering vector $\bar{G}$ was not restricted to being normal to the substrate, and (2) a smaller area of irradiation could be obtained (1 $\times$ 4 mm). Cr-K α radiation (2.29 Å) filtered through a vanadium filter was used.

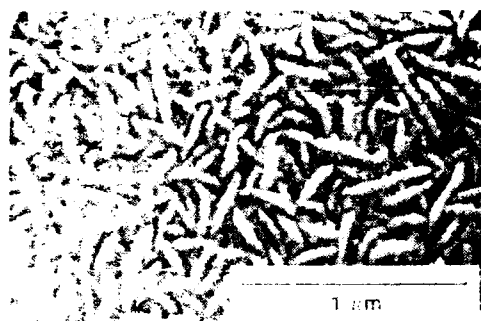
The nanostructure of the RF films was investigated by transmission electron microscopy (TEM) using lattice imaging and dark-field techniques along with electron diffraction. Specially prepared RF MoS₂ films deposited on amorphous carbon were analyzed. Deposition times ranged from 20 to 90 s, and substrate temperatures were fixed at 70 or 220°C.

III. RESULTS AND DISCUSSION

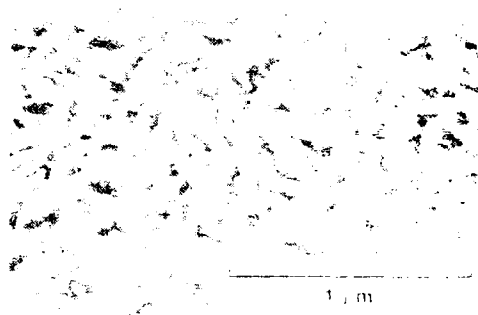
A. AS-DEPOSITED FILMS

SEM revealed that the RF and DC films have a similar morphology (see Figs. 1a and 1b) consisting of acicular islands, which have been observed before.^{3,5,8-22} Cross-sectional analysis revealed that these islands are the tops of columnar plates (see Figs. 1c and 1d). The plates generally have a microstructure that is denser and finer near the interface than at the surface, and this relative difference increases with deposition temperature, suggesting that increased adatom mobility facilitates a coarsening of the morphology. Columnar morphology is often observed in thin films, but the columns are generally equiaxed (in the plane parallel to the surface) and not platelike. However, most thin-film morphological investigations reported in the literature have been of metals or simple metal compounds (e.g., TiC, TiN, Al₂O₃), which generally have isotropic crystal structures, while MoS₂ has an anisotropic, planar structure.

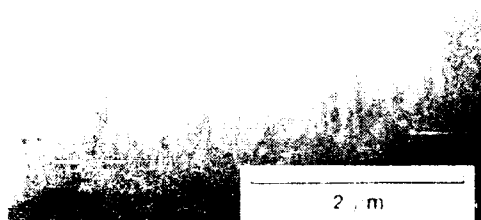
XRD indicates that the (001) basal planes are oriented normal to the substrate (and parallel to the columns), because (100) and (110) planes are observed parallel to the substrate (and normal to the columns). The perpendicular orientation of the basal surface forms because the edge surfaces are more reactive than the basal surfaces; under most deposition conditions²⁸ this favors edge nucleation at reactive sites on the substrate. With regard to crystallography, diffusion kinetics, and reactivity, it can be assumed that MoS₂ grows faster on the edge planes than on the basal plane, which would explain why platelike, as opposed to equiaxed, columns form. If an island nucleates with the (100) plane parallel to the surface, horizontal growth is fast in the [010] and [0 $\bar{1}$ 0] directions and slow in the [001] and [00 $\bar{1}$] basal directions, while vertical growth is fast in the [120] direction. Nucleation with a (110) plane parallel to the surface will yield fast horizontal growth in the [1 $\bar{1}$ 0] and [$\bar{1}$ 10] edge directions, slower growth in the [001] and [00 $\bar{1}$] basal



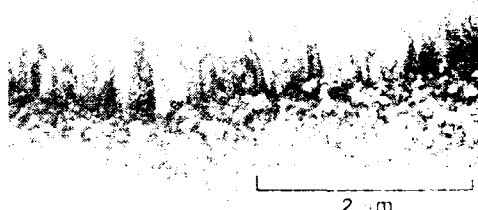
A



B



C



D

Fig. 1. SEM micrographs showing the morphology of some of the zone 2 films. (A) RF AT, top; (B) DC with Ni, top; (C) RF AT cross-section, 30° tilt; (D) DC with Ni, cross-section, 30° tilt.

