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DEVELOPMENT OF POLYIMIDE BONDED SOLID FILM LUBRICANTS

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DEVELOPMENT OF POLYIMIDE BONDED
SOLID FILM LUBRICANTS

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ABSTRACT

The results of work conducted to develop heat and radiation resistant organically bonded solid film lubricants are described. These solid film lubricants are composed of an organic resin binder (polyimide and amide-imide resins) and several transitional element compounds such as the selenides of tungsten, molybdenum, niobium and tantalum. The approach, the data and the procedures necessary to mix, apply and cure these films are outlined. Friction and wear properties of these films at elevated temperatures (550°F) under high loading (15,000 psi) in addition to those parameters affecting the aforementioned properties such as, resin type, filler and its concentration, film thickness, test surface, radiation, long term static bonding are also presented.

A polyamide-imide resin designated as AI-131 and loaded with 40 volume or 79 weight percent WSe_2 was found to give the best combination of friction and wear properties under the severe conditions of the test. This composition is unaffected by humidity, gamma radiation and does not bond significantly under conditions of long-time heat and pressure. Coefficients of friction between 0.13 - 0.27 have been maintained under

test conditions for 3700 sliding motion cycles. Procedures for applying this coating composition onto large metal plate sections using either the draw bar or spray coating technique are also described.

INTRODUCTION

There exists a need for a material which can be applied to mating metal substrates and act as a lubricant for these metal substrates. The lubricant is to be applied to all mating surfaces of a stack of five plates. These coated plates will be subjected to high bearing loads (15,000 psi). Conditions of operation involve infrequent intervals of small amplitude cyclic motion at plate to plate contact at elevated temperatures (550°F) for long periods of time.

Preliminary tests on doped polyimide and polyamide-imide polymers with MoS_2 indicated that these materials could be used as lubricants for this application. As a result a program was proposed and investigated concerning the applicability of these polymers containing various lubricants to the application at hand.

The objective of the program is to develop a polyimide (family) type lubricant containing a powdered lubricant filler (preferably tungsten diselenide, WSe_2) which can be applied to a metal substrate and then cured to meet the following requirements:

1. Capability of being applied to a 5 ft by 5 ft plate such that a uniform thickness of approximately 1 mil is obtained which is adherent to the substrate.

2. Ability to withstand a minimum of 2000 motion cycles with 4000 cycles as objective at temperatures of 550-650°F under 15,000 psi loadings. A motion cycle is defined as 160 mils total travel from 0 to +40 back through 0 to -40 and returning through zero at a rate of 0.20 in./min.
3. Compatibility with Inconel.
4. Ability to withstand a radiation field of 5×10^8 ergs/gm.
5. Lubricating properties must not be affected by humidity.
6. Mating lubricated surfaces must not bond together if held motionless for long periods of time (90 days).

This program was divided into the following four phases of study.

- Phase I: Materials and Filler Studies - includes the preparation of the polyimide resins and the dispersion studies with various lubricants.
- Phase II: Preparation of Samples - includes the preparation of the resin-filler blends - coating and curing of samples.
- Phase III: Testing - including friction tests - humidity tests and static load testing - irradiation - compatibility with Inconel.
- Phase IV: Coating of large plate sections - including application and cure.

EXPERIMENTAL

Materials and Filler Studies

Three polymers of the imide and amide-imide family known to have high thermal stability have been chosen for this study. These include I-7, I-8 and AI-131. The lubricants (fillers) studied were MoS_2 , MoSe_2 , WSe_2 , NbSe_2 , TaSe_2 . Pertinent information on the resins and fillers is listed in Table I. The resin-filler blends were examined for dispersion, filler type, particle size, concentration (weight and volume) and surface smoothness of a cast film doped with the filler.

Preparation of Samples

The results of Phase I will dictate the proper filler to use for further testing. Initially MoS_2 ($< 5 \mu$ particle size) was used and excellent results were obtained. Because of compatibility problems with the alloys in the reactor MoS_2 could not be used and some other filler, preferably WSe_2 , would have to be used. The filler finally decided upon based on the results of Phase I was WSe_2 (1.1μ , $< 0.5\%$ free W) and was obtained from the Cerac Co., Menomonee Falls, Wisc. This filler was added to the various resins described in Table I at filler loadings of 10-20-40 volume percent. (See Appendix I). The resin-filler blend was thoroughly mixed using a Brookfield Rotating Mixer to ensure complete dispersion of the filler particles.

TABLE I
Identification of Resins and Fillers

<u>Resin</u>	<u>Composition</u> ¹	<u>Solids %</u>	<u>Gardner Viscosity</u>
I-7	BTDA + DAPE	8	Z-3
I-8	BTDA + MPD	18	Z-3 ⁺
AI-131	BTDA + MPD + DAB	25	Z-3 ⁺

1 - Recrystallized materials

BTDA = 3,3', 4, 4' - Benzophenone tetracarboxylic dianhydride

MPD = Meta - phenylenediamine

DAPE = 4, 4' - Diaminophenylether

DAB = 3, 4' - Diaminobenzanilide

<u>Filler</u>	<u>Identification</u>	<u>Particle Size</u>	<u>Supplier</u>
MoS ₂ *	Molybdenum disulfide	< 5μ	Climax Molybdenum Co.
MoSe ₂	Molybdenum diselenide	200 mesh (≈60μ)	Westinghouse
W Se ₂	Tungsten diselenide	200 mesh (≈60μ)	Westinghouse
NbSe ₂	Niobium diselenide	200 mesh (≈60μ)	Westinghouse
TaSe ₂	Tantalum diselenide	200 mesh (≈60μ)	Westinghouse
MoSe ₂	Molybdenum diselenide	2-10 μ	Cerac, Inc.
WSe ₂	Tungsten diselenide	5μ	Cerac, greater than 0.5% free W
NbSe ₂	Niobium diselenide	5μ	Cerac
TaSe ₂	Tantalum diselenide	5μ	Cerac
W Se ₂	Tungsten diselenide	5μ	Cerac, less than 0.5% free W

* Used as a reference standard

The sample plates illustrated in Figure 1 were then coated with the resin-filler blend using a Gardner Coating Knife with an adjustable gap setting to control the thickness of the wet film. The plates were cured in a stepwise fashion using the following cure schedule: 20 min 100°C - 20 min 150 - 20 min 200 - 20 min 225 - 20 min 250 - 20 min 275 - 5 min 300°C. The dry film thickness ranged between 1 and 2.5 mils.

Testing

The coated plates were friction tested at 500°F under a load of 15,000 psi. The fixtures used for the friction tests were modified to permit installation in a Baldwin Model FGT testing machine.

Strain gages were added to facilitate alignment and thus reduce bending in the fixture. Four axial gages were mounted equally spaced around the lower connecting rod, near the base (Fig. 2). Strain differences noted during setup indicate misalignment which can be minimized by shimming and shifting connecting rod attachments.

The friction force, "seen" by the testing machine alternately as a tensile and a compressive load, is indicated by the machine load dial and also recorded on a strip chart recorder. The normal force is sensed by the cylindrical compression load cell shown in Fig. 3. The full bridge strain gage output is recorded on a second strip chart recorder.

The testing machine is cycled on a displacement basis. Relative displacement of the mating surfaces is measured by two dial gages with extensions to the beams shown in Fig. 3.

The furnace and controller maintain the test temperature at 550°F ± 10°F.

Figure 4 illustrates the friction test schematically.

The effects of moisture on the friction properties of lubricant films were determined by exposing two compositions (I-7 and AI-131 + 20 vol % WSe₂) coated on the metal plates to 100% R. H. at 150°F for 30 days and then testing within 24 hours after removal from the humidity exposure.

The effects of radiation on the friction properties were determined by exposing the coated plates (AI-131 + 40 vol % WSe₂) to a Cobalt 60 gamma radiation source and testing the plates after exposure. Plates were irradiated to a dose of 5-6 x 10⁸ ergs/gm.

Friction testing was also performed against Inconel to determine compatibility with this alloy.

Friction tests were performed with film to film and film to bare metal interfaces. The substrates for the coated surfaces were grit blasted with 220 mesh Al₂O₃ and the bare surfaces were machined to 32-63 microinches rms. In some cases dry WSe₂ was rubbed on the mating surfaces prior to testing.

The metal plates were carbon steel and conformed to specification ASTM-GA-515.

To determine if the coated plates would bond together samples coated with AI-131 + 40 vol % WSe₂ were placed in face-to-face contact, loaded to 10-15,000 psi in a jig assembly and placed in an oven at 550°F

for 90 days. The tensile machine was used to get a relative measure of the force required to pull the samples apart after the 90 day exposure period. Samples were pulled apart while hot and under a load of 15,000 psi.

Coating of Large Plate Sections

Two carbon steel plates (12" x 15") grit blasted with 220 mesh Al_2O_3 were coated with the preferred materials (AI-131 + 40 vol % WSe_2) to determine the best coating technique (Spray or draw bar). Two coats using the draw bar technique were applied to one of the plates giving a total dry film thickness of 2.1 mils (1st coat - 1.2 mils). The second plate was spray coated using a Devilbiss spray gun giving a total dry film thickness of 1.4 mils (1st coat 0.9 mils). Detailed information on the resin-filler blend properties and coating technique is reported in Table II. The plates were cured in the same way as the small samples (see Page 9).

TABLE II

Data on Coating Large Plate Sections

<u>Method</u>	<u>Resin-Filler Blend</u>	<u>Solids Content (Resin) %</u>	<u>Viscosity Zahn Cup No. 3</u>	<u>Gardner</u>
Draw Bar Technique	AI-131 + 40 vol % (79 wt %) WSe ₂	21.5	64 sec	T

Wet film thickness was 4 mils. Dry film thickness first coat 1.2 mils.

Two coats dry film thickness is 2.1 mils.

Spray Coating	AI-131 + 40 vol % (79 wt %) WSe ₂	21.5 18.3	64 35	T J-K
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Wet film laid down by spraying vertically and horizontally at a distance of 4"-6" from the substrate. Forty lbs and 25 lbs were the gun pressures for the 21.5% and 18.3% solids content respectively. Dry film thickness is 1.4 mils. The first coat was applied at the higher solids level and gave a dry film thickness of 0.9 mils.

RESULTS AND DISCUSSION

Materials and Filler Studies

The photomicrographs in Figures 5 to 8 illustrate very clearly the distribution of the lubricant in the resin solution, the effect of particle size and the overall appearance of the films doped with various lubricants. As mentioned in Table I, MoS_2 was used as a reference standard because of its excellent earlier performance. It should be remembered however that the density of MoS_2 (4.8 g/cc) is much less than any of the other fillers and its effect on the doped film will be seen later. The effects of particle size are apparent from Figures 5 and 6. The larger particle size fillers tend to form agglomerates in the film and do not disperse as well as the small size fillers. The small particle size WSe_2 and MoSe_2 disperse very well but some agglomeration is evident as shown in Figure 6. What might seem to influence the friction properties of the doped films is the particle-to-particle contact, i.e. the volume of resin between each particle should be held to a minimum. Clearly this is evident with MoSe_2 and WSe_2 and the opposite effect can be seen with NbSe_2 and TaSe_2 (Figures 5 and 6). Another factor which must be considered is surface roughness. This should be kept to a minimum if the surfaces are to slide smoothly over each other. Any great roughness or significant irregularity in the doped film surface could result in poor contact and tearing of the film.

This roughness would be a function of the particle size of the filler, the dispersion, density, and its tendency to form agglomerates. Surface roughness measurements made on 1.5 mil thick doped films show a range of roughness values that range from 4 microinches or less for glass, to 8 microinches for MoS_2 , to 45 - 150 for WSe_2 and from 40 - 65 for MoSe_2 . However, instances occurred where the values for W and MoSe_2 were interchanged and this depended upon the curing technique. In no instances were any of the doped films made with a surface roughness as low as that for MoS_2 . Figure 7 shows the kind and variation of this surface roughness with the type of filler on cured films. All of these factors influence the quality of the doped film and by careful control of these variables good films can be made. However, differences will exist depending on the filler used in the film. Figure 8 depicts the kind of quality (dispersion, etc.) that can be obtained in doped films if one exercises tight control over the variables mentioned above. However, it should be noted that in no instance were doped films obtained as good as those containing MoS_2 .

As far as incorporating the fillers on a weight or volume basis, it should be noted that the more dense the filler the less amount of it will be used on a weight basis. If one compensates for this density difference all of the fillers will occupy an equal volume in the resin-filler mixture. In addition, particle-to-particle contact will be achieved more readily on a volume basis. Experiments made along these lines indeed point out the difference between the weight and volume effect and support the fact that the fillers should be incorporated into the resin solution on a volume basis.

To review, those factors giving rise to good doped films include the following:

1. The smaller the particle size the better the dispersion, film properties and less the tendency to agglomerate.
2. The less dense the material the more unit weight can be incorporated into the resin solution. And, apparently, the smoother the surface.
3. The incorporation of the fillers on a constant volume basis more nearly produces the dispersion and other properties necessary to achieve good films like those obtained with MoS_2 doped films.

Based on the visual and microscopic examination of the fillers MoSe_2 and WSe_2 are the preferred choices as lubricants. The lubricant properties of these two fillers are roughly equivalent. Films have been made from resin solutions containing these fillers with fairly good reliability although shortcomings are evident since these films with these fillers did not approach the quality of films with MoS_2 . There are slight differences between the lubricants, namely a lower density and a smaller particle size for MoSe_2 compared to WSe_2 which offer a means of variation over WSe_2 . However, even with these small differences WSe_2 (<0.5% free W) will be used initially in this study.

Preparation of Samples

The mixing procedure described in the experimental section (page 7) appears to be adequate to ensure proper dispersion of the filler in the resin solution. Hand mixing is not recommended if the resin has been left to stand for more than 5 hours. A two phase system will occur if the resin-filler blend is left to stand for any length of time 3 hours

or more, and remixing is necessary before use. The draw bar coating technique was used to cast films on the small test plates and seems to offer uniform thickness. The plates could easily have been spray coated with minimum thickness variation. The coating and curing of the test samples presented no problem at all of the filler coatings, however the curing schedule outlined on page 7 can be reduced to the following cure schedule 15' 100°C - 15' 150°C - 10' 250°C - 5' 300°C. Fairly smooth bubble free coatings were obtained. Information regarding the characteristics of the cured films on the test samples is reported in Table III. Figure 9 gives an overall view of the coated samples. A close-up of the coated sample is shown in Figure 10. Note that there is an upper (resin) and lower (WSe₂) layer. Apparently a very thin film of the resin is formed on the surface of these coatings with the WSe₂ lubricant comprising most of the underlying layer.

Test Results

In Figures 9-21 general views of the samples before and after testing are illustrated. In Figures 22-38 are depicted the curves showing coefficient of friction versus cycles for the various compositions tested.

The friction and wear properties of plastics are influenced by many variables, among these are the lower yield and shear strengths, thermal conductivities and elastic moduli compared to metals.¹⁻² In metals plastic deformation is a major mechanism even under low loads; however, because of the viscoelastic nature of plastics it is generally believed that the deformation of these materials is partially plastic and partially

TABLE III

Friction Data for Resin Filler Compositions

Resin	Filler ¹ Concentration	Aver. Film Thickness	Test Surface	Initial Coefficient of Friction μ	Final Coefficient of Friction μ
I-7	10 vol % 19.4 wt %	0.0020"	film on film	0.08	0.22 at 200 cycles
I-7	20 vol % 39.4 wt %	0.0017"	film on film	0.08	0.26 at 100 cycles
I-7	40 vol % 79 wt %	0.0018"	film on film	0.21	0.29 at 2000 cycles
I-7	40 vol %	0.0020"	film on bare metal (32-63 rms surface)	0.15	0.15 at 3500 cycles
I-8	10 vol %	0.0018"	film on film	0.24	0.28 at 1000 cycles
I-8	20 vol %	0.0020"	film on film	0.27	0.25 at 2700 cycles
I-8	40 vol %	0.0019"	film on film	0.20	0.27 at 2700 cycles
AI-131	10 vol %	0.0017"	film on film	0.29	0.25 at 1850 cycles
AI-131	20 vol %	0.0020"	film on film	0.40	0.24 at 3000 cycles
AI-131	40 vol %	0.0017"	film on film	0.21	0.21 at 3700 cycles
AI-131	40 vol %	0.0013"	film on bare metal WSe ₂ rubbed in on surface of film & bare metal	0.14	0.32 at 3700 cycles
AI-131	40 vol %	0.0016"	film on bare metal	0.14	0.32 at 2500 cycles
AI-131	40 vol %	0.0025"	WSe ₂ rubbed in on film surface - film on film	0.14	0.27 at 3550 cycles
AI-131	90 wt %	0.0009"	film on bare metal	0.19	0.28 at 2600 cycles
AI-131	90 wt %	0.0015"	film on film	0.13	0.37 at 2150 cycles
I-7	20 vol %	0.0022"	film on film humidity exposed	0.32	0.29 at 2000 cycles
AI-131	20 vol %	0.0021"	film on film humidity exposed	0.29	0.27 at 3650 cycles

1. Tungsten diselenide - 1.1 μ particle size with < 0.5% free tungsten.

elastic over a wide load range.³⁻⁵ Because of these facts and due to the severity of the tests, the records may not exhibit the same actions more commonly encountered in friction tests. Three friction or friction-related mechanisms are suggested by the test records and are shown by Figure 39. Part (a) is an example of steady sliding action. Part (b) suggests a higher coefficient of friction as sliding is impending and then a reduced value during sliding. This is a common occurrence when static friction forces are higher than those occurring after sliding commences. Part (c) resembles part (b) but is followed by galling or abrading away of the film. In any event, maximum loads are used to calculate the coefficients of friction.

