NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2628

BONDING OF MOLYBDENUM DISULFIDE TO VARIOUS MATERIALS TO FORM A SOLID LUBRICATING FILM

I - THE BONDING MECHANISM

By Douglas Godfrey and Edmond E. Bisson

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Cleveland, Ohio

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I - THE BONDING MECHANISM

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SUMMARY

The use of molybdenum disulfide MoS_2 as a solid film lubricant in applications where designs or temperatures preclude liquid lubricants is dependent upon successful bonding of the powder to the surface to be lubricated. An experimental investigation was conducted to determine the basic mechanism of bonding and to extend application of the bonding to a variety of materials. The results indicated that when MoS_2 was applied to a surface as a mixture of MoS_2 powder and some liquid vehicles, the liquid vehicle decomposes or polymerizes to a resin which binds the particles of MoS_2 together and to the surface to be lubricated. By use of resin-forming viscous liquids such as asphalt-base varnish, silicones, glycerine, ethylene glycol, polyglycol ether, and corn syrup, MoS_2 can be bonded to materials such as steel, aluminum, brass, stainless steel, and glass. The reduction of Fe_2O_3, formed by preheating steel in air, to Fe_3O_4 by one of the liquid vehicles (syrup) improves the frictional properties of the solid lubricating film.

Rubbing of MoS_2 whether dusted or built up on or bonded by a resin to a surface produced distinct preferred orientation.

INTRODUCTION

Molybdenum disulfide MoS_2 used as a solid-film lubricant has been shown to have high load-carrying capacity at high pressures (reference 1); to maintain low coefficients of friction over a wide range of sliding velocities (reference 2); and to maintain a low friction coefficient during its oxidation (which begins at a very low rate at 750° F) as long as an effective subfilm of MoS_2 remains (reference 3). Such desirable properties are extending the use of MoS_2, particularly where designs or temperatures preclude liquid lubricants, such as compressor blade-root lubrication (reference 4). The low coefficient of friction and minimization of surface damage are dependent upon the relatively low shear strength and high load-carrying capacity of MoS_2, but lubrication is sustained only as long as the material remains between the rubbing surfaces in effective amounts. The life of an effective film is dependent upon its resistance to being ploughed up and pushed out of the way. The resistance to ploughing and removal is dependent upon the toughness of the film and its tenacity for one or both of the sliding surfaces. With MoS_2, of fixed toughness, shear strength, and load-carrying capacity, an increase of the lubricating life may be possible by an increase in the tenacity of the bond to metals.

The adherence of pure dry MoS_2 powder to clean metals, as a result of placing the two in contact only, could hypothetically be dependent on: (1) the sum of forces of attraction and repulsion extending from ions of the respective crystals (these forces are negligible, from a practical standpoint, because of the relatively great average distances of separation); (2) the forces of stability of new intermediate crystals formed at the interface by reaction between MoS_2 and the metal; and (3) the lodging of particles of MoS_2 in valleys and other irregularities of the metallic surface. Greater adherence in all these cases would result from increased intimacy of contact by reduced particle size and increased purity of the powder and by improved cleanliness of the metal surface. Greater adherence in the second case would result from an increase in chemical activity by an increase in temperature. Causing greater adherence between MoS_2 powder and metals by control of particle size, purity of powder, cleanliness of metal surface, and temperature is difficult and virtually limited to the laboratory.

An experimental investigation was conducted to determine the mechanism(s) of bonding of MoS_2 and to extend application of the bonding of MoS_2 to a variety of materials. Studies were made of (1) the adherence of dry MoS_2 powder to steel and aluminum; (2) the physical and chemical nature of dusted, rubbed, and bonded MoS_2 films; (3) chemical reactions in bonding mechanism; and (4) application of MoS_2 to a variety of metals and to glass. Qualitative tests to determine relative adherence of MoS_2 to materials were conducted. Electron diffraction was employed to detect: (1) chemical composition of solid lubricating films; and (2) presence of preferred orientation of MoS_2.