directions, and fast vertical growth in the $[110]$ edge direction. The (100) parallel orientation is the dominant one observed.²¹

If one assumes that growth is slower in the $[001]$ directions, the basal planes should lie parallel to the major axis of the islands. Unfortunately, the XRD data cannot confirm this assumption, because XRD can detect only those orientations that are parallel to the surface. However, TEM lattice images of the (002) basal planes of RF MoS_2 on amorphous carbon clearly show that this hypothesis is the correct orientation (see Fig. 2). Bichel et al. have published a similar lattice image.¹⁴ A corresponding dark-field image (see Fig. 3) shows that the islands do not contain perfectly straight, single-crystal basal planes; some deviation is present. Further investigation and interpretation are in progress.

The RFM films examined possess a very different morphology (see Fig. 4a) consisting of fibrous, equiaxed columns that are more densely packed than the columnar-plate structure. The equiaxed morphology suggests that surface diffusion is inhibited, which would cause atoms to incorporate into the lattice at or near the point of impingement, thus inhibiting the formation of anisotropic plates. XRD revealed no peaks for the RFM material, indicating there is no long-range crystallographic order; this is consistent with the hypothesis that adatom mobility is limited. This morphology has been observed by other investigators.^{6,14-19,22}

The RFM morphology studied has a zone 1 (perhaps zone T) appearance, while the RF and DC morphologies are a variation of the zone 2 structure. Columnar plates are formed instead of equiaxed columns because of the anisotropic crystal structure and differing chemical reactivities of the various crystal facets of MoS_2 . The deposition conditions for the RF, DC, and RFM films were slightly different in pressure (RF/RFM: 20 mT; DC: 23 mT) and in measured temperature (RF: 70-220°C; DC: 135-190°C; RFM: 25-70°C). The generally lower deposition temperature of the RFM process would inhibit adatom mobility. We believe another important factor in the RFM process is that the plasma is magnetically confined away from the film surface. Thus the enhancement of adatom mobility from secondary-electron surface-



0 20 40 60 80 100 nm

Fig. 2. TEM lattice image of an RF AT film on carbon, showing that the early nucleation morphology consists of anisotropic islands. The (002) basal planes are oriented normal to the substrate and parallel to the major horizontal axis of the islands, indicating that rapid growth occurs along the edge planes.



0 20 40 60 80 100 nm

Fig. 3. TEM dark-field image, corresponding to Fig. 2, showing that the islands do not contain perfectly straight, single-crystal basal planes; some deviation is present.