Particles of film found on the test fixture and the appearance of the samples after testing give further evidence of abrasion.

The variation in normal force can also be related to the abrasion as the following calculation will show. Design characteristics of the cylindrical load cell are as follows:

Length, $L = 2.00$ inches

Outside diameter, $D = 2.00$ inches

Inside diameter, $d = 1.68$ inches

Modulus of elasticity of 304 stainless at 550°F, $E = 25 (10)^6$ psi

From D and d , cross-sectional area, $A = 0.927$ in²

From elementary mechanics, the change in length, ΔL , due to an applied axial load, P , is given by

$$\Delta L = \frac{PL}{AE}$$

Thus a 1000 lb. change in the normal force can be caused by a change in load cell length of

$$\Delta L = \frac{(1000 \text{ lb})(2 \text{ in})}{(0.927 \text{ in}^2)(25)(10)^6 \text{ lb/in}^2} = 8.63 (10)^{-5} \text{ in.}$$

Of course, if .001 inches of film is abraded away the load cell will not be the only component to expand and fill the gap. But this does show that a decrease in film thickness of less than 1 mil can cause an appreciable decrease in normal force.

The three mechanisms of friction described above and combinations thereof are found in most of the friction force records. Examples from two tests with the AI-131 resin are found on Figures 40 and 41.

Data Reduction

From the records of friction force and normal force, the coefficient of friction is easily determined from the relation

$$\mu = \frac{F}{N}$$

where F is one-half the recorded friction force since two pairs of sliding surfaces are involved. The friction forces recorded on the tension half of the loading cycle are not always equal to those of the compression portion. This anomaly is attributed to slight misalignment and possible variation in the film surface. Generally the variation is small, about $\pm 3\%$ from the average, and at most $\pm 6\%$. Therefore, the average force from the tension and compression portions of the cycle is used in all coefficient of friction calculations.

Effect of Resin Type and Filler Concentration

The data in Table III show the differences one can expect when the binder resin is changed. The resin type does affect the coefficient of friction and the wear properties. Resin AI-131 is considerably better than I-7 and I-8 resin at all filler concentrations whether the sliding surface is film on film or film on bare metal. However, anomaly exists which is unexplainable at the present time. Friction tests with I-7 resin 40 vol % filler loading with film on bare metal (32-63 microinches rms surface) give very low friction coefficients at long wear cycles (0.15 over 3500 cycles at a film thickness of 2 mils). This is better than any of the other compositions but is only an isolated case whereas AI-131 compositions give consistently longer wear cycles at all loadings even though the friction coefficient was slightly higher. A possible explanation for the improved performance of AI-131 resin could be that it offers more adhesion and holds the WSe_2 particles together better than I-7 or I-8 resin. This would mean that AI-131 is a more efficient binder resin.

After examining the samples after testing one notices that in all cases there was a shiny jet black film (undoubtedly WSe_2) that remained on the sliding surfaces. This film was soft and transfer of the lubricant was evident if one rubbed his fingers across the surface of the film. On the other hand when one attempted to lift a section of the film it was brittle. Undoubtedly compaction of the WSe_2 particles was obtained under the high test loads. The amount of film present seemed to depend on the type of resin used and the concentrations. With AI-131 resin this

jet black film seemed to be present in greater amounts. This observation however is based on a microscopic inspection of the samples and is only qualitative in nature. Quantitative measurements were not made on the film remaining on the surface. However, the greater the initial film thickness on the samples the more residual jet black film seemed to remain. Also, more of this film remained if both surfaces had been coated initially compared to only coating one surface. This transfer of lubricant to the sliding surfaces has been shown to be related to high adhesion and shearing within the bulk of the softer material.³ The softer material in this case being WSe_2 which is transferred to the carbon steel metal surface. The resin only acts as the binder, keeping the lubricant in place.

In addition to the above there was considerable grooving and plowing of the sliding surfaces. This can be seen in the photographs. Inspection of the test surfaces showed a fine dark brown powder mixed in with the WSe_2 lubricant. This powder is probably the resin which was ground during the constant sliding motion of the plates under the high test loads. This powder could account for the grooving of the metal surface. However, even with the presence of this powder reasonably low friction coefficients and long wear cycles were still obtained.

One notices also from the data in Table III and Figures 22-31 that high friction coefficients are obtained initially and are highest with AI-131 resin. Microscopic examination of the surface of the cured test samples indicated that a thin film of resin was present which has to be worn down slightly to make contact with the underlying lubricant film (see Figure 10). The fact that the initial friction coefficient

was highest with AI-131 indicates that a high shearing force was necessary to break the film surface. This tends to indicate better adhesion with this resin.

The effect of high filler loadings are indicated in Figures 36-38. Apparently, a critical filler concentration is reached above which no improvement in wear properties is obtained. However, the initial frictional coefficient is reduced at these high loadings but at the expense of wear. It seems reasonable that high filler loadings would give low frictional coefficients but on the other hand there is much less resin available to act as a binder for the filler at these high loadings. As a result the lubricant particles are only loosely held together and can separate from the sliding surfaces decreasing the wear rate.

Effect of Humidity

Friction and wear properties of both I-7 and AI-131 containing 20 vol % filler are not affected by the presence of moisture. The 20 vol % compositions were tested before the optimum resin-filler composition was chosen. However, there is no reason to believe that the optimum composition (AI-131 + 40 vol % filler) would be adversely affected by moisture. Figures 32-33 show the friction and wear cycles for the resins exposed to the humidity conditions.

Effects of Radiation

Several sets of carbon steel plates were coated with AI-131 + 40 vol % WSe₂ and sent to the Bettis Laboratory to arrange for irradiation

of these samples before returning them to Westinghouse Research for friction tests. Detailed information on the thickness and weight of coating on the plates is reported in Table IV. Only one set of samples was returned to Westinghouse Research for further tests, the rest were retained by Bettis for their evaluation. Those items identified with an asterisk are the samples that were tested at Research. All of the samples were irradiated with gamma radiation from a Co^{60} source to a dose rate of $5-6 \times 10^8$ ergs/gm (C). Figures 42-43 show the coated plates before and after testing. The latter figure contains the curves of the coefficient of friction data as a function of time.

Examination of the samples after testing revealed the same kind of failures as the other samples described on Page 18. The initial coefficient of friction (μ_1) of 0.14 is quite low and substantiates the fact that when WSe_2 powder is rubbed into or onto the surface of the coated materials a lower starting friction coefficient is obtained.

Although it might appear that the irradiation adversely affected the friction properties because of the short cycle time this is not the opinion of the authors. Examination of the samples after testing indicated an uneven wear pattern. Apparently there was a film buildup in the center of the coated plates which would serve as a pivot to give rise to a "see-saw" type of motion in combination with the sliding motion. As a result of this considerable plowing of the edge of one surface on the other flat surface would result. This would tend to leave more film on the plate doing the plowing. This is evident from Figure 42 (top plate in bottom photo). This kind of sliding motion would short the wear life considerably.

TABLE IV

Coating¹ Data on Radiation Test Samples1" Raised Surface Test Plates

<u>Sample No.</u>	<u>Coating Thickness, inches</u>	<u>Total Weight of Coating, gms.</u>	<u>Weight of WSe₂, gms.</u>	<u>Weight of Resin, gms.</u>
3*	0.0022	0.0631	0.0278	0.0353
4*	0.0022	0.0625	0.0276	0.0349
5	0.0022	0.0568	0.0251	0.0317
6	0.0026	0.0775	0.0343	0.0432
7	0.0023	0.0611	0.0270	0.0341
8	0.0022	0.0574	0.0254	0.0320

Large Test Plates

1	0.0023	0.160	0.071	0.089
2	0.0022	0.200	0.089	0.111
7	0.0020	0.230	0.102	0.128
9*	0.0020	0.180	0.075	0.105
10*	0.0019	0.140	0.062	0.078
11	0.0022	0.160	0.071	0.089

1. AI-131 resin + 40 vol. % WSe₂.

Compatibility with Inconel

Sample plates having the 1" square raised surface were coated with AI-131 + 40 vol % WSe₂ to a dry film thickness of 2.4 mils and tested against bare Inconel. Figure 44 shows the samples before and after testing. Coefficient of friction data as a function of time is depicted in Figure 45. Fairly good results were obtained. Again as with the irradiated samples the same kind of wear phenomenon is observed. Friction coefficients at the start is about 0.18 not quite as low as with the previous samples and 0.29 after 2600 cycles.

Static Load Test

After the 90 day exposure period the jig containing the coated samples was positioned in the Baldwin Testing machine described on page 10 and the break away force necessary to start sliding was recorded. This force measured 8200 lbs which is equivalent to the initial friction loads recorded in previous tests on samples not exposed under the static load condition. Examination of the samples after they had been pulled apart revealed that the lubricant film still remained on both surfaces. However on one of the samples the lubricant film was completely removed (see Figure 46). Apparently, cohesive and adhesive failure can be obtained. These results indicate that negligible bonding (film to film contact) occurs over the exposure period.

Compatibility

Stainless steel shim material (10" x 6" x 0.005") was coated with AI-131 + 40 vol % WSe₂ to a dry film thickness of 2 mils. In addition, two plates with the 1" square raised surface were also coated with the

same resin to a dry film thickness of 2.5 mils on each plate. A free film was also prepared by casting the above resin solution (18% solids) on a glass plate and curing 15 min 100°C - 15 min 150°C; stripped off glass plate and further cured to 300°C on a frame. (See Figure 47 for an illustration of the coated stainless steel and the free film.)

Effect of Film Thickness and Surface Condition of Sliding Plates

Detailed experiments in trying to keep the various parameters constant in order to determine the effect of film thickness on the friction and wear properties have not been made and no definite conclusions can be drawn in this area. The results in Table III seem to indicate that the thicker the film the better the properties. However, there is considerable scatter in the data and one should take this into account in the final interpretation. It may well be that there is a minimum and/or maximum critical film thickness above and/or below which satisfactory friction properties cannot be maintained. This has not been established and should be kept in mind when reviewing this data.

On the other hand, one can draw conclusions regarding the effects of test surface conditions on the friction properties. The initial coefficient of friction is lower for film on the bare metal than for film on film at a given filler concentration. However, greater surface damage is noticed on the sliding surfaces with film on metal. Another way of lowering the initial coefficient of friction is to use higher filler

loadings. These results are shown in Table III. However, wear life is reduced at high filler loadings. If powdered WSe_2 is rubbed into the surface of the sliding plates the friction coefficient is similarly reduced. Again these results can be found in Table III.

It should be noted that considerable scatter in the data is obtained. In this regard samples prepared from the identical resin composition do not show good reproducibility. Compare AI-131 compositions at 40 vol % (79 wt %) in Table III and also AI-131 at 20 vol % loadings. The conditions of the film surface may account for some of the variation.

Coating of Large Plate Sections

Both the draw bar and the spray coating technique have been used successfully to apply the lubricant coatings on the metal plates. The draw bar technique, although it allows a thicker coating to be applied, seems to have limitations. Because of the irregularity of the plates (holes) plugs have to be used. When the wet coating is applied and the plugs removed there is considerable buildup of the coating at the edge of the holes in the plate. This is attributed to the surface tension energy required to break the wet coating free from the plugs. The excess resin then builds up on the edges. Thickness variations were between 0.3-0.5 mils. Smooth homogeneous coatings were obtained but the buildup at the edges may be prohibitive. Only one side of the plate can be coated at a time so that the curing process would be quite long if both sides are to be coated.

The spray technique, on the other hand, seems to be more favorable at least from the standpoint of coating two sides simultaneously and ease of operation. Thickness variations ranged between 0.3-0.5, about

the same as obtained for the draw bar technique, however, no buildup at the edges was obtained. Final dry film thickness was lower for the spray coating procedure than for the draw bar but this presents no problem. No run off or sag was noticed during the spraying and "fish eyes" prevalent in the first coat were covered over with the second coat. Twenty-five psi gun pressure seemed to offer satisfactory results. The resin solution was quite thin at 18.3% resin solids. The 21.5% resin solids solution seemed to be more satisfactory than the lower solids. Besides a thicker build could probably be obtained with the higher solids solution (see Table II and Figures 20-21).

Effective Life of Lubricating Film

It is recognized that no present technique exists by which a 20 year life prediction at a temperature of 550 or 600°F may be made on the basis of short time accelerated tests for a lubricant system. There are no standard test procedures which can be extrapolated or have been extrapolated to this time/temperature limit for a lubricant system of this kind. However, the literature abounds in time/temperature relationships for predicting the service life of organic polymers. Use is made of the Arrhenius equation in which some property of the material is measured as a function of time at various temperatures. A value of this property (determined arbitrarily by its end use requirement) and the log of it is then plotted as a function of the reciprocal of absolute temperature. Extrapolation to a lower temperature limit is then made.

One could do exactly the same thing in the present situation. For example, one could measure the friction coefficient versus wear cycles at several temperatures. Then one could plot the time for the friction coefficient to reach a certain value versus the reciprocal of the absolute temperature on semi-log paper and extrapolate. This method is easy enough to do, but in the present situation we would have to run tests at temperatures of about 700-800°F to obtain values for the extrapolation. At such high temperatures the chemical reactions taking place are so different than those at the lower temperatures that extrapolating this data would be very hazardous. It is our opinion that a method for predicting the life behavior of a lubricant under the test conditions of this program would not be feasible from the standpoint of extrapolating the test results.

Conclusions

Polyimide lubricants have been developed and shown to perform satisfactorily under the conditions of the test requirements. Friction and wear properties of these polyimide lubricants are affected by many parameters some of which are resin type, filler concentration, film thickness, test surface, etc. The data in this report indicate the following:

1. The best combination of friction and wear properties is obtained with AI-131 resin containing 40 vol % (79 wt %) of WSe_2 (particle size 1.1 μ).

2. Optimum film thickness for the lubricating film appears to be about 2 mils.
3. Both sliding surfaces should be coated to give film to film contact.
4. Lower initial friction coefficients are obtained with film to metal but film to film gives longer wear cycles.
5. The lubricating properties of the resin-filler compositions are not adversely affected by humidity.
6. WSe_2 containing <0.5% free W having a particle size of $\approx 1.1\mu$ is satisfactory for the filler in polyimide resins.
7. Friction and wear properties of AI-131 + 40 vol % WSe_2 are not adversely affected by gamma radiation.
8. Draw bar and spray coating can be used to apply the resin-filler compositions to large plate sections.
9. The preferred system AI-131 + 40 vol % WSe_2 is compatible with Inconel.
10. Initial coefficients of friction can be reduced by rubbing powdered WSe_2 into the surfaces of the sliding metal plates.
11. A critical filler concentration is reached above or at which low friction coefficients are obtained but wear is considerably reduced.
12. Film to film contact at 550°F under 15,000 psi for long periods of time shows negligible bonding.

Recommendations

The preferred resin-filler composition is AI-131 (at 18% resin solids) + 40 vol % (79 wt %) WSe_2 (with <0.5% free W) having a particle size of less than 5μ and preferably $\approx 1.1\mu$. Both contacting plates should be coated with the lubricant coating to a thickness of 1.8-2.2 mils on each

plate. Spray coating can be used satisfactorily. Powdered WSe_2 should be rubbed into the coated plates to lower the coefficient of friction. It is expected that the above recommendations will give a lubricant coating with a friction coefficient between 0.14-0.27 for 3700 cycles. The suggested coating procedure is outlined in Appendix II.

Acknowledgement

The authors wish to thank Mr. D. J. Boes for his helpful discussions on lubricants and Messrs. W. R. Koryak and L. Galata for their assistance in the preparation and testing of the samples.

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4. Ibid, p. 723.
5. G. Kitchen and H. Azzan, "Realistic Friction Testing", Machine Design, p. 195, March 16, 1967.

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APPENDIX I

Calculation of Filler Concentration

The amount of lubricant to be added to x grams of the resin solution was calculated on the basis of MoS_2 as the reference. The amount of MoS_2 was calculated to give a 10-20-40 weight percent of the filler in the resin solution. The volume occupied by this weight of filler was then computed and used to calculate the weight of WSe_2 needed to give an equal volume in the resin solution. The calculation shown below is for AI-131 resin.