MATERIALS

Specimens and Specimen Preparation

All metals used as specimens were first subjected to precleaning by scrubbing with surgical cotton in an acetone-benzene mixture (50-50) followed by 10 consecutive rinses in freshly distilled acetone and acetone vapors in a Soxhlet extractor. The metal surfaces so cleaned were shown to be grease free by the water-wet test. Glass was scrubbed in sulfuric acid - sodium dichromate cleaning solution, washed in tap and distilled water, and oven-dried. The surfaces of the metals were either: (1) as rolled, or (2) blasted by sand-water-air mixture. The following materials were used:

For flat specimens:

(1) Steels

  (a) SAE 1020, cold rolled

  (b) SAE 1085, spring

  (c) SAE 52100, chrome

  (d) 347, stainless steel

(2) Copper and copper alloys:

  (a) Copper (99.8 percent pure)

  (b) Brass (nominal composition, 65 percent Cu; 35 percent Zn; trace of Pb)

(3) Aluminum alloy (52SO)

(4) Glass, double-strength window

For lubricants:

(1) MoS_2 powder, 99.9 percent pure: Screen analysis: over 200 mesh, 1 percent (by weight); under 200 and over 400 mesh, 10 percent; under 400 mesh and over 22 microns, 30 percent; under 22 and over 11 microns, 27 percent; under 11 and over 5 microns, 15 percent; under 5 microns, 17 percent.

(2) Petroleum oil, MIL-0-6081A, grade 1010. Viscosity, 9.95 centistokes at 100° F.

For resin forming:

(1) Glycerine, chemically pure

(2) Ethylene glycol, chemically pure

(3) Polyglycol ether, chemically pure

(4) Asphalt-base varnish, commercial wire insulating type, GE457

(5) Silicone-base varnish, commercial wire insulating type, DC996

(6) Corn syrup, commercial brown

(7) Dextrose, chemically pure

Other materials:

(1) Flaky graphite, C

(2) Powdered iron, Fe

(3) Granular iron, Fe

(4) Iron wire, Fe

(5) Powdered ferroso-ferric oxide, Fe_3O_4

(6) Powdered ferric oxide, Fe_2O_3

(7) Powdered aluminum oxide, Al_2O_3

PROCEDURE

Experiments were conducted to detect the bond to surfaces of MoS_2 applied both dry and with a liquid vehicle.

MoS_2 Applied as a Dry Powder

A quantity of 2 grams of one of the powders was deposited on the metal specimen by (1) simple dusting, or (2) simple dusting followed by rubbing powder onto metal with 10 strokes (1 in. long) of a 35-pound stainless steel weight. The specimens were then inverted, supported at the corners, and subjected to a sharp blow by a 50-gram weight dropping 20 centimeters. The amount of powder adhering was revealed by microscopic examination and weighing before and after shock. Each of the powders was applied to the following specimens, which had been blasted and cleaned:

(1) Cold rolled steel at room temperature

(2) Cold rolled steel with oxide film at room temperature

(3) Cold rolled steel with oxide film at 300° C

(4) Aluminum alloy at room temperature.

MoS_2 Applied with Liquid Vehicle

The procedure for brushing on and curing to form a solid film is as follows:

(1) Clean material free of grease and dirt.

(2) Apply thin coating of one of following mixtures to specimen with soft brush:

  (a) MoS_2, 50 parts; silicone varnish, 40 parts; xylene, 10 parts.

  (b) MoS_2, 50 parts; asphalt-base varnish, 40 parts; xylene, 10 parts.

(3) Allow film to air-dry tack free. (Asphalt-base varnish will air-dry throughout in approximately 24 hours at room temperature.)

(4) Cure solid film by heating at a temperature and for time required to produce a hard film, for example, 3 hours at 150° C for silicone varnish film.