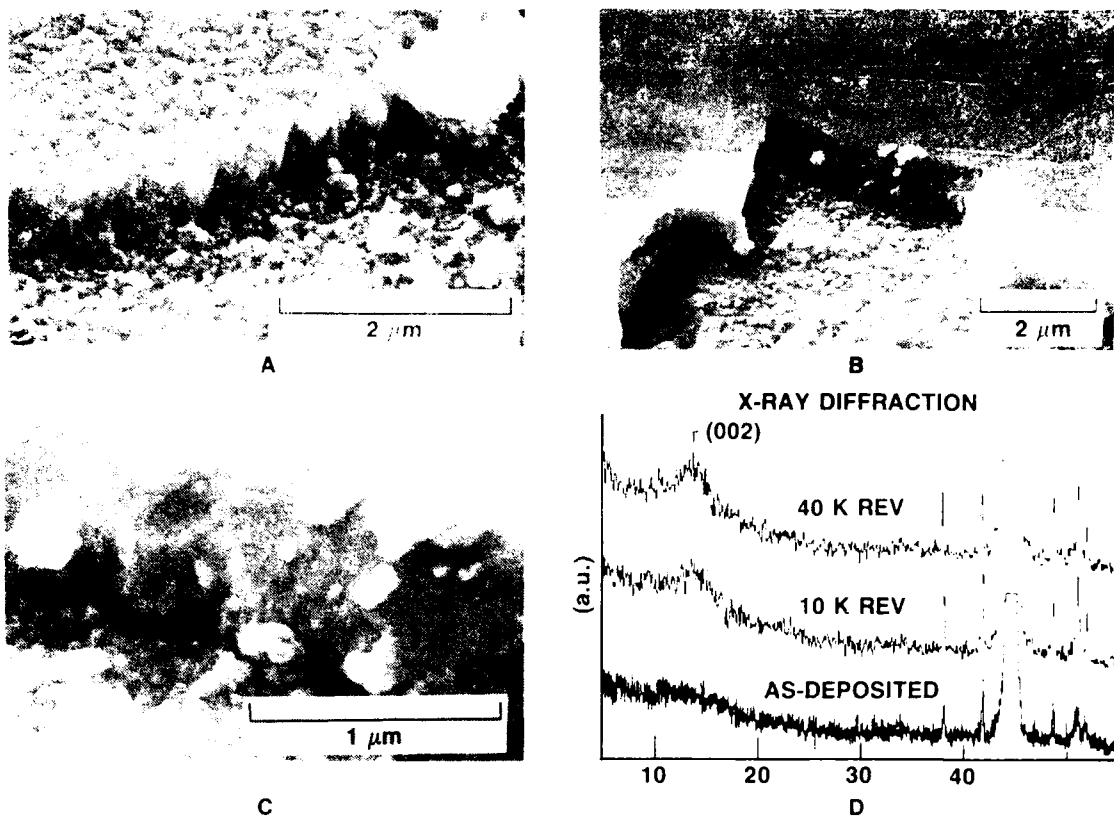


Fig. 4. SEM micrographs of the cross sections of an RFM film that has a zone 1 (zone T) structure in the as-deposited state (A). During sliding wear a smooth deformed region forms at the surface (B) with fibrous zone 1 (zone T) structure remaining intact underneath (C). (D) X-ray diffraction indicates that the as-deposited material has no long-range order, but a basal orientation parallel to the substrate in the surface region develops during sliding wear as a result of stress-induced crystallization.

recrystallization heating is much lower in the RFM process. Sparvins has shown that one can induce the zone 1 morphology in MoS_2 by lowering the substrate temperature.¹⁷

Chemical effects can also influence morphology. The zone models were developed in the context of experiments on single-element film formation, and when more than one element is present, chemical interactions can affect mobility and structure. Such interactions can arise from heats of formation²³ or the presence of impurities.²⁴⁻²⁵ Buck has clearly shown that the presence of water during deposition inhibits mobility.¹⁵ The addition of water vapor during deposition induced a zone 1 morphology, even at 100 °C. Roberts has been able to produce RFM films that have zone 2 morphologies, by operating at higher growth rates than the rate used to synthesize the RFM films reported here.¹⁷ He suggests that increasing the MoS_2 deposition flux while maintaining a fixed water-vapor flux decreases the relative immobilizing effect of the water. There is support for the idea that some alteration of the chemical influence of water occurs as a result of an increased MoS_2 flux, because in general changing the film-deposition rate alone, even over a wide range of rates, has not been found to change morphology.²⁴ Recent XPS studies in our laboratory indicate that the RF zone 2 material reported here is really an MoS_2 -like phase that contains some oxygen (5-15%) in a 2^- state, along with sulfur (i.e., O is substitutional with S and is not from MoO_3), which is evidence that water was present during deposition.

B. WORN FILMS

When sliding-wear tests are performed on the zone 2 films, a crystal reorientation occurs in which some of the basal planes are realigned parallel to the substrate, thus facilitating lubrication.²⁰⁻²¹ XRD shows that this reorientation occurs, on a significant scale, faster on films formed at higher temperatures (HT, approx. 220°C) than on those formed at ambient temperatures (AT, approx. 70°C). The wear life of the AT films is longer than that of the HT films.²¹ Both types of film show a similar dependence of wear life on thickness: below a critical thickness,

effect of lubrication does not occur, while above some optimum thickness, additional gains of wear life with increased thickness are small.²¹ Spalvins observed this critical thickness phenomenon, although he did not find any gains in wear life above the optimum thickness.¹⁰ In his wear test,⁶ wear debris was quickly removed from the wear track, while debris is generally retained in our test for long periods; this would explain the difference in our observed effect of larger thickness.