10 vol % - 19.4 wt % - 30 g of resin solution at 26% solids = 7.8 g.
dry resin, 10% of which is added $\text{MoS}_2 = 0.78$ g. $D = M/V$, $4.8\text{g/cc} = 0.78/V$,
 $V = 0.162$ cc. The amount of WSe_2 to be added is: $D = M/V$, $9.5\text{g/cc} =$
 $M/0.16$, $M = 1.53$ gms.

20 vol % - 39.4 wt % - 30 g of resin solution at 26% solids = 7.8 g.
dry resin, 20% of which is added $\text{MoS}_2 = 1.56$ g. $D = M/V$, $4.8\text{g/cc} = 1.56/V$,
 $V = 0.325$ cc. The amount of WSe_2 to be added is: $D = M/V$, $9.5\text{g/cc} =$
 $M/0.32$, $M = 3.08$ gms.

40 vol % - 79 wt % - 30 g of resin solution at 26% solids = 7.8 g. dry
resin, 40% of which is added $\text{MoS}_2 = 3.12$ g. $D = M/V$, $4.8\text{g/cc} = 3.12/V$,
 $V = 0.65$ cc. The amount of WSe_2 to be added is: $D = M/V$, $9.5\text{g/cc} =$
 $M/0.65$, $M = 6.16$ gms.

APPENDIX II

Suggested Coating Procedure

The following coating procedure is not the final recommended one but should serve only as a guide for applying the lubricant coating to the metal plates.

The resin-filler composition should have a Gardner viscosity of JK (Zahn Cup No. 33, 35 sec)*. This composition can be sprayed satisfactorily using a gun pressure of 25 lbs. A wet film thickness of 4 to 5 mils yields a dry film thickness of 0.9 to 1.1 mils. The metal plates should then be cured: 15 min. 100°C -- 15 min. 150°C -- 15 min. 200°C -- 10 min. 250°C -- 35 min. 300°C.

*The viscosity of the resin-filler composition can be reduced by diluting with an appropriate solvent. Dimethylacetamide is the recommended solvent.

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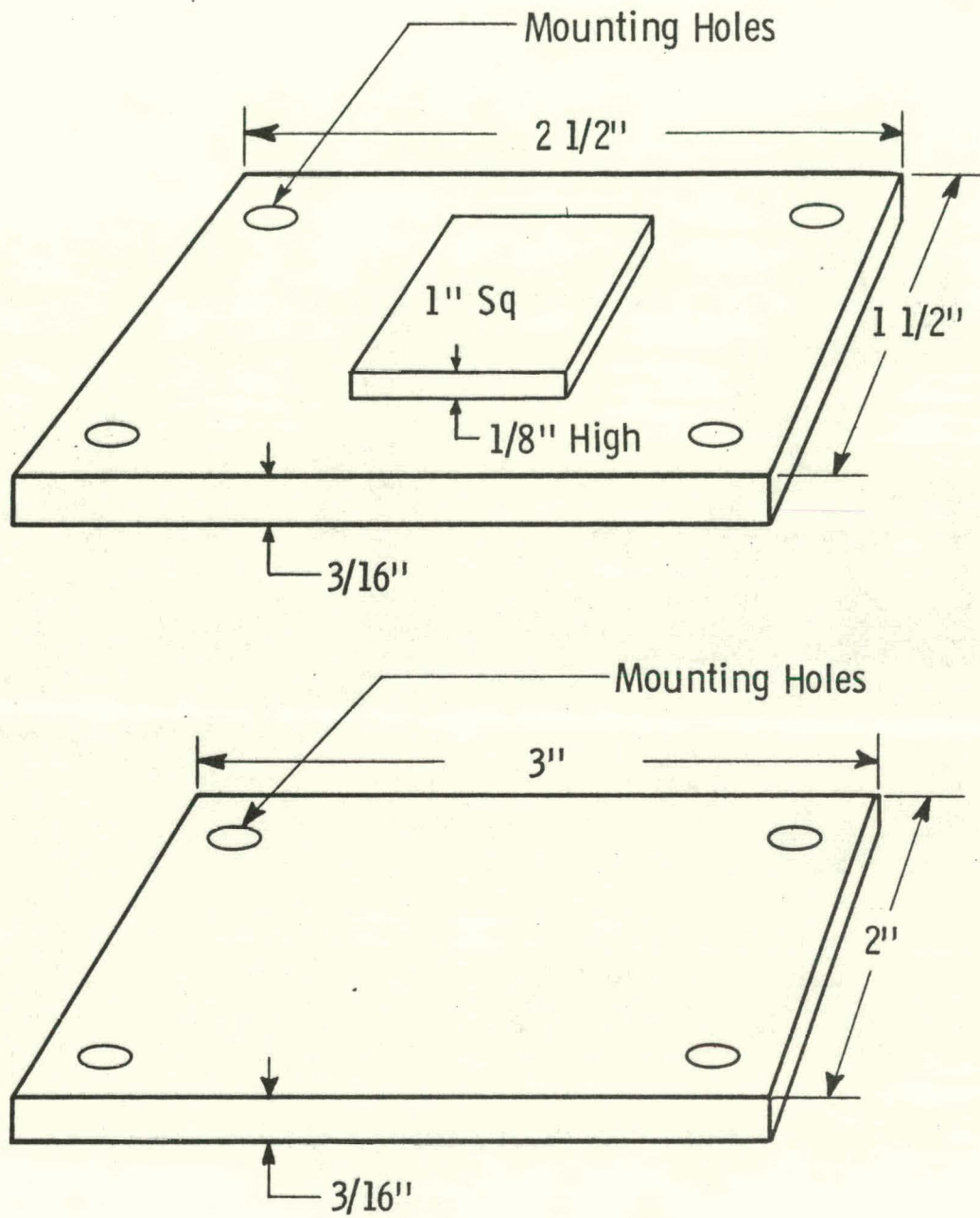


Fig. 1—Illustration of metal test plates that were coated with the resin-filler compositions

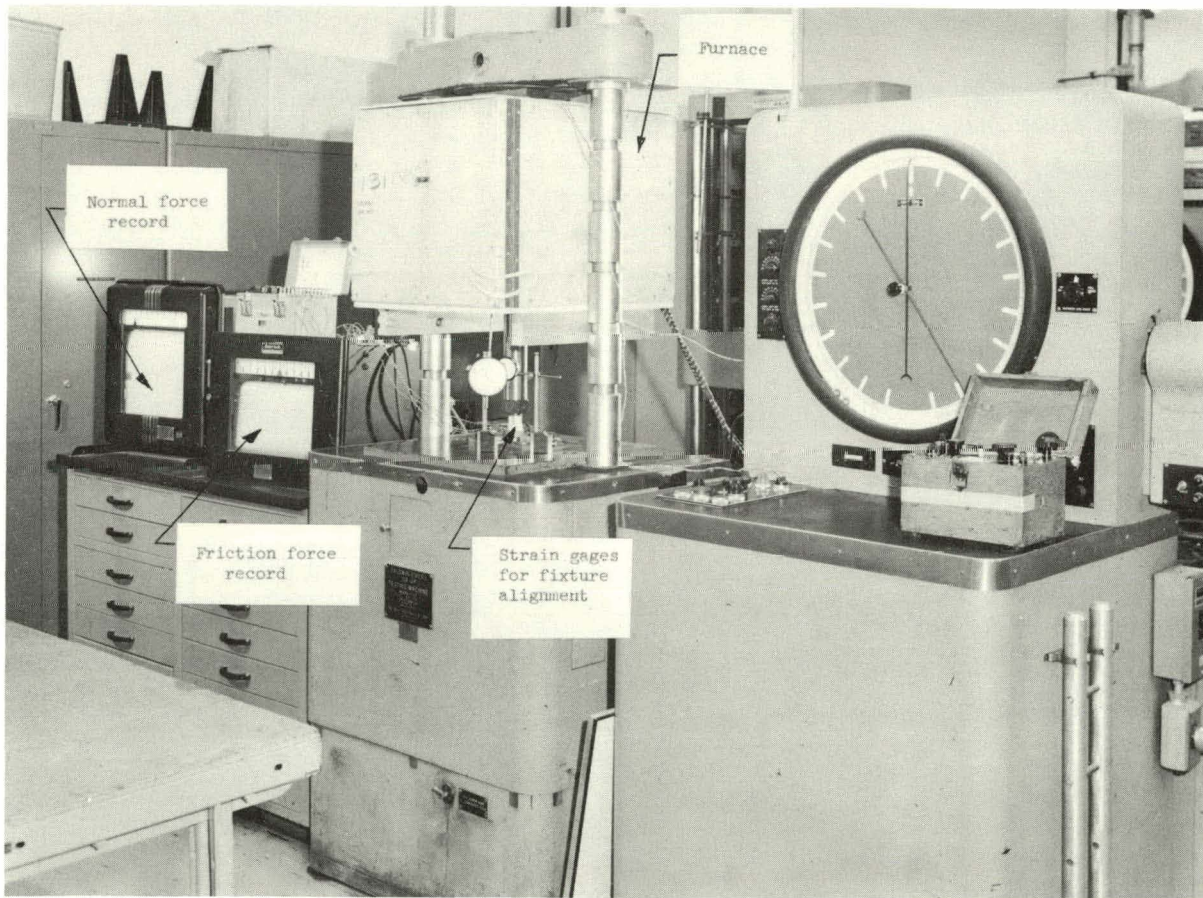


FIGURE 2 - OVERALL VIEW OF SLIDING FRICTION TEST APPARATUS

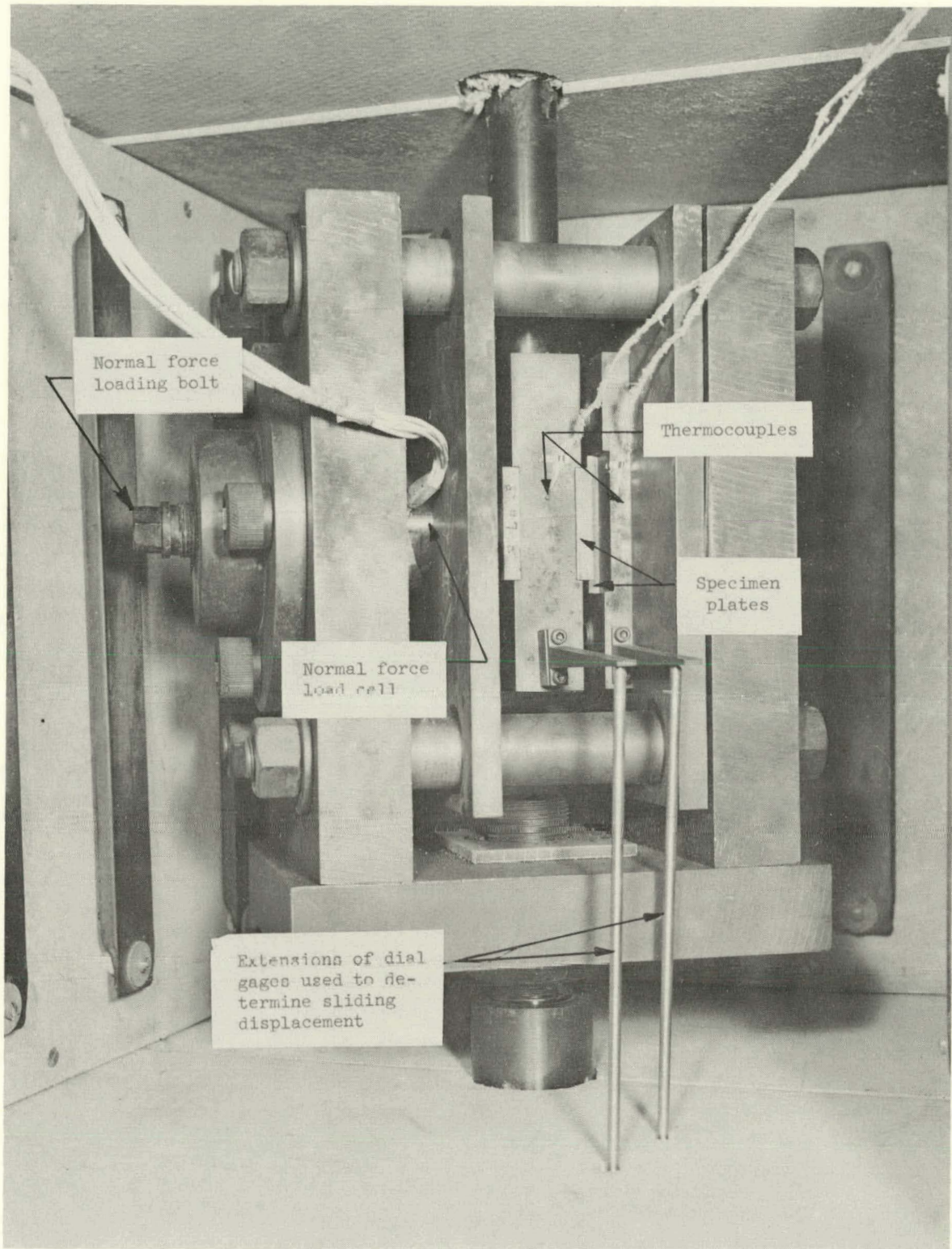


FIGURE 3 - VIEW INSIDE FURNACE SHOWING
DETAILS OF SLIDING FRICTION TEST FIXTURE

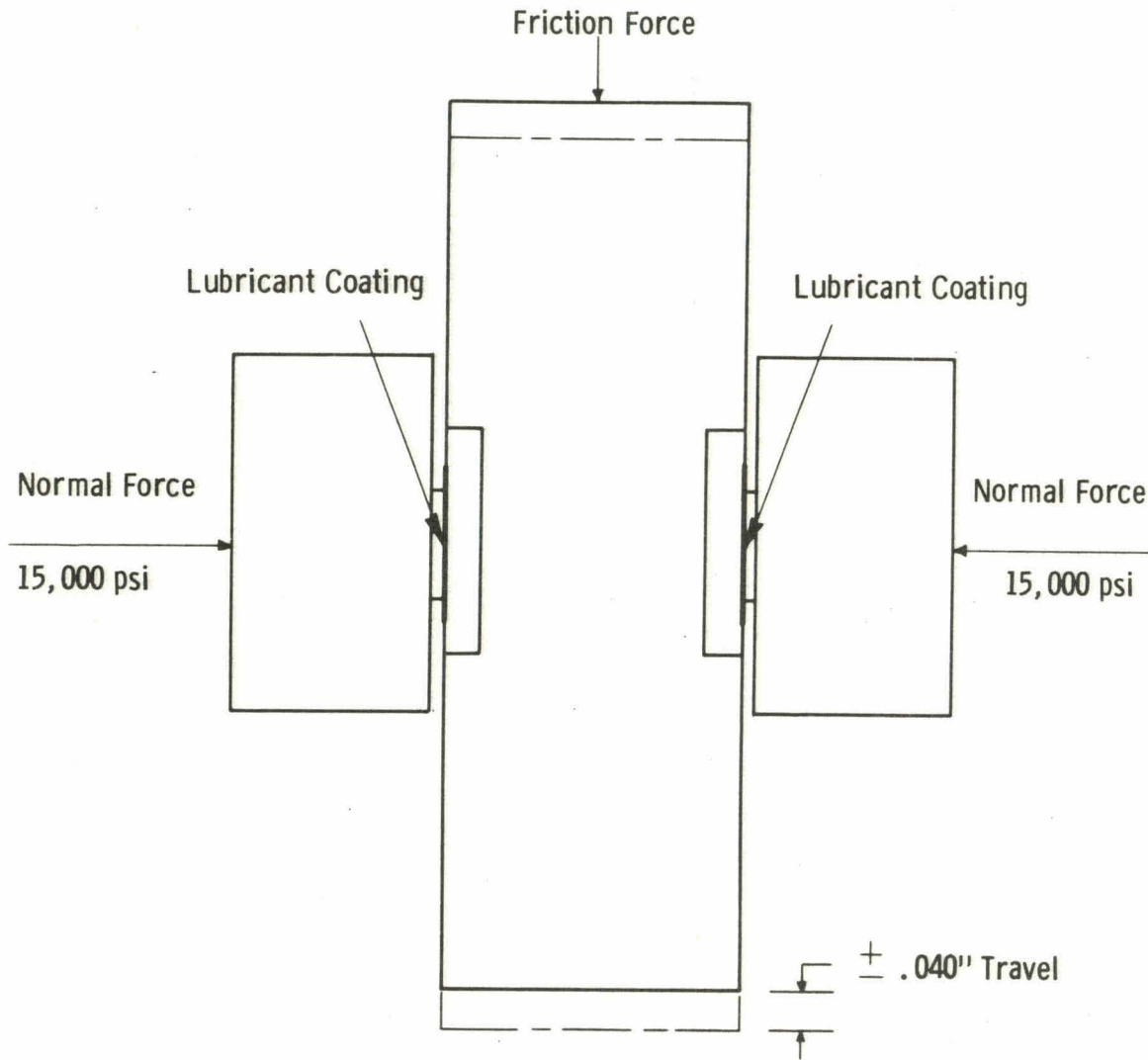
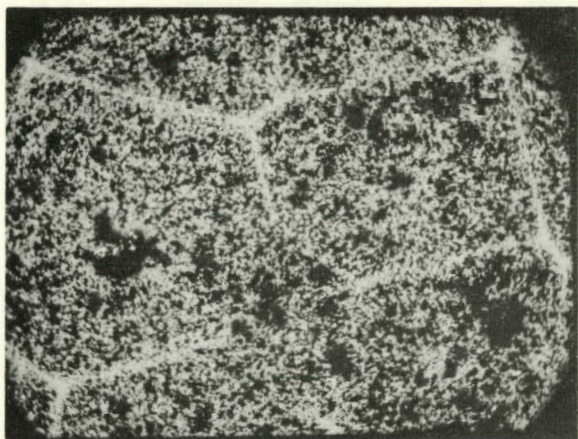
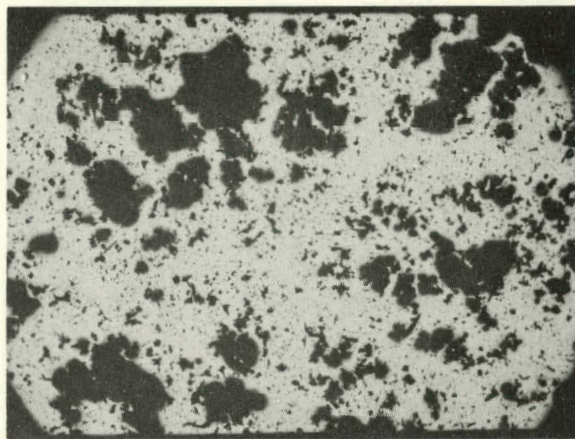