The procedure for formation of a solid film lubricant by preheating and brushing on, based on method as proposed in reference 5, is as follows:

1. Clean material to make grease free.
2. Preheat material. (For example, heat 0.050-in. steel flat stock 5 min at 300° C, or longer time at lower temperature, to obtain blue oxide film.)
3. Apply 50-50 mixture of MoS_2 powder and liquid vehicle to hot material with rubbing; then bake until dry.
4. Scrape off excess coating, leaving tenacious underlying film.

Other experiments were conducted to detect occurrence of chemical reaction between various combinations of MoS_2 powder, Fe powder, Fe granules, Fe wire, steel flat stock, Fe_2O_3 powder, and corn syrup. Each material was intimately mixed with the other material and the mixture heated in air in a crucible 10 minutes at 300° C. After cooling, the contents were examined chemically and physically.

The formation of FeS was determined by treating the product with 10-percent solution of hydrochloric acid and noting the presence and relative concentration of H_2S gas.

Examination by Electron Diffraction of MoS_2 Powder

Dusted on, Rubbed on, and Bonded to Steel

An analysis of the electron diffraction patterns taken of surfaces treated with MoS_2 revealed interplanar spacings, chemical composition, and existence of preferred orientation of the exposed materials. Patterns were obtained from eight surfaces exposed by successive scraping away, with the edge of a glass microscope slide, of a MoS_2 solid film formed by the method of reference 5.

The tenacity of any bonded powder to a flat specimen was qualitatively (but not quantitatively) determined by examination after subjecting the solid films to five scraping passes with a knife edge loaded to 15 pounds, and to ten rubbing passes with a $\frac{1}{2}$-inch steel ball loaded to 15 pounds. The exposure of base material by cracking, flaking, and chipping, the degree of burnishing, and the general completeness of the film was noted.

RESULTS AND DISCUSSION

Examination by Electron Diffraction of MoS_2

Dusted and Rubbed on Steel

With MoS_2 powder simply dusted onto clean steel, the laminae assume random orientation and vary in size as shown by the electron diffraction pattern of figure 1(a). If, however, the MoS_2 is subjected to rubbing of only three passes, the lamina become highly oriented as shown by the electron diffraction pattern in figure 1(b). The (0001) plane of the crystals is parallel to the plane of the metal surface. Further examination of the pattern of figure 1(b) indicates that the particles may have become smaller. Part of the lubricating properties of MoS_2 may be attributed to the ease of preferred orientation demonstrated by this experiment.

Adherence of Dry Powders to Metals

The results of experiments conducted to detect the adherence of MoS_2 powder to steel and aluminum specimens by measurement of the gain in weight are presented in table I. For comparison purposes, the adherence of graphite and Al_2O_3 was also determined. For all combinations of powder and metal applied at 20° C without rubbing, no significant differences in weight gain were evident, which indicates that, under these circumstances, MoS_2 shows no particular adherence to or attraction for steel. That is, approximately the same weight of MoS_2, graphite, and Al_2O_3 adhered to steel as well as to aluminum when applied by simple dusting at 20° C; these results suggest that dry MoS_2 powder has no unique ability to adhere to steel when the two are simply brought into contact. Of the powders applied to oxidized steel without rubbing at 20° C and at 300° C, however, only MoS_2 showed an increase in weight, the increase being more than two-fold. This evidence supports the hypothesis that increasing the temperature favors the formation of new crystals at the interface of MoS_2 and steel. The new crystals then contribute to the adherence of the film.