Spalvins proposed that additional material above the critical thickness was of little value, because the columnar plates fractured at this thickness, creating the loose debris that was quickly discarded from the wear track.¹⁰ The material remaining after fracture provided the effective solid lubricant, although the details of the crystal reorientation needed for lubrication were not reported. We have observed the fracture, detachment, and complete reorientation of the columnar plates (see Fig. 5a), which can form lubricating sheets of debris in our test (see Fig. 5b). However, the plates do not need to detach completely; rather, they can tilt partially (see Fig. 5c) or they can bend (see Fig. 5d), with both conditions forming highly deformed, smooth, lubricating regions at the surface of the plates. Buck has also observed this surface deformation when films are lightly loaded ("wiped").¹¹ We see variations in the deformation morphology within a given sample, which indicates that the contact load in our test varies across the sample. However, on average, large-scale deformation (i.e., plate detachment or the reorientation of the entire plate) occurs very quickly in the HT films relative to the AT films, which have a denser morphology. During wear the AT films can develop a deformed zone at the surface, with some tilting and bending of plates. In each sample the depth and degree of deformation vary with location. The DC films tend to wear in a manner similar to that of the RF AT films (see Fig. 6.)

The zone 1 (zone T) films also exhibit a smooth, deformed region at the surface, beneath which the fiber structure is still intact (see Figs. 4b and 4c). XRD reveals that a basal-plane orientation develops in