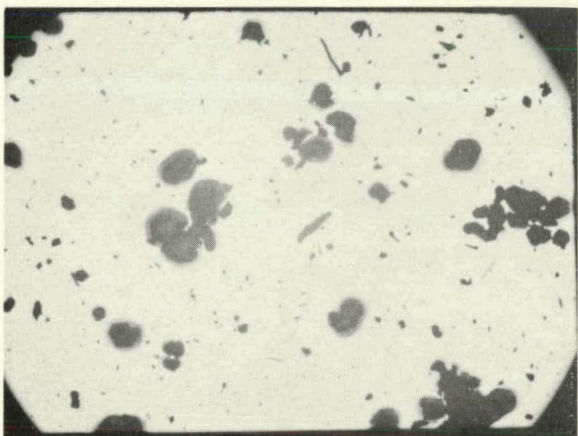
Fig. 4—Schematic diagram of friction test



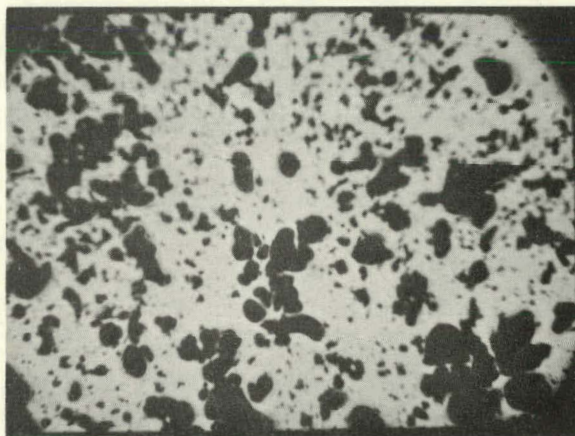
MoSe₂ (200 mesh)



WSe₂ (200 mesh)

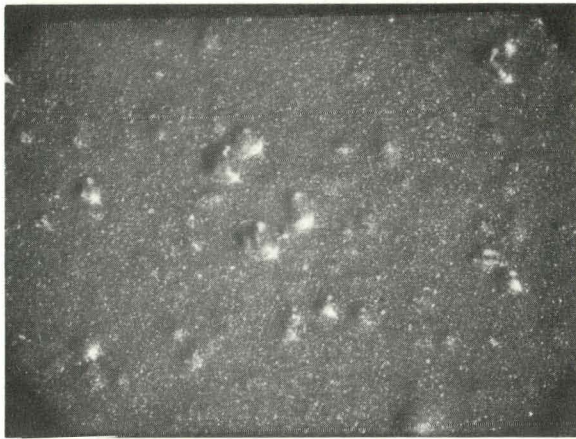


TaSe₂ (200 mesh)

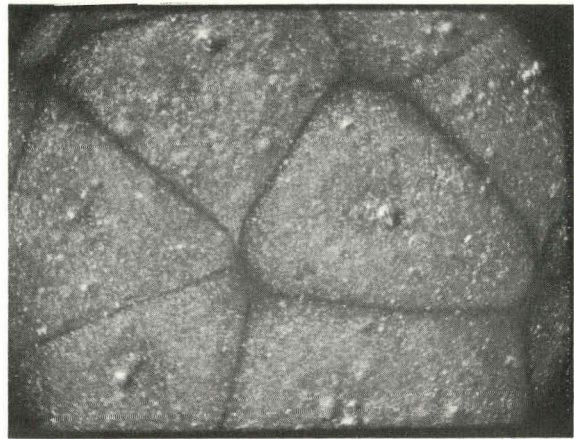


NbSe₂ (200 mesh)

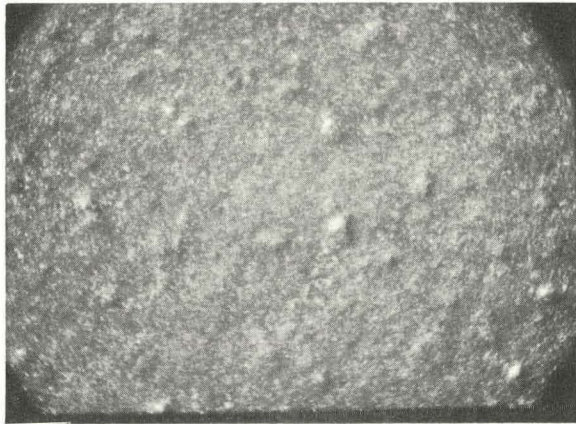
FIGURE 5 Microphotographs of doped polyimide films (1.5 mils) containing 40% by volume of various large particle size lubricants and cured on glass plates. (Mag. 50X)



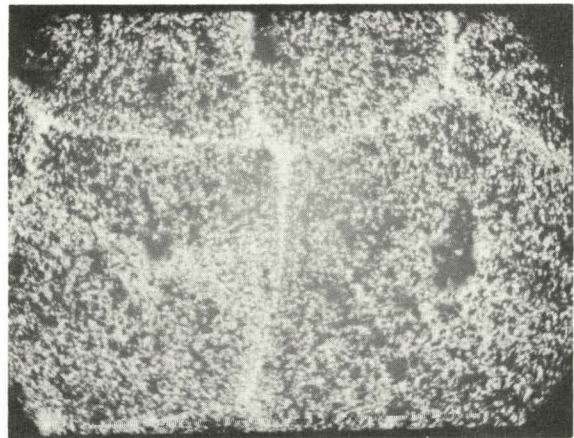
MoSe₂ (2 - 10 μ)



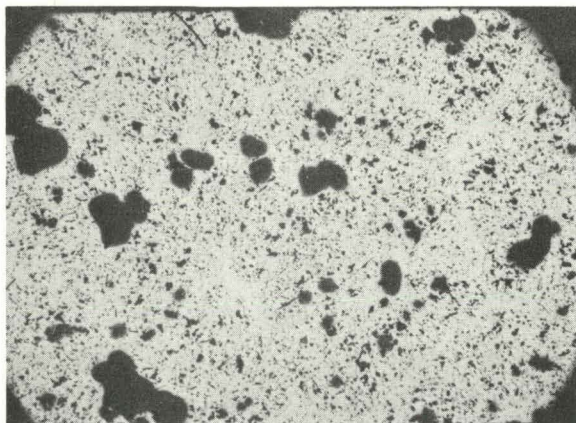
WSe (< 0.5% free W)



WSe₂ (5 μ)

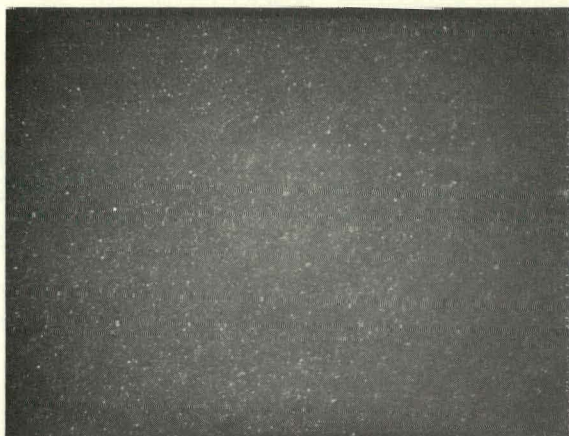


NbSe₂ (5 μ)

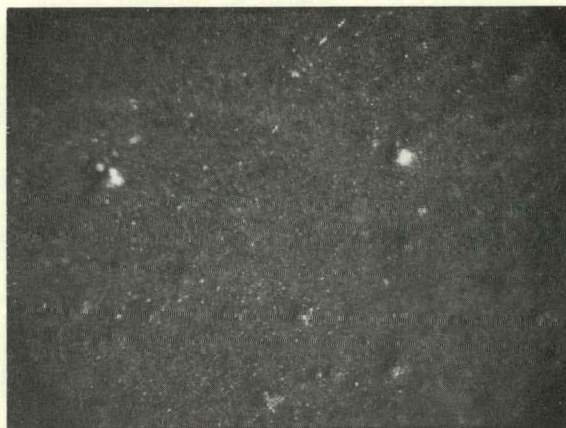


TaSe₂ (5 μ)

FIGURE 6 Microphotographs of doped polyimide films (1.5 mils) containing 40% by volume of various small particle size lubricants and cured on glass plates. (Mag. 50X)



MoS_2 ($\sim 5 \mu$)



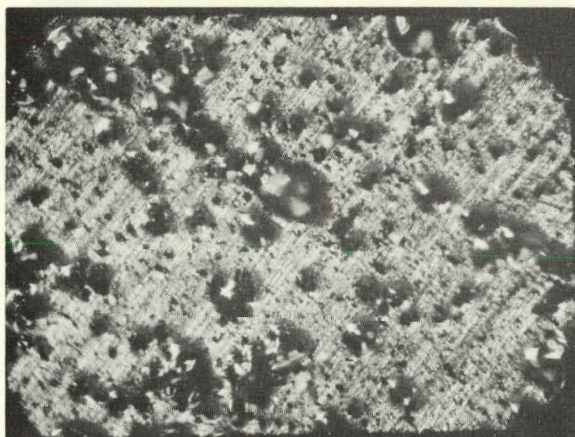
MoSe_2 (2 - 10 μ)



WSe_2 (5 μ)



NbSe_2 (5 μ)

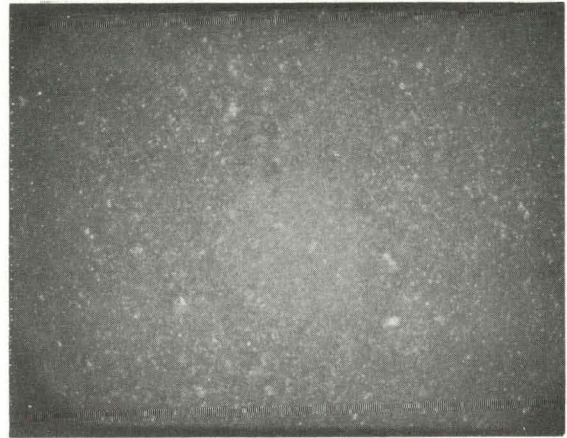


TaSe_2 (5 μ)

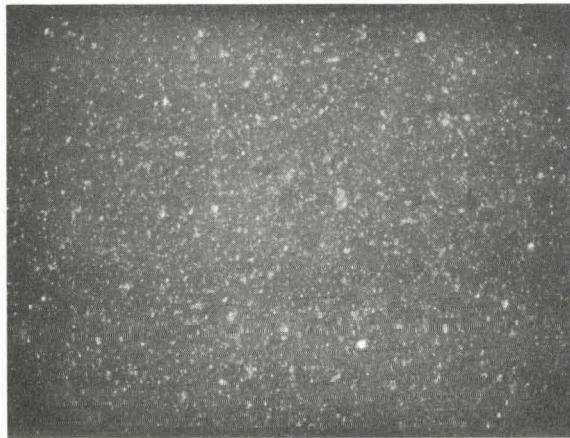
FIGURE 7 Microphotographs of doped polyimide films (1.5 mils) containing 40% by volume of various small particle size lubricants and cured on aluminum. (Mag. 50X)



MoSe₂ (2 - 10 μ)



WSe₂ (< 0.5% free W)



WSe₂ (5 μ)



MoS₂ (~5 μ)

FIGURE 8 Microphotographs of doped polyimide films (1.5 mils) containing 40% by volume of various small particle size lubricants between glass slides. (Mag. 50X)

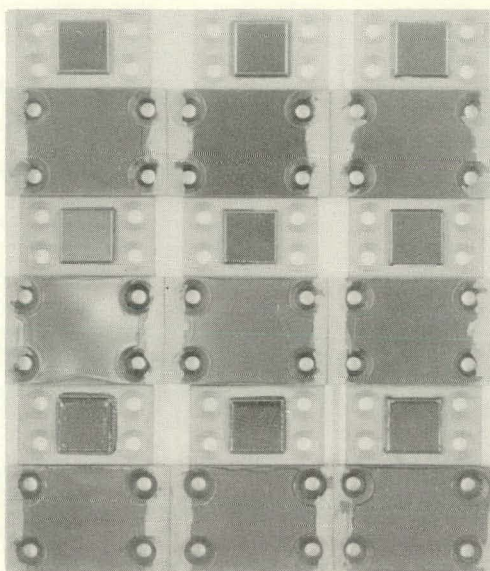


FIGURE 9 - Overall view of coated test samples. Top-I-7 resin, Middle-I-8 resin, Bottom-AI-131 resin containing 79 wt% WSe_2 .



FIGURE 10 - AI-131 + 40 vol% (79 wt%) WSe_2 . Upper Layer - organic film.
Bottom layer - WSe_2 film (Right).

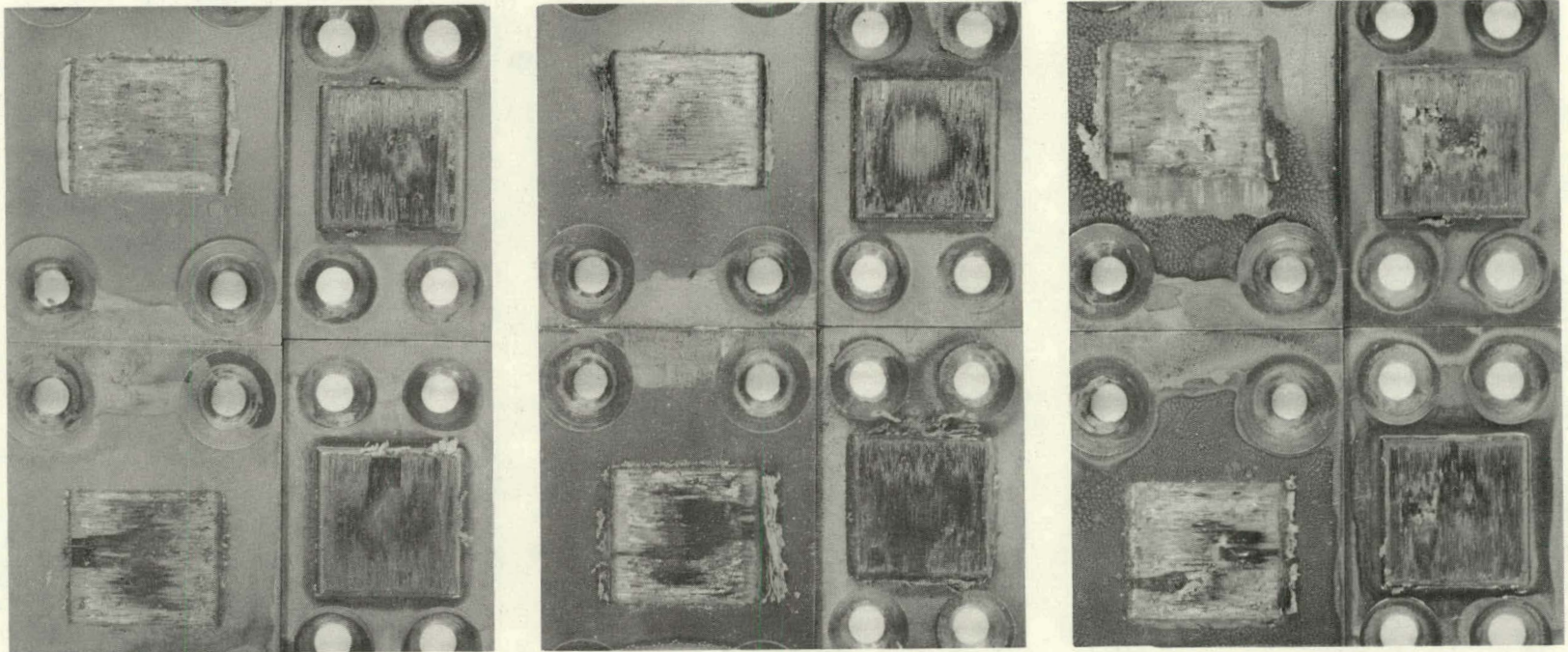


FIGURE 11 - Sample plates of AI-131 (top), I-8 (middle), I-7 (bottom) after testing. (40 vol%, 79 wt% WSe_2).