When the powders were rubbed on the metal surfaces, the amount of powder adhering after shock changed markedly. In particular, the tests of rubbing MoS_2 on steel at 20° C showed a six- to ten-fold increase over those without rubbing. The rubbing resulted in building up of several high spots of MoS_2. The results showed further that gain in weight caused by rubbing MoS_2 on unoxidized steel was twice as great as the gain caused by rubbing MoS_2 on aluminum. The data indicate that adherence of graphite was not appreciably improved by rubbing or by application at higher temperatures, whereas adherence of Al_2O_3 was improved with rubbing, particularly on oxidized steel at 300° C. Particle size and shape no doubt influenced the results. The Al_2O_3 was fine and powdery, the MoS_2 was of fine and varied-size particles, and the graphite was of uniformly large flakes. The assumption that small particles are more readily lodged in the microscopic irregularities of the surface may account for the lack of adherence of graphite with rubbing and the very great adherence of Al_2O_3 on oxidized steel at 300° C. The results suggest that the presence of an oxide film on steel reduced adherence of rubbed MoS_2 whether applied at 20° C or 300° C. These experiments provide evidence that the adherence of MoS_2 to steel can be improved by rubbing and to a lesser extent by application of heat without rubbing. Also, the presence of a preformed oxide film on steel may reduce rather than increase the adherence of dusted MoS_2 powder.

Chemical Action in Bonding Mechanism

The results of experiments conducted for the purpose of detecting chemical reactions due to heating of (1) principal materials and (2) mixtures of principal materials are presented in table II. The formation of FeS when steel flats were heated in the presence of MoS_2 powder was expected but not detected. However, when the particle size (of the iron) was successively reduced to: (a) fine wire; (b) granules; and (c) fine powders; the formation of FeS was readily detected in successively increasing amounts.

A method of bonding MoS_2 to steel, based on that of reference 5, utilized corn syrup. When corn syrup is heated alone, it decomposes to a loose crumbly carbonacious mass; the walls of the vessel are, however, coated with a thin, not readily detected tenacious resin. Thus, the bonding of MoS_2 to steel by the method based on reference 5 could be a result of the binding action of MoS_2 to the steel by the corn syrup resin. The chemical reaction between MoS_2 and Fe, Fe_2O_3, or Fe_3O_4 would be inhibited by the presence of a resin-forming fluid because of contamination of reactants.

Another important observation was the reduction of Fe_2O_3 to Fe_3O_4 by the syrup during heating. The presence, in the resultant solid film, of Fe_3O_4 rather than Fe_2O_3, which is normally formed when steel is heated ir air, should be advantageous because Fe_3O_4 produces a lower coefficient of friction than Fe_2O_3 (reference 2).

Other Resin-Forming Liquid Vehicles

Experiments were conducted to indicate further that a resin will serve to bond MoS_2 to a variety of materials. The term resin will herein be applied to complex mixtures of long-chain hydrocarbons which are often formed in decomposition and polymerization of organic compounds. Their molecular weight can be approximated, but the exact structure of the components is difficult to determine. The results are presented in table III, which shows that resin-forming liquids such as asphalt-base and silicone varnishes bonded MoS_2 to a variety of materials. The bonded film was formed by brushing a mixture of MoS_2 and the liquid onto the specimen and curing or polymerizing to a solid film by air-drying or baking or both. Further, other resin-forming viscous liquids such as glycerine, ethylene glycol, and polyglycol ether bonded MoS_2 to various materials. Glycerine formed an excellent solid lubricating film with MoS_2; this film was readily burnished, was tenacious, and was free of voluminous carbonacious product. Dextrose, the major constituent of corn syrup, served to bond MoS_2 to steel also. The varnish or resin from light petroleum oil did not produce a tenacious film.

Bonding of MoS_2 and Other Powders to Various Materials

Additional experiments were conducted in which powders other than MoS_2 were bonded to steel by resin from corn syrup for further evidence to support the resin theory. Powders of Al_2O_3, Fe_2O_3, Fe_3O_4, and graphite were all successfully bonded by the usual procedure. Al_2O_3 and Fe_2O_3 produced, as expected, solid films of relatively high friction. Fe_3O_4 and graphite produced solid lubricating films with lower friction than Al_2O_3 and Fe_3O_4 but not as low as MoS_2.