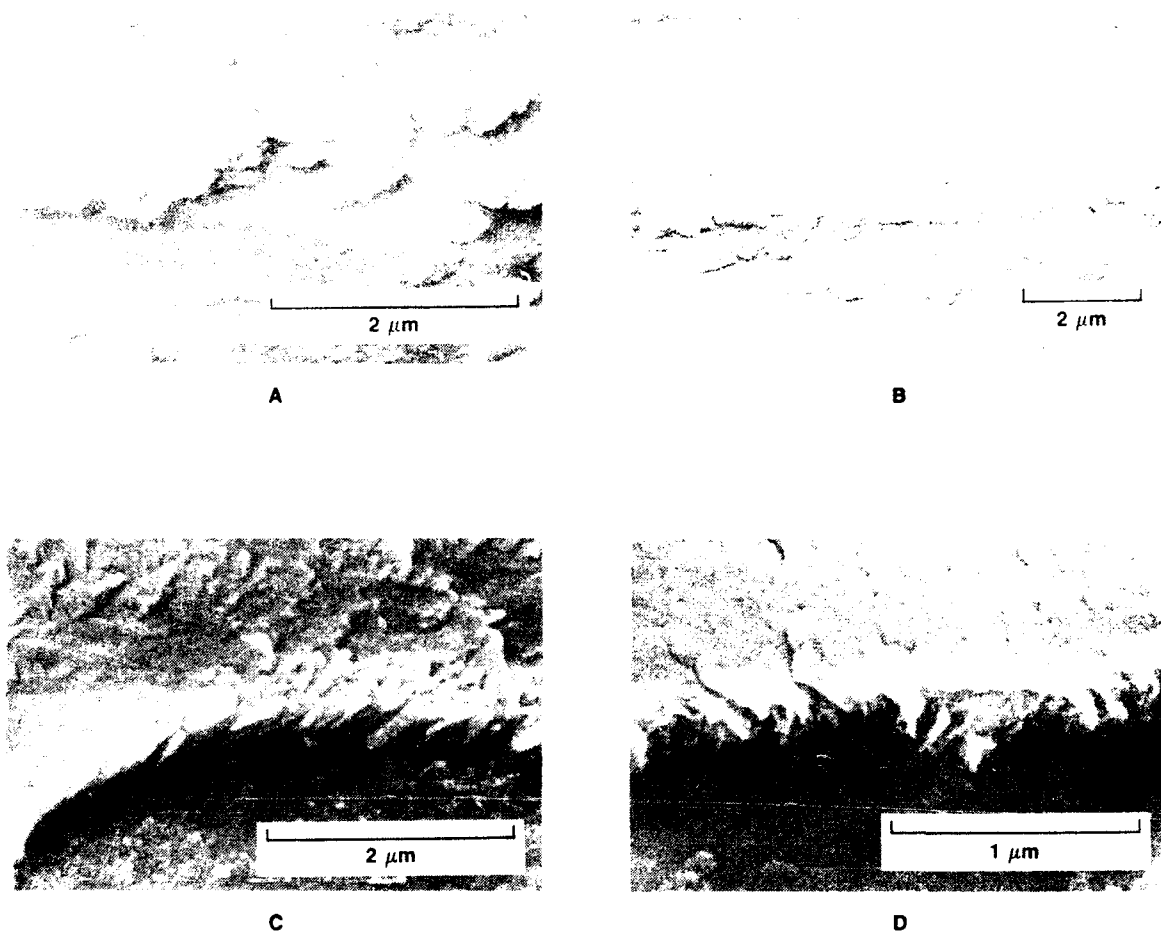


Fig. 5. SEM cross-sectional micrographs of RF HT films, showing various sliding-wear deformation morphologies. (A) Complete detachment and reorientation of columnar plates that form (B) lubricating sheets of debris in our test. (C) Partial tilting of plates. (D) Bending of plates.

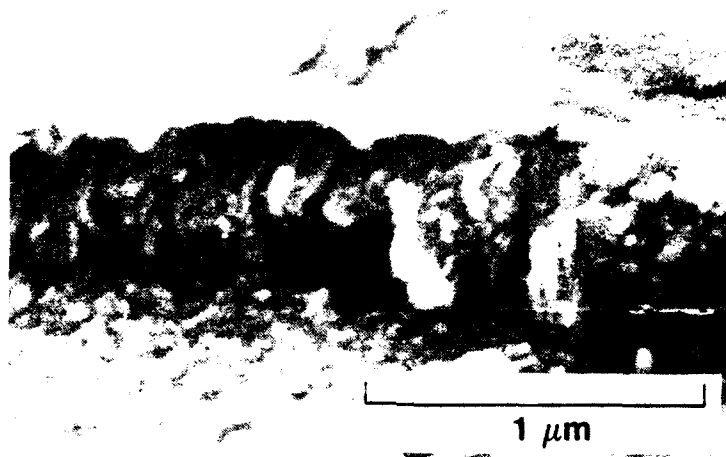


Fig. 6. SEM cross-sectional micrograph of a DC film (with Ni), showing the development of a highly deformed surface region during sliding wear. The DC films appeared to be similar to the RF AT films after sliding wear.

the wear track parallel to the substrate, suggesting that a stress-induced crystallization has occurred in the deformed layer (see Fig. 4d). This crystallization apparently provides sufficient lubrication to yield good wear life in vacuum tests.¹⁷ We suspect that these zone 1 (zone T) films synthesized by RFM at room temperature have some degree of short-range order that facilitates stress-induced crystallization. Buck has also observed a similar material, which he implied (by labeling it "Type II"; this nomenclature is explained elsewhere³⁰) had an as-deposited parallel substrate and basal-plane orientation.¹⁶ In contrast, Spalvins has shown that when films are deposited at cryogenic temperature, wear performance is poor.⁷ We believe that Spalvins' films were completely amorphous because of the lower deposition temperature, and that this complete disorder inhibited stress-induced crystallization. Buck has also observed an "amorphous" structure, which he distinguishes from his "Type II" material.¹⁶

IV. SUMMARY

In summary, when the lubrication ability of sputter-deposited MoS_2 film is assessed, both the as-deposited and deformed microstructures, which include crystalline orientation and morphology, must be considered. Some types of zone 1 (zone T) and zone 2 morphologies yield films of good lubricity; however, some variations are inferior. A morphologically dense film -- either a low-temperature zone 2 or a zone 1 (zone T) film possessing sufficient short-range order to allow stress-induced crystallization to occur -- is desirable. For large-load, long-wear-life applications, particularly in vacuum, the present study shows that the morphology should be dense enough to provide good load-bearing capability, preventing large-scale film reorientation early in sliding wear. This morphology is also desirable when the wear application quickly ejects debris, such as in a ball bearing. However, in a sliding application in which the debris is trapped, such as in a telescoping mechanism, a thick HT zone 2 film would be acceptable. If a reduced wear-life is allowable, the retardation of oxidation during spacecraft storage on earth would favor an HT zone 2 morphology, which is more resistant to such oxidation.^{12,21,30} Crystallographically, depositing a material whose basal orientation is parallel to the substrate in the as-deposited microstructure is still a desirable goal with regard to improved lubricity and greater resistance to oxidation, although the morphology of the material must be dense. Other factors that influence film performance are the presence of water during wear¹⁸ and the adhesion of the film to the substrate, with the latter property being strongly dependent upon interface composition (this issue is not discussed in the present work). Both topics are the subject of continuing research.

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