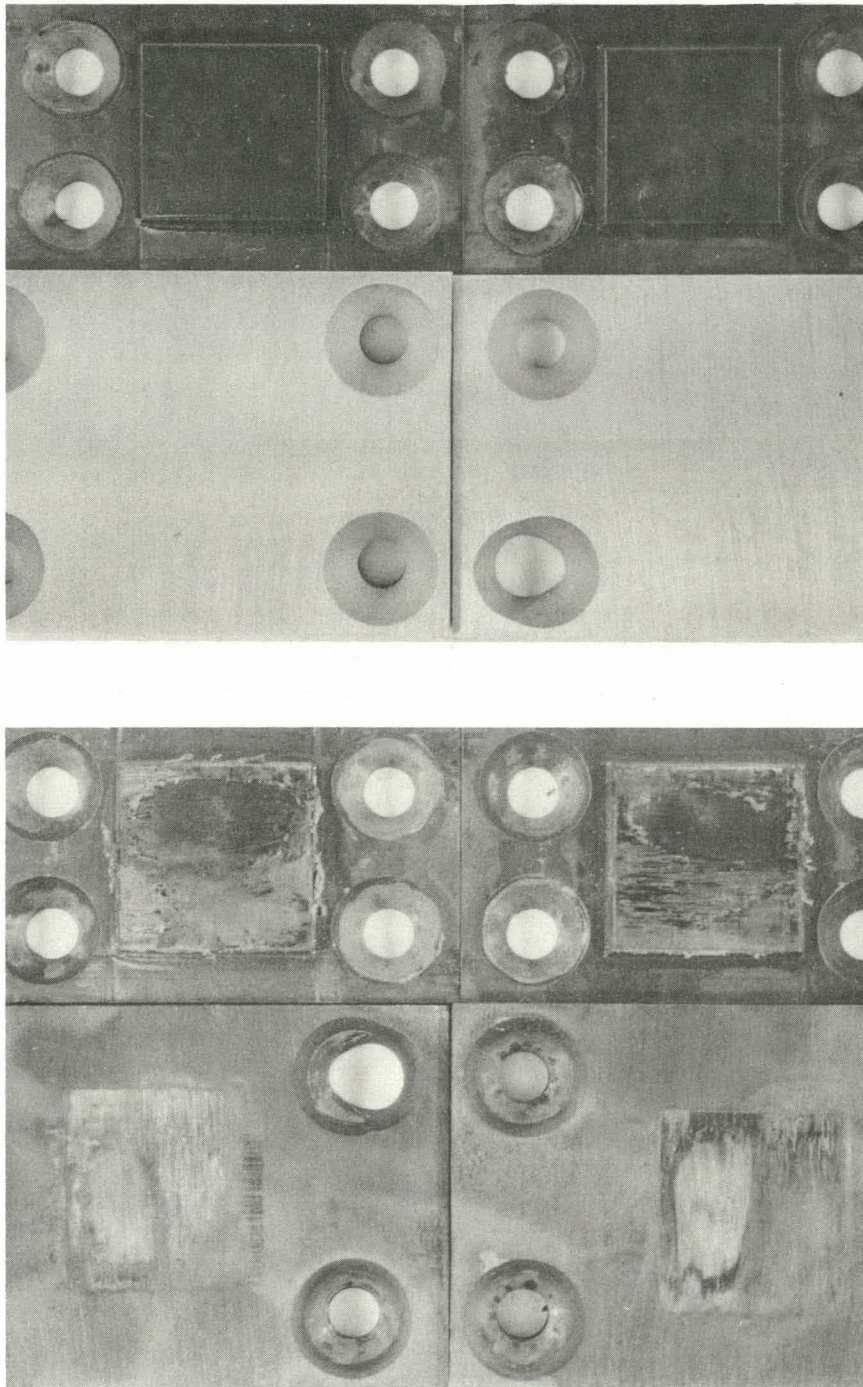


FIGURE 12 - I-7 resin + 40 vol% WSe₂ film against bare metal before and after testing.

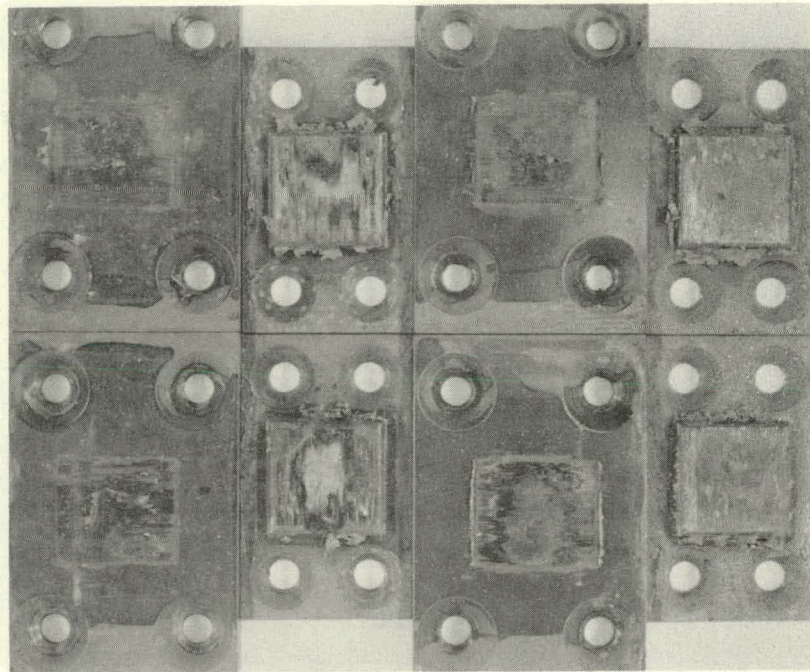


FIGURE 13 - I-7 (left) + AI-131 (right) after exposing to 100% RH and testing.
(20 vol% WSe₂).

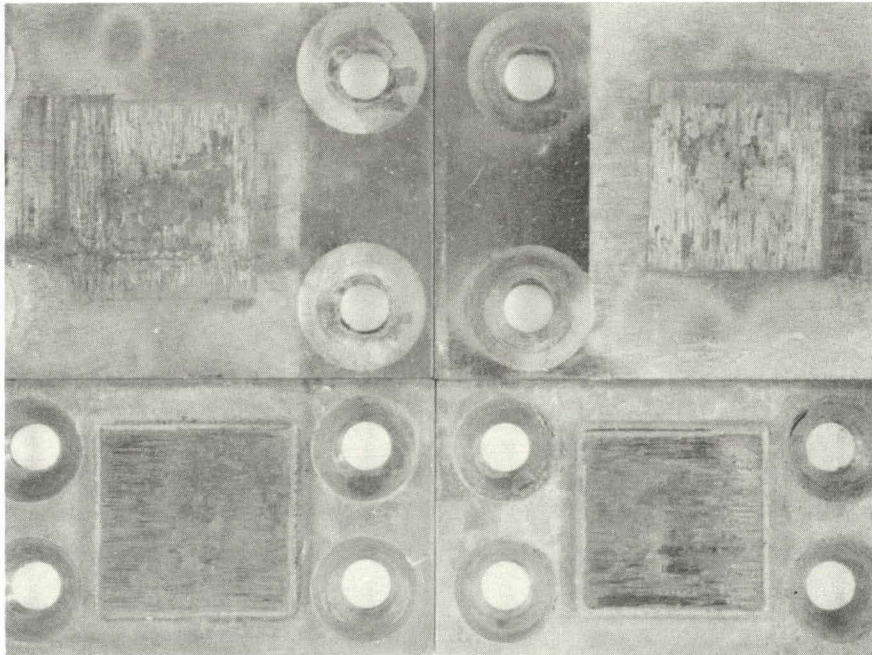


FIGURE 14 - AI-131 + 40 vol% WSe film against bare metal after testing.
(WSe₂ rubbed in on sliding surfaces.)

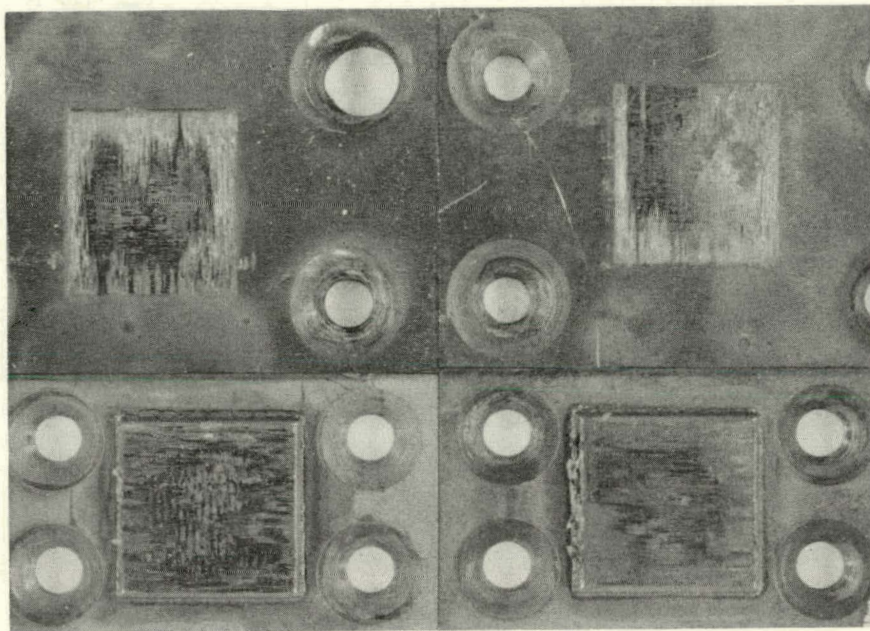


FIGURE 15 - AI-131 + 90 wt% WSe₂ film against metal.

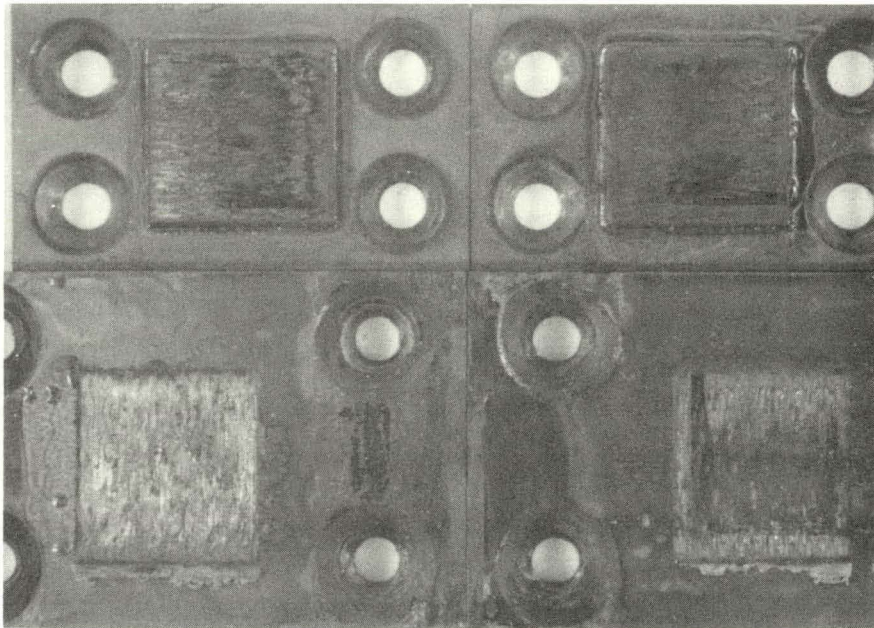


FIGURE 16 - AI-131 + 90 wt% WSe₂ film against film.

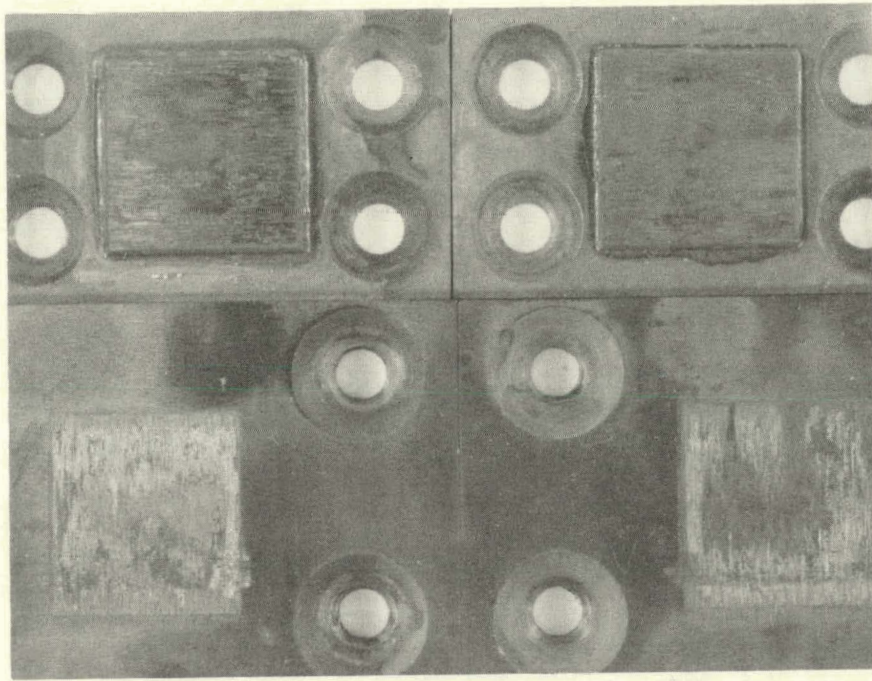


FIGURE 17 - AI-131 + 40 vol% film against metal.

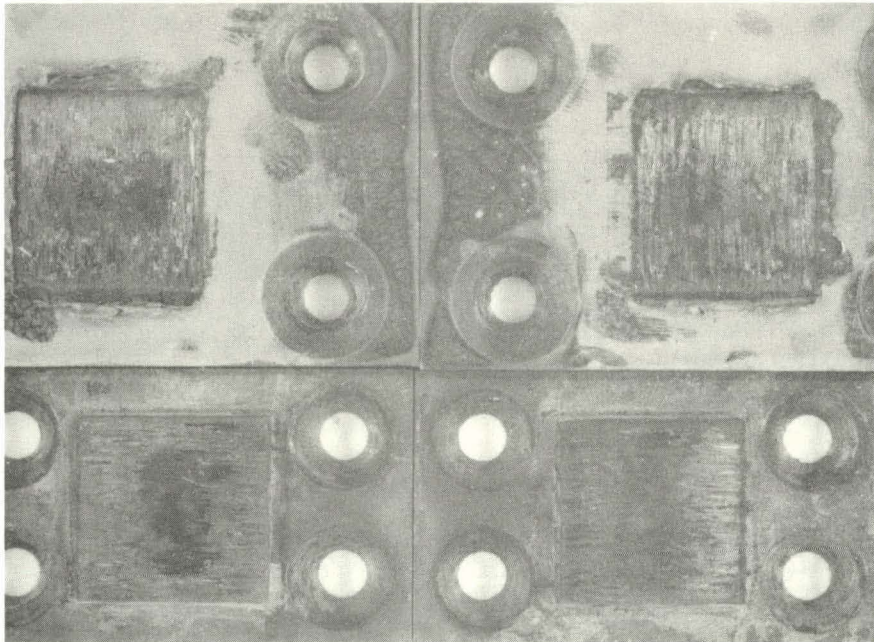
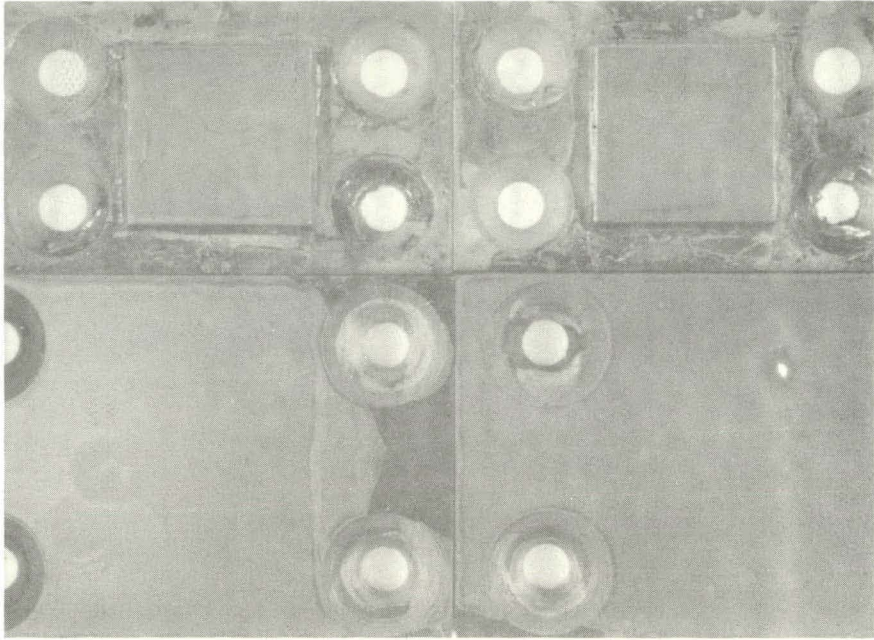


FIGURE 18 - AI-131 + 40 vol% WSe₂ also WSe₂ rubbed in on sliding surfaces.
Before and after testing.