Other experiments were conducted to determine limitations of bonding. Attempts were made to bond MoS_2 to brass, copper, aluminum, spring steel, stainless steel, and glass by methods employing heating for the polymerization of the liquid to the resin. The results are presented in table III and show that MoS_2 can be bonded to many materials of different types. Exposure of the metal, due to chipping, breaking, or removal of film during the knife scrape or ball rub test, rated the test as "fail" whereas protection of the surface by a thin effective film or by burnishing rated the test as "pass." The results of the bonding experiments indicated that the method should be so chosen that a loose, crumbly oxide is not formed. For example, the oxide formed on copper during heating for 10 minutes at approximately 300° C in the presence of MoS_2 and corn syrup was loose and crumbly; this loose oxide may explain the poor adherence of the resin to the base metal.

The results also show that MoS_2 can be bonded fairly well to glass. The bonding mechanism is thus shown to be independent of metals, surface valleys, and chemical action; therefore, the resin must be the bonding agent. A resin formed on glass by syrup displayed tenacity comparable to the whole MoS_2 film. On all materials which form adherent oxides, the MoS_2 was burnished by rubbing, probably with orientation of the lamina; continued rubbing resulted in continued burnishing and the formation of a very "slick" surface.

The cumulative evidence indicates that the mechanism of bonding to surfaces of MoS_2, applied as a mixture of powder and liquid vehicle, is one of binding the particles of MoS_2 into a resin and onto the surface. The resin is formed by decomposition and polymerization during heating or air drying. Thus MoS_2 can be bonded by resin-forming viscous liquids such as glycerine, ethylene glycol, and asphalt- and silicone-base varnishes to a variety of materials such as steel, aluminum, brass, stainless steel, and glass.

Analysis of Solid Film by Electron Diffraction

A solid film of MoS_2 bonded to cold rolled steel by resin from corn syrup was subjected to a series of light scrapings by the edge of a glass microscope slide and electron diffraction patterns were obtained from the subsequently exposed surface. The purpose of the analysis was to determine the crystalline structure and chemical composition of a typical solid lubricating film from the surface to the base metal. The results are presented in table IV. Analysis of the patterns showed that only MoS_2 in randomly oriented state existed throughout the main body of the solid film. The specimens always picked up a static charge from the electron beam, and the patterns revealed excess background scattering, indicating the presence of an insulating material throughout. As the base metal was approached in the scraping procedure, the MoS_2 pattern was almost completely replaced by a pattern of Fe_3O_4 and strong lines of α-Fe; finally the clean surface gave the α-Fe pattern of the cold rolled steel. No lines were found to suggest the presence of FeS or any other new compound. The insulating material is the resin and is evidently present in the MoS_2 and the Fe_3O_4 film down to the metal. The presence of the resin throughout the components of the film supports the previous statements of its importance in the bonding mechanism.

The absence of Fe_2O_3 and the presence of Fe_3O_4 as an intermediate layer proves the reducing effect of syrup on the Fe_2O_3 film that is formed on the steel during the preheat (in air) step. The character of the patterns revealed the MoS_2 to be in its original flaky and randomly oriented state throughout. No orientation was induced as a result of its contact with the materials, the process of deposition, or the scraping; rubbing the solid lubricating film with a burnishing tool, however, produced preferred orientation of the MoS_2. Additional experiments indicated that preferred orientation existed in a built-up layer of MoS_2 only (no resin) produced by continuous rubbing of dry powder deposited on a clean surface. The electron diffraction patterns from these two specimens were the same as that shown in figure 1(b).

SUMMARY OF RESULTS

An investigation of the mechanism of bonding MoS_2 to steel and other materials produced the following results:

1. When MoS_2 was applied to a surface as a mixture of MoS_2 powder and some liquid vehicles, the liquid vehicle decomposed and polymerized to a resin which bound the particles of MoS_2 together and to the surface to be lubricated.

2. MoS_2 can be bonded by resin-forming viscous liquid vehicles, such as asphalt- and silicone-base varnishes, glycerine, ethylene glycol, polyglycol ether, and corn syrup to a variety of materials, such as steel, aluminum, brass, stainless steel, and glass.