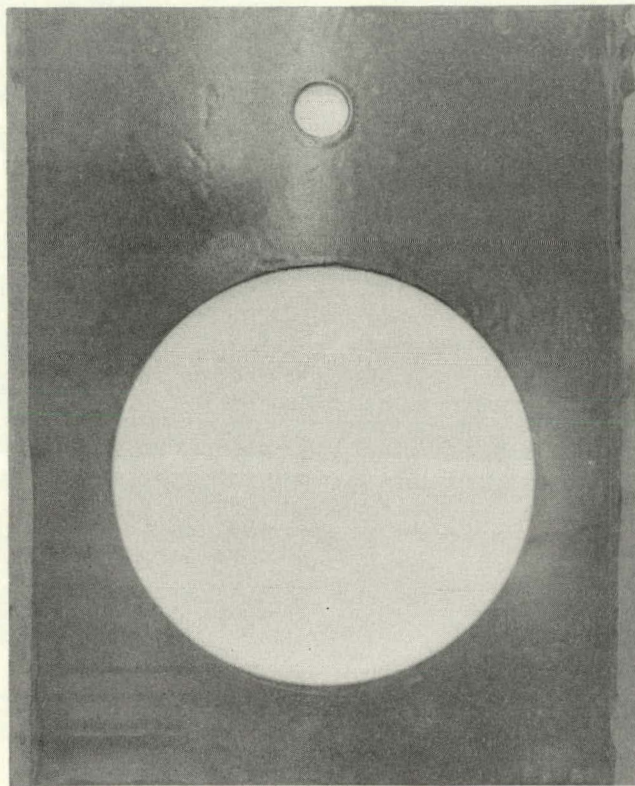


FIGURE 20 - Metal plate coated by the draw bar technique.

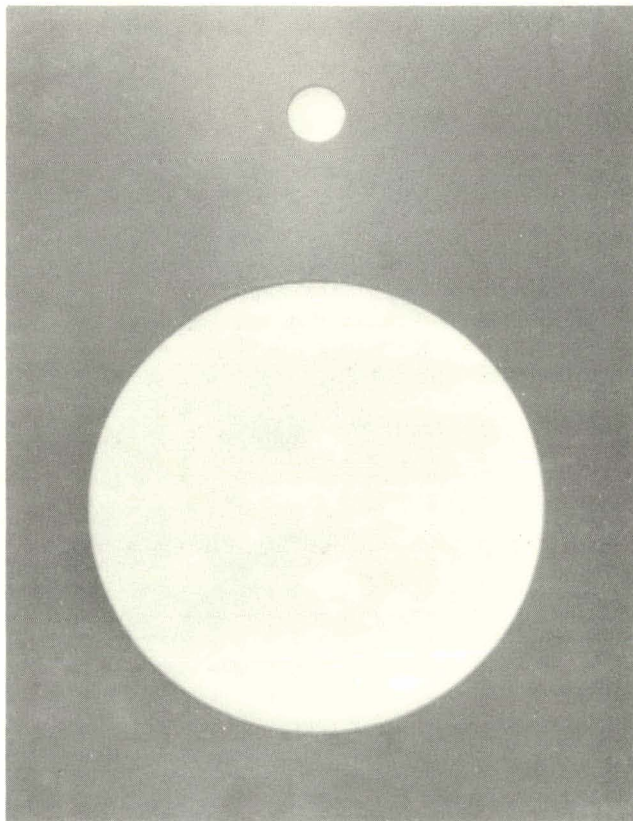
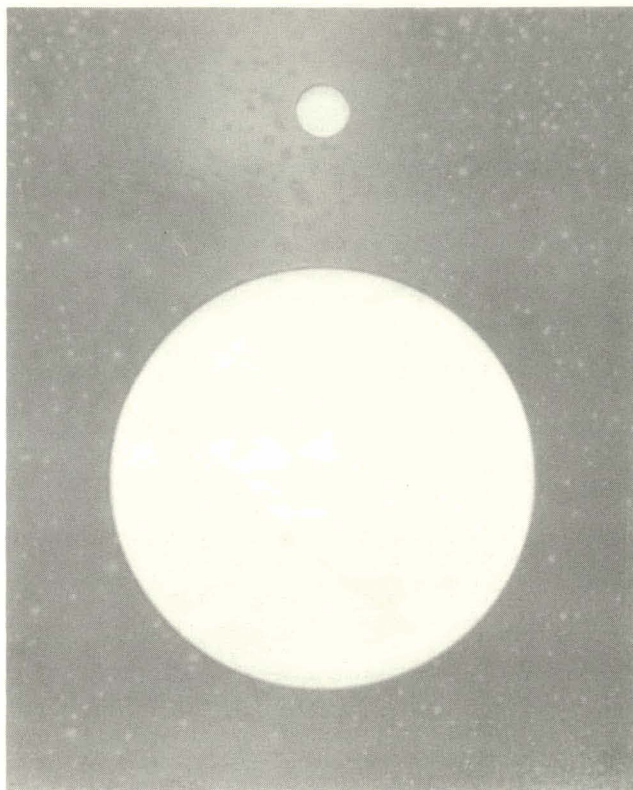


FIGURE 21 - Metal plate coated by the spray technique.

Curve 585907-A

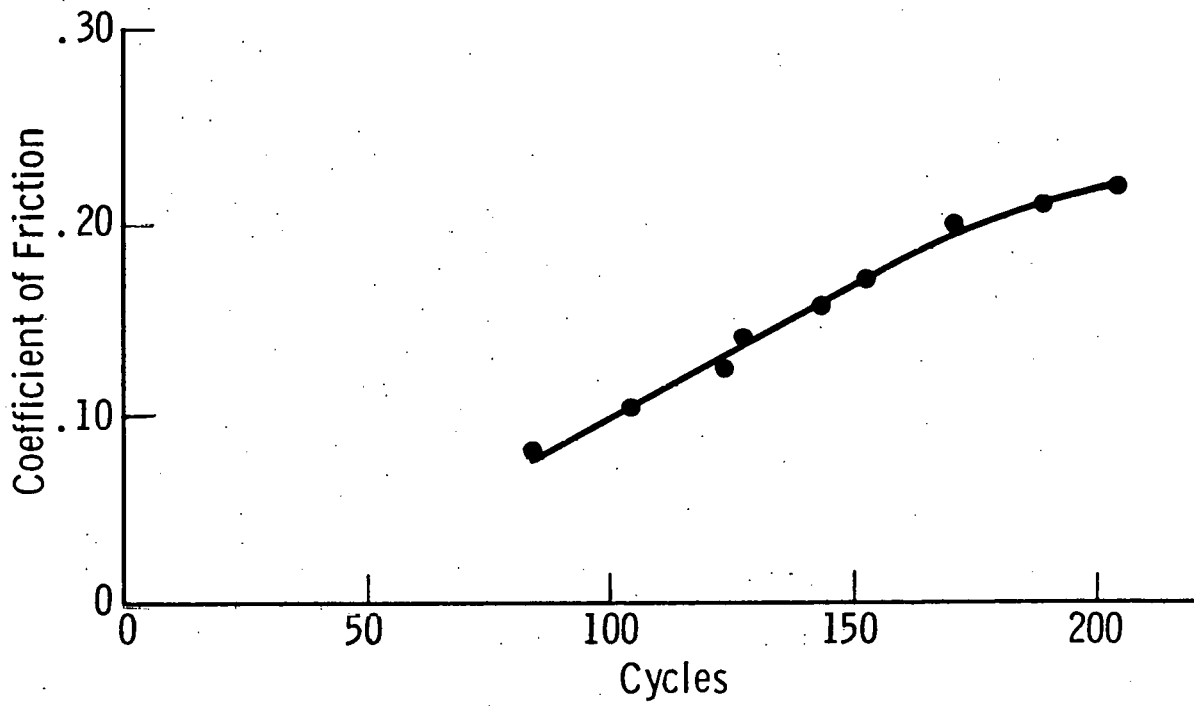
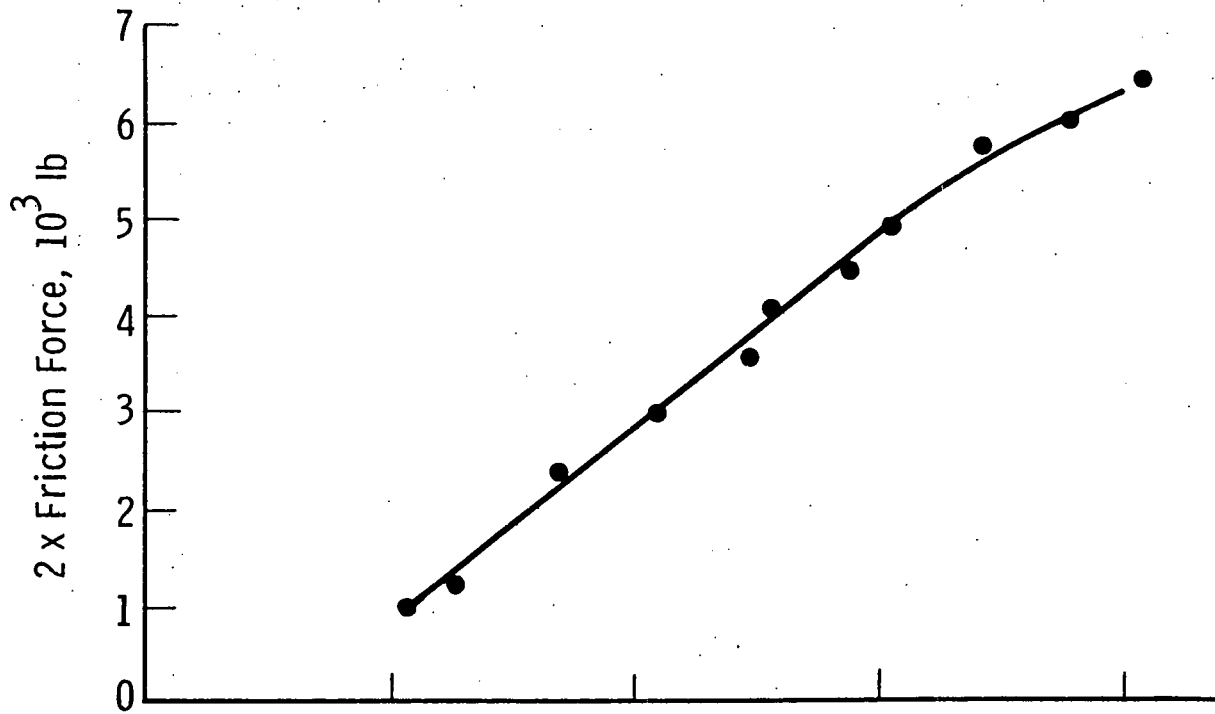


Fig. 22-I-7 resin + 10 vol % (19.4 wt %) WSe₂.
Film against film

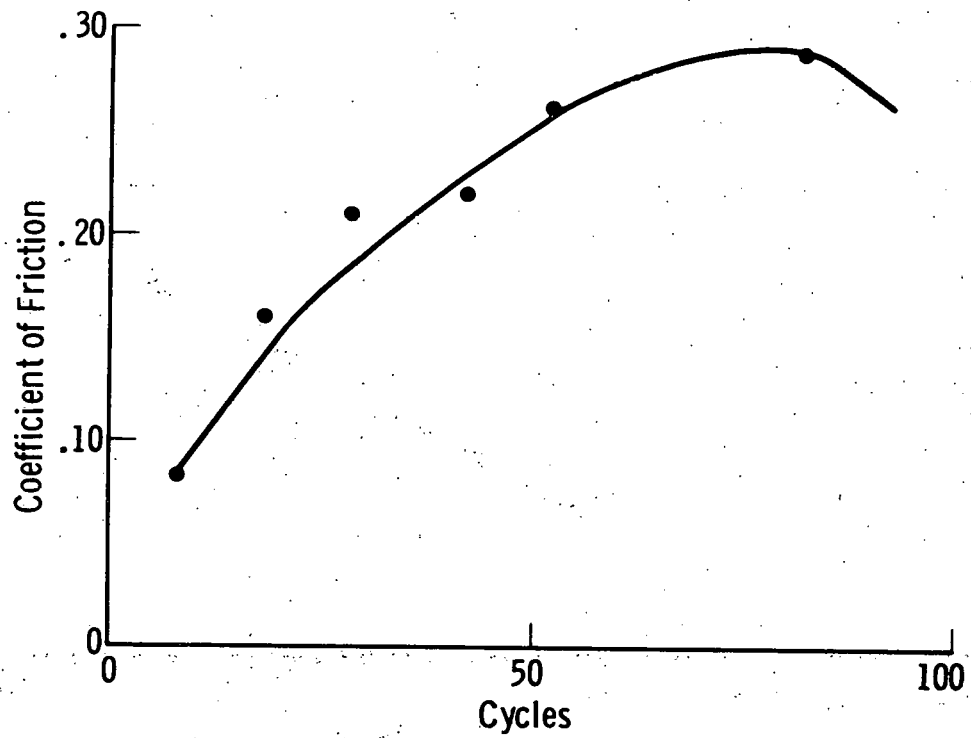
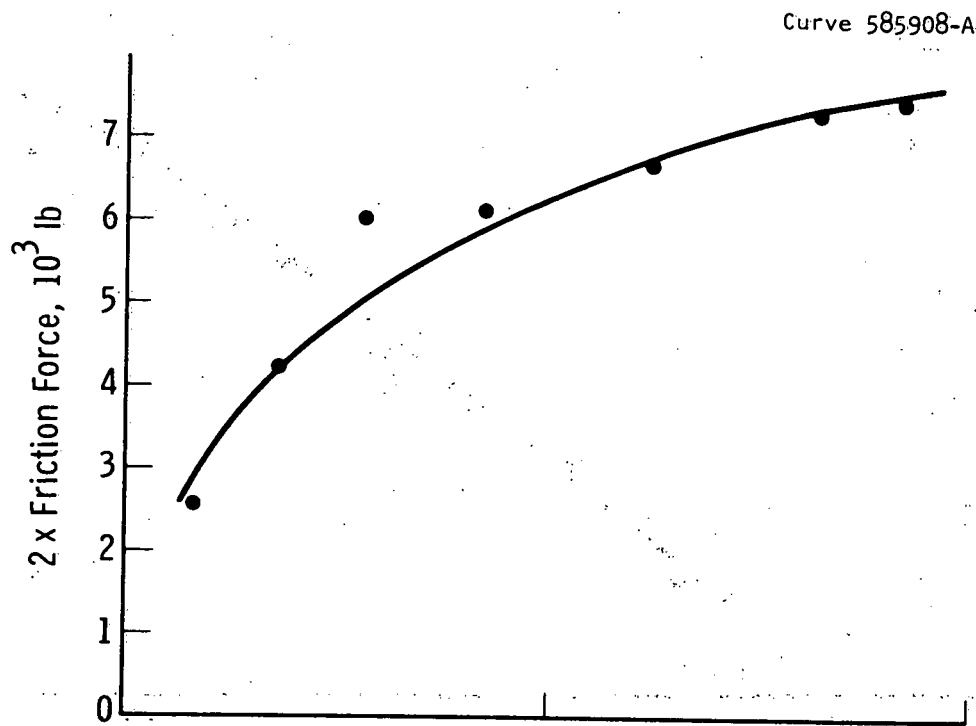