3. The reduction of Fe_2O_3, formed by preheating steel in air, to Fe_3O_4 by one of the liquid vehicles (syrup) improves the frictional properties of the solid lubricating film.

4. Rubbing of MoS_2 whether dusted, built-up, or bonded by a resin to a surface produced distinct preferred orientation.

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TABLE I - ADHERENCE OF MoS_2, GRAPHITE, AND Al_2O_3 POWDERS TO COLD ROLLED STEEL[a] AND ALUMINUM[a]

Combination of metal and powder		Temperature of application (°C)	Amount of powder adhering after physical shock (mg)[b]			
			Dusting without rubbing		Dusting followed by rubbing	
				av.		av.
Steel	MoS_2	20	0.5, 0.4, 0.5	0.5	5.2, 5.7, 4.5	5.1
	Graphite	20	1.0, .4, 1.0	.8	.5, .7, .8	.7
	Al_2O_3	20	.3, .6, 1.3	.8	2.0, 2.9, 2.0	2.3
Steel with Fe_2O_3 film	MoS_2	20	0.4, 0.5, 0.3	0.4	2.5, 2.4, 3.0	2.6
	Graphite	20	.5, .6, .8	.6	.5, 1.2, .9	.9
	Al_2O_3	20	.3, .8, .7	.6	1.0, 1.4, 1.3	1.2
Steel with Fe_2O_3 film	MoS_2	300	1.4, 1.2, 1.5	1.4	3.0, 2.6, 2.5	2.7
	Graphite	300	.3, .3, .5	.4	1.2, 1.2, .7	1.0
	Al_2O_3	300	.5, .8, .4	.6	96.0,87.0 101.0	95.0
Aluminum	MoS_2	20	0.7, 0.4, 0.4	0.5	2.0, 2.4, 2.0	2.1
	Graphite	20	.2, .1, .2	.2		
	Al_2O_3	20	.4, .3, .7	.5		

[a] All metals blasted and cleaned prior to experiments.

[b] Accuracy of weighing, ±0.1 mg.

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TABLE II - PHYSICAL AND CHEMICAL CHANGES RESULTING FROM HEATING BONDING MATERIALS TO 300-350° C FOR 20 MINUTES

Components	Chemical change	Physical change
MoS_2 only	Slight oxidation to MoO_3	None
MoS_2 and steel flats	Oxidation of MoS_2 and steel; no FeS	None
MoS_2 and Fe wire	Slight oxidation of MoS_2 to MoO_3; slight oxidation of Fe to FeO and Fe_3O_4; formation of FeS detected	None
MoS_2 and Fe granules	Same as MoS_2 and Fe wire except FeS readily detected	None
MoS_2 and Fe powder	Same as MoS and Fe wire except considerable FeS formed	Formed lumps
MoS_2 and Fe_2O_3	Slight oxidation of MoS_2 to MoO_3	None
Syrup alone	Charred to carbon and resin	Lumpy crumbly mass with underlying resin
Syrup and Fe_2O_3	Syrup charred to carbon and formed resins; partial reduction of Fe_2O_3 to Fe_3O_4	Lumpy voluminous mass
Syrup and fine powdered Fe	Syrup charred to carbon and formed resins	Lumpy voluminous mass
MoS_2 and syrup	Syrup charred to carbon and formed resins	Lumpy voluminous mass

TABLE III - OTHER RESIN-FORMING LIQUIDS FOR BONDING MoS_2

Liquid vehicle	Method	Bonded to	Knife scrape test	Ball rub test	Tenacity
Asphalt-base varnish	Brush on and cure	Glass	Pass - thin film remains	Pass - burnishes	Good
		Spring steel	Pass - thin film remains	Pass - burnishes	Good
		Stainless steel	Pass - thin film remains	Pass - burnishes readily	Good
		Stainless steel blasted	Pass - thin film remains	Pass - burnishes readily	Good
		Brass	Pass - thin film remains	Pass - burnishes	Good
		Aluminum	Pass - thin film remains	Pass - burnishes	Good
Silicone varnish	Brush on and cure	Glass	Pass - thin film remains	Pass - burnishes with difficulty	Fair
		Spring steel	Pass - thin film remains	Pass - burnishes with difficulty	Fair
		Stainless steel	Pass - thin film remains	Pass - burnishes with difficulty	Fair
		Stainless steel blasted	Pass - thin film remains	Pass - burnishes with difficulty	Fair
Glycerine	Preheat and brush on	Glass	Fail	Pass - burnishes	Fair
		Spring steel	Pass - burnishes	Pass - burnishes	Excellent
		Brass - blasted	Pass - burnishes	Pass - some flaking	Good
Ethylene glycol	Preheat and brush on	Spring steel	Pass - thin film remains	Pass - burnishes, some flaking	Good
Polyglycol ether	Preheat and brush on	Spring steel	Pass - thin film remains	Pass - burnishes, some flaking	Good
Corn syrup	Preheat and brush on	Spring steel	Pass - burnishes	Pass - burnishes readily	Excellent
Dextrose	Preheat and brush on	Spring steel	Pass - burnishes	Pass - burnishes	Good
Petroleum oil	Preheat and brush on	Spring steel	Fail - chips off	Fail - no burnish	Poor

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TABLE IV - ELECTRON DIFFRACTION DATA FROM MoS_2 SOLID FILM FORMED BY METHOD OF REFERENCE 5 AND COMPARATIVE DATA FROM A.S.T.M. X-RAY STANDARDS

As prepared		After 1st scraping		After 2nd scraping		After 3rd scraping		After 4th scraping		A.S.T.M. standard X-ray pattern for MoS_2		After 5th scraping		A.S.T.M. standard X-ray pattern for Fe_3O_4		After 6th scraping		After 7th scraping		A.S.T.M. standard X-ray pattern for α-Fe		A.S.T.M. standard X-ray pattern for FeS		A.S.T.M. standard X-ray pattern for Fe_2O_3	
Solid film MoS_2		Surface of solid film removed		Further removal of solid film		Further removal of solid film		Further removal of solid film				Base of film and metal exposed				Further exposure of base and metal		Solid film completely removed							
[a]d (A)	[b]I_r	d (A)	I_r	d (A)	I_r	d (A)	I_r	d (A)	I_r	d (A)	I/I_0	d (A)	I_r	d (A)	I/I_0	d (A)	I_r	d (A)	I_r	d (A)	I/I_0	d	I/I_0	d (A)	I/I_0
												2.94	fs	2.97	0.28							2.97	0.33		
																						2.88	.04		
2.73	vs	2.73	vs	2.74	vs	2.74	vs	2.74	fs	2.74	0.70														
										2.66	.30	2.57	vs			2.58	fs					2.65	.33	2.69	1.00
2.50	s	2.50	s	2.54	s	2.50	s	2.54	s	2.49	.50			2.53	1.00									2.51	.75
						2.47	w							2.42	.11										
2.27	vs	2.27	vs	2.27	vs			2.27	vs	2.27	1.00													2.20	.18
						2.11	vw	2.11	w			2.07	w	2.10	.32										
								2.00	fw	2.040	.70	2.01	vs			2.01	vs	2.01	vs	2.03	1.00	2.06	1.00		
1.805	s	1.823	s	1.823	s	1.823	s	1.823	s	1.820	1.00													1.84	.63
														1.71	.16							1.71	.33	1.69	.63
		1.609	vw							1.635	.30	1.591	fs	1.61	.64							1.61	.07	1.60	.13
1.559	fs	1.573	fs	1.559	fs	1.569	fs	1.569	fs	1.578	.70														
1.519	fs	1.525	fs			1.520	fs			1.530	.90														
				1.452	s			1.473	w	1.475	.20	1.470	vs	1.483	.80					1.45	.46	1.48	.04	1.485	.50
		1.355	fw	1.372	fw	1.360	fw			1.365	.2					1.405	fs	1.418	fs			1.442	.09	1.452	.50
1.347	fs	1.325	fs			1.323	fs			1.335	.7			1.326	.06							1.321	.13	1.351	.03
1.284	fs	1.287	fs			1.284	fs			1.295	.7													1.308	.18
1.237	fs	1.248	fs			1.237	fs			1.251	.7			1.279	.20									1.259	.13
1.169	w	1.188	w			1.187	w			1.222	.2			1.210	.05							1.299	.05	1.230	.03
										1.195	.5	1.156	s	1.121	.10	1.159	s	1.156	s	1.16	.54	1.179	.01	1.190	.08
1.084	w	1.094	fw			1.092	fw			1.100	.7			1.092	.32							1.105	.13	1.163	.05
										1.034	.8			1.049	.10			1.020	w			1.050	.07	1.140	.13
		1.011	w			1.019	w			1.021	.5					1.004	fw	1.004	fw	1.005	.24			1.056	.08
.990	fs	.995	fw			.987	fw			1.002	.7			.970	.16							.995	.01		
						.947	w			.968	.3			.966	.08									.962	.10
										.953	.7			.940	.06									.954	.05
										.912	.3														
										.901	.2													.900	.03
										.894	.7			.880	.10	.893	w	.888	w	.910	.18			.881	.05
														.859	.20					.823	.16			.843	.05