Fig. 23-I-7 resin + 20 vol % (39.4 wt %) WSe_2 .
Film against film

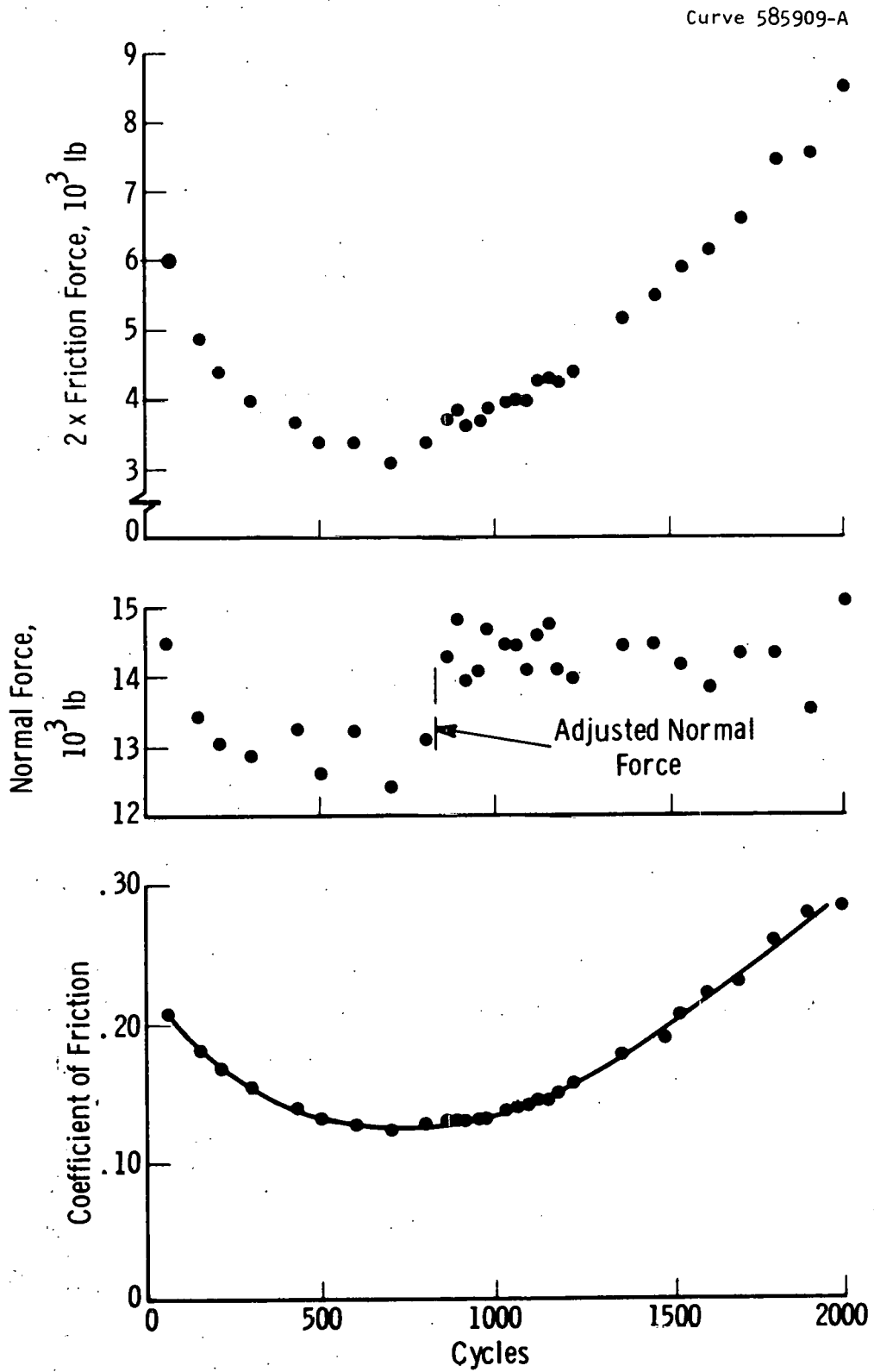


Fig. 24-I-7 resin + 40 vol % (79 wt %) WSe₂. Film against film

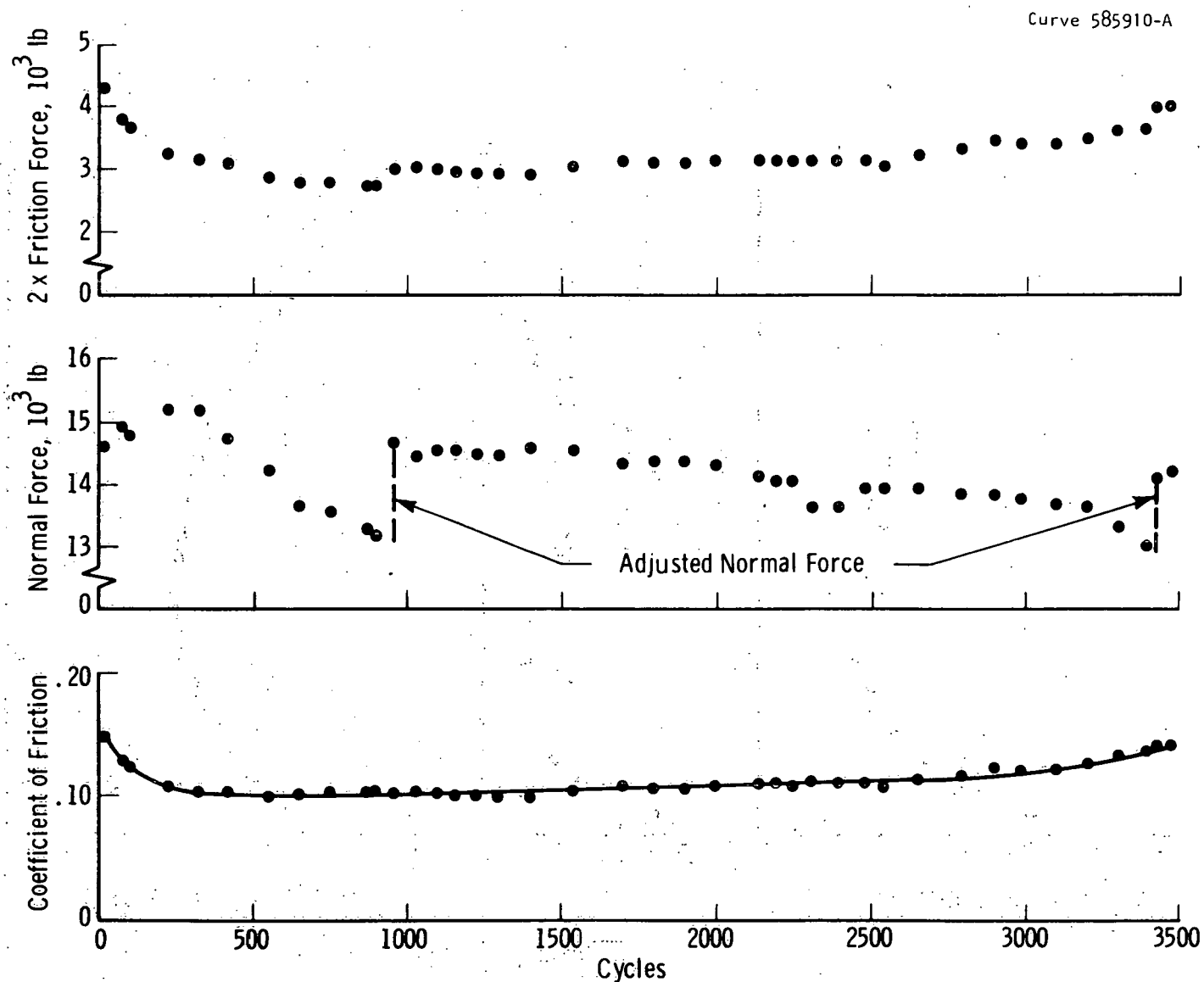


Fig. 25-I-7 resin + 40 vol % WSe₂ Film against bare metal

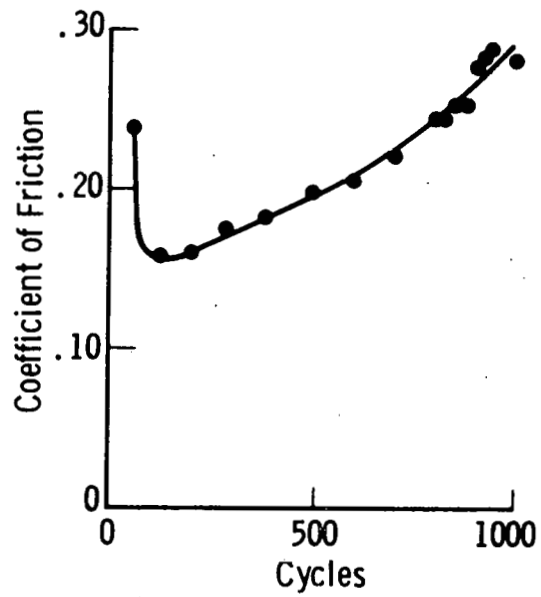
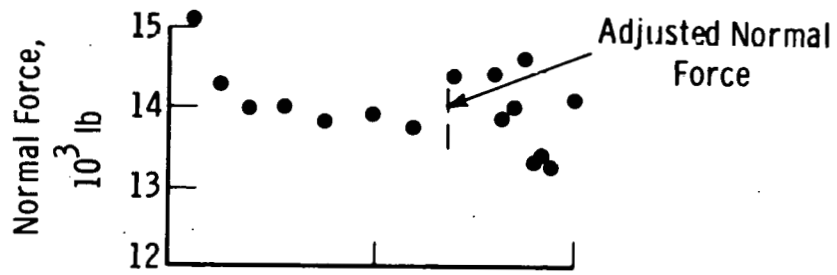
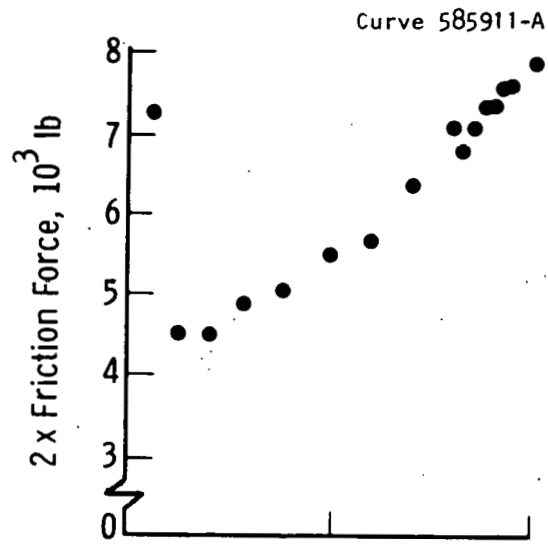