[a]d interplanar distance in Angstrom units (A)

[b]I_r estimated relative intensity; vs, very strong; s, strong; fs, fairly strong; fw, fairly weak; w, weak; vw, very weak

[c]I/I_0 A.S.T.M. standard pattern intensity ratio

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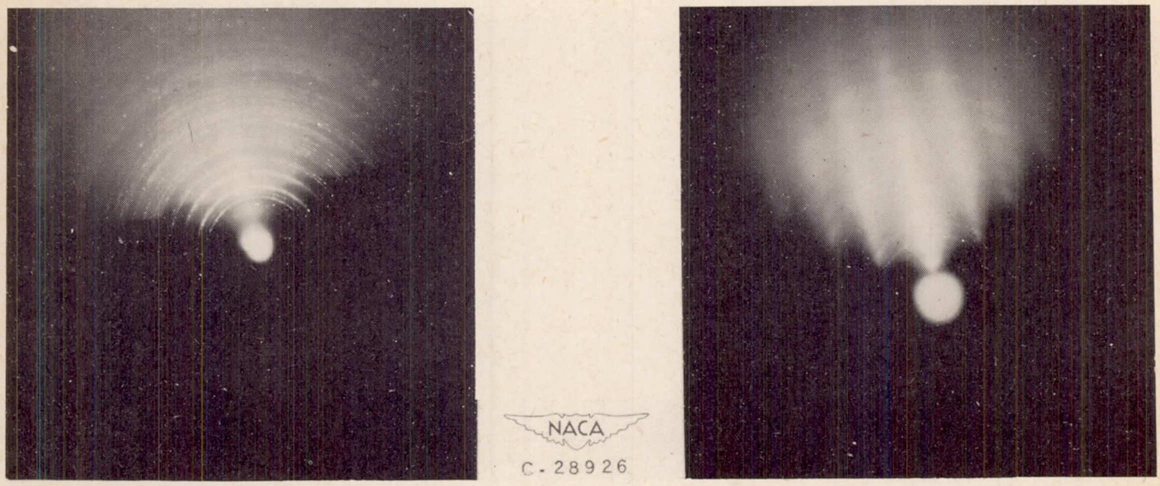

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(a) Dusted.

(b) Rubbed.

Figure 1. - Electron diffraction pattern of MoS_2 dry powder on steel showing preferred orientation of (0001) plane with rubbing.