Fig. 26-I-8 resin + 10 vol % WSe_2 .
Film against film

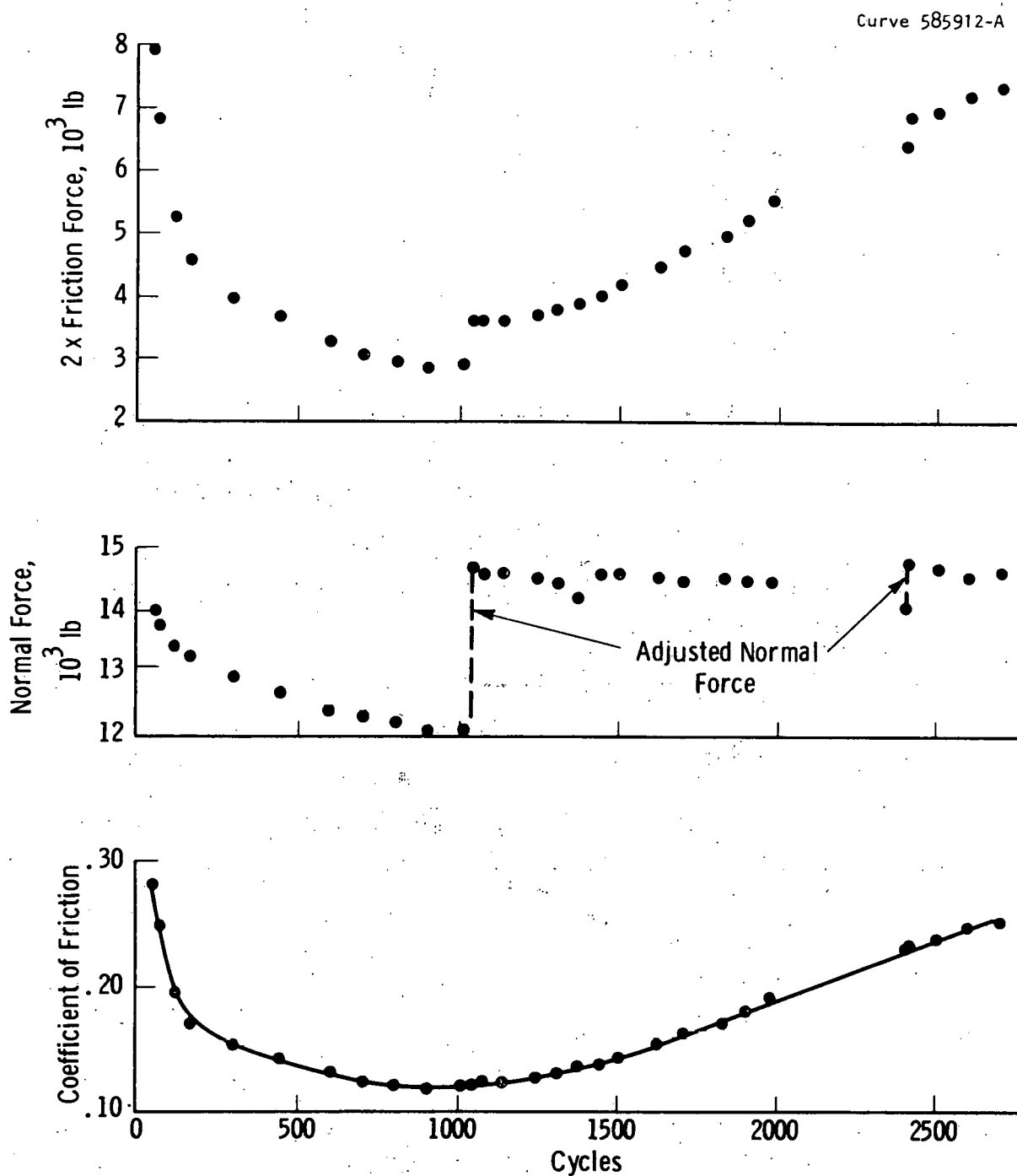


Fig. 27-I-8 resin + 20 vol % WSe₂. Film against film

Curve 585913-A

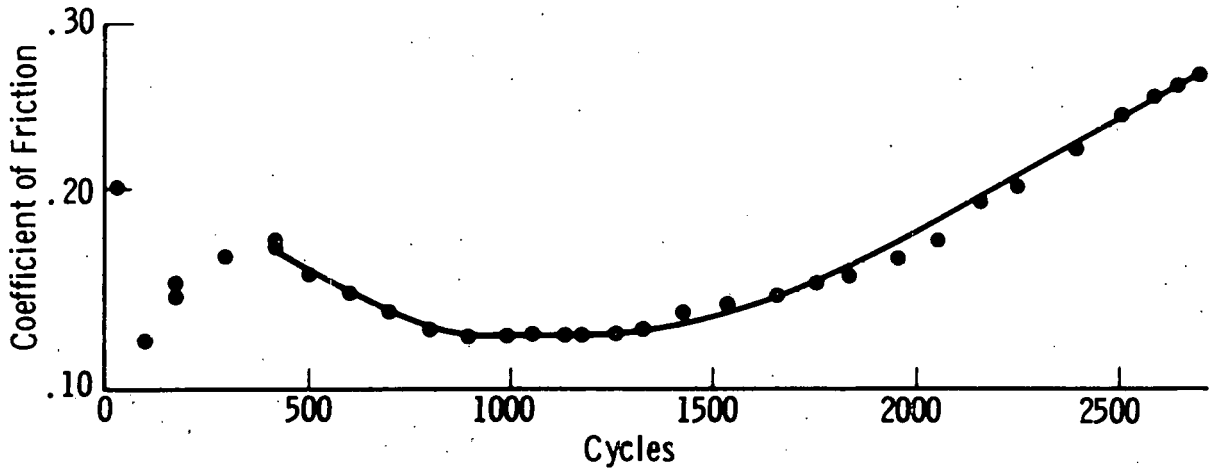
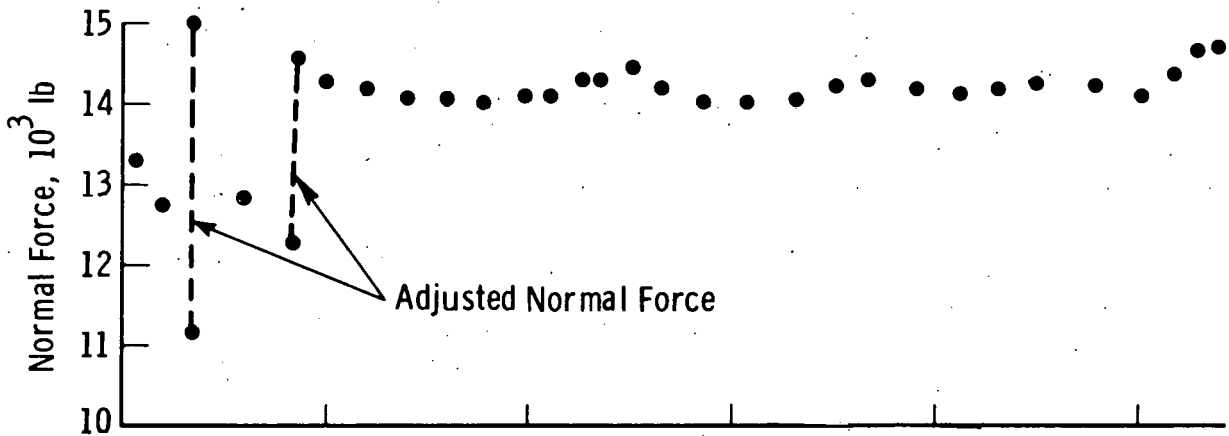
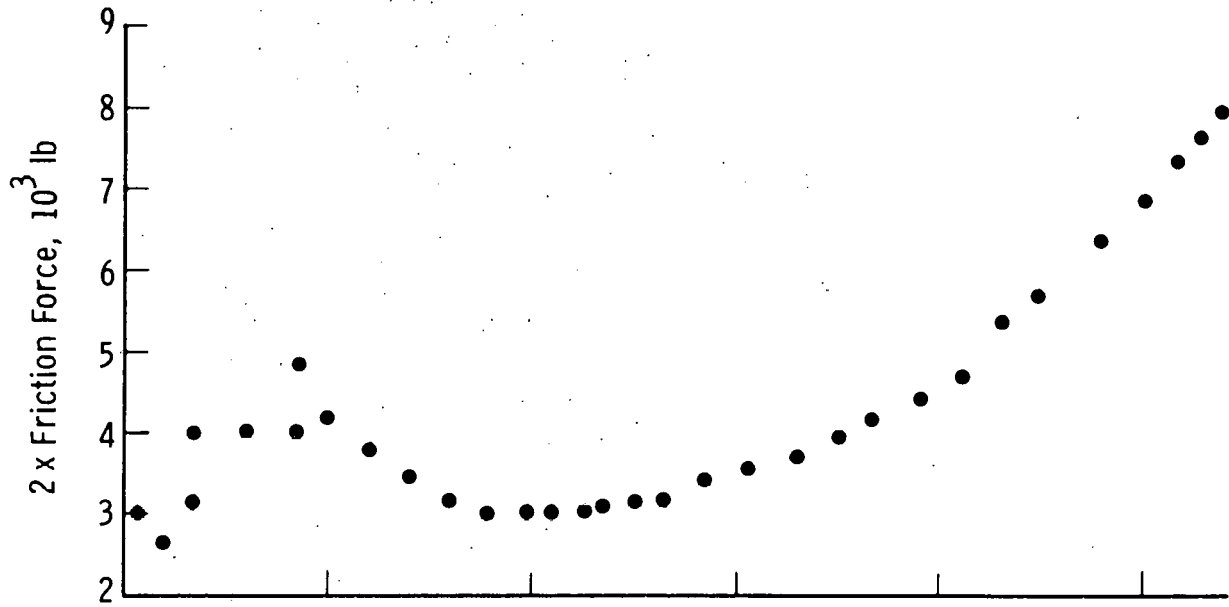


Fig. 28-I-8 resin + 40 vol % WSe₂. Film against film

Curve 585914-A

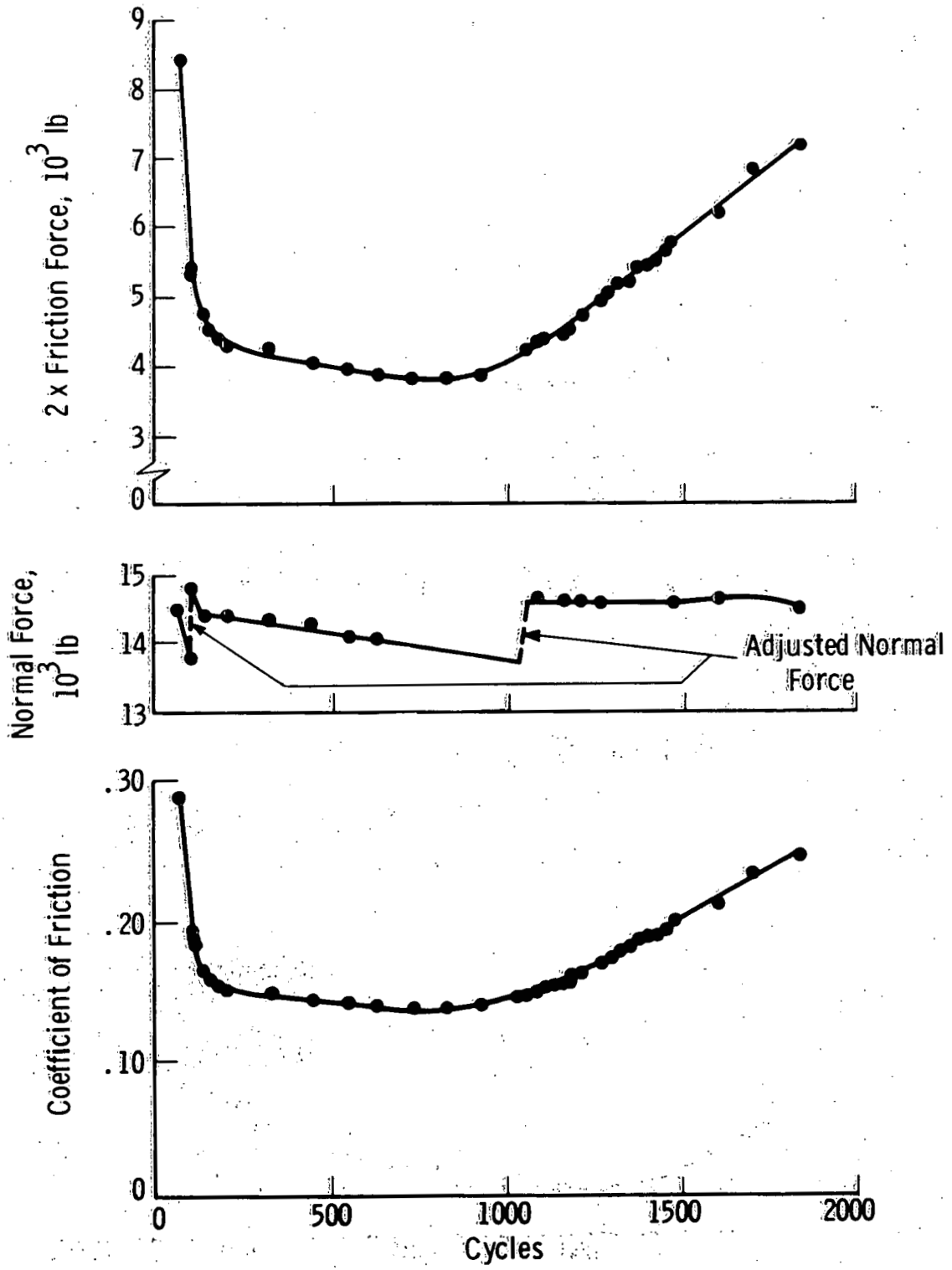


Fig. 29-AI-131 resin + 10 vol % WSe₂ Film against film

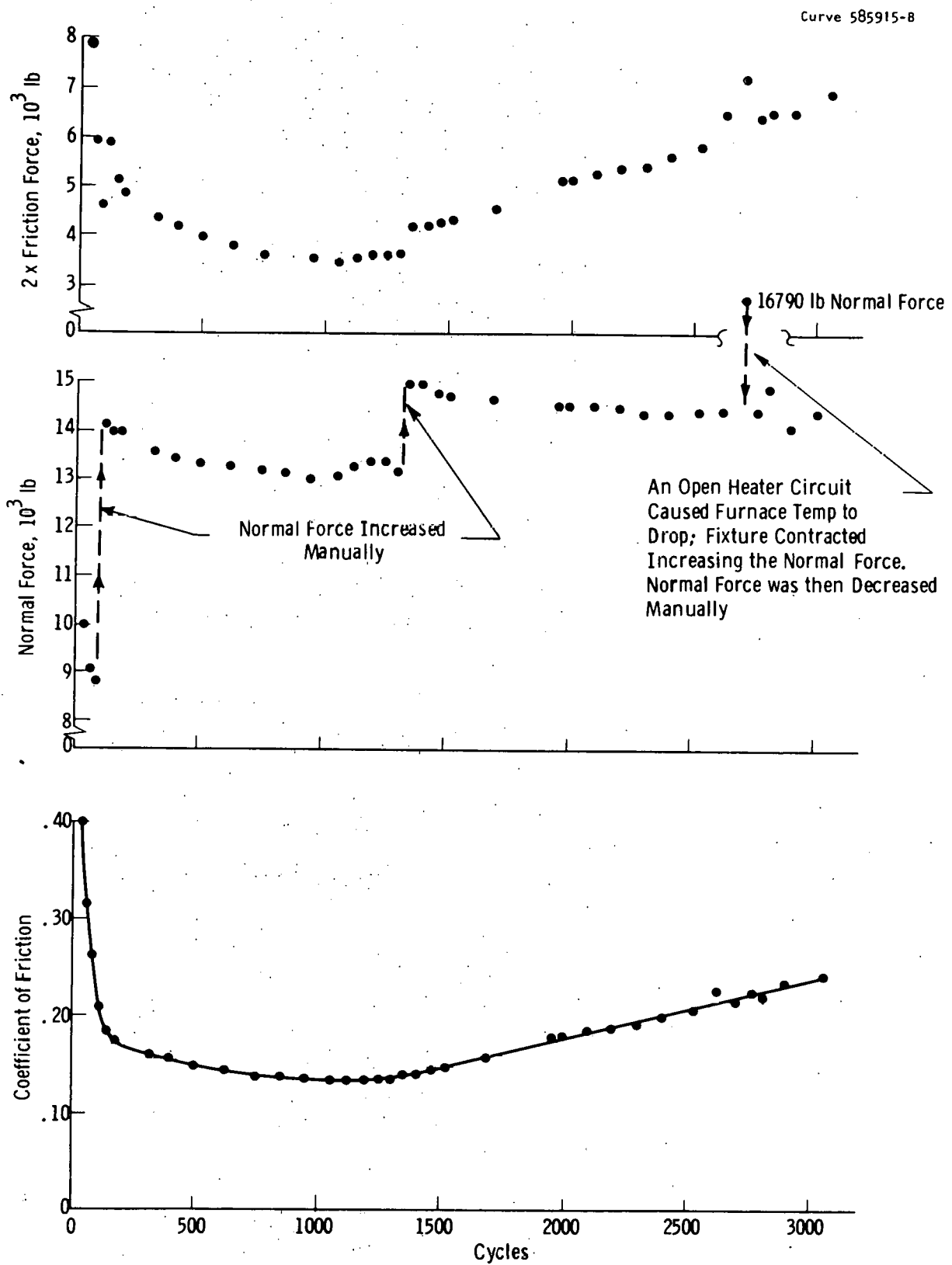


Fig. 30—AI-131 resin + 20 vol % WSe₂. Film against film

Curve: 585916-B

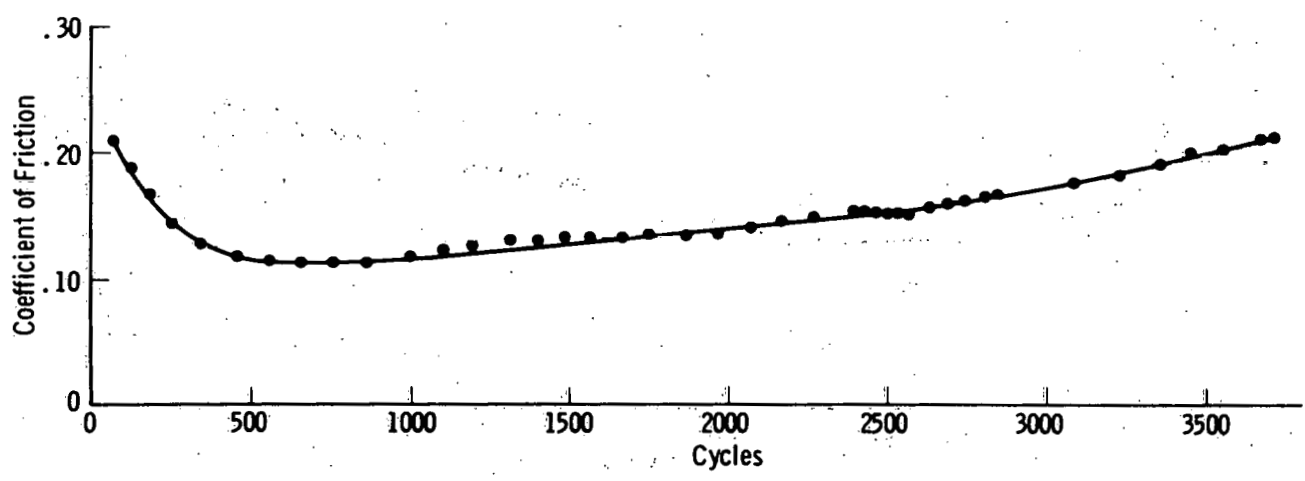
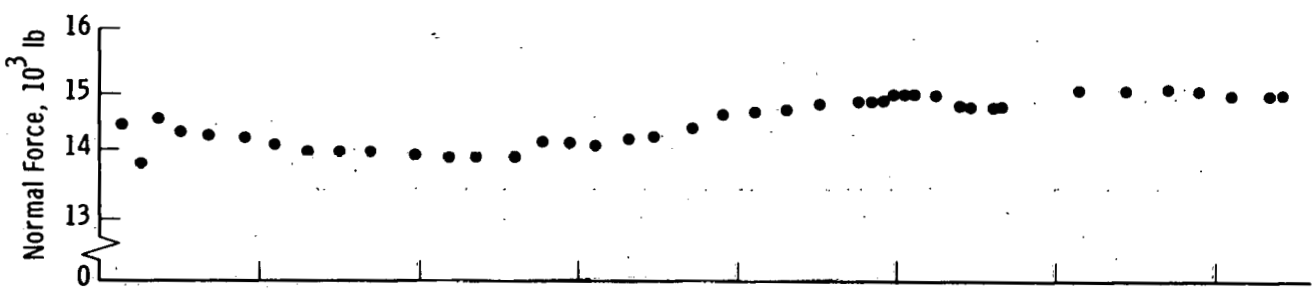
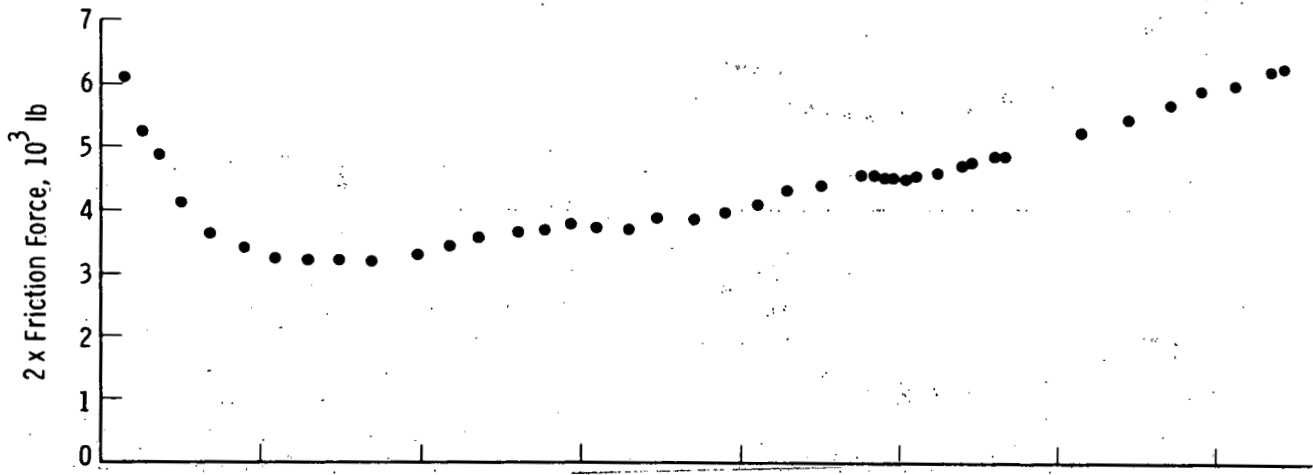


Fig. 31-AI-131 resin + 40 vol % WSe₂ Film against film

Curve 585917-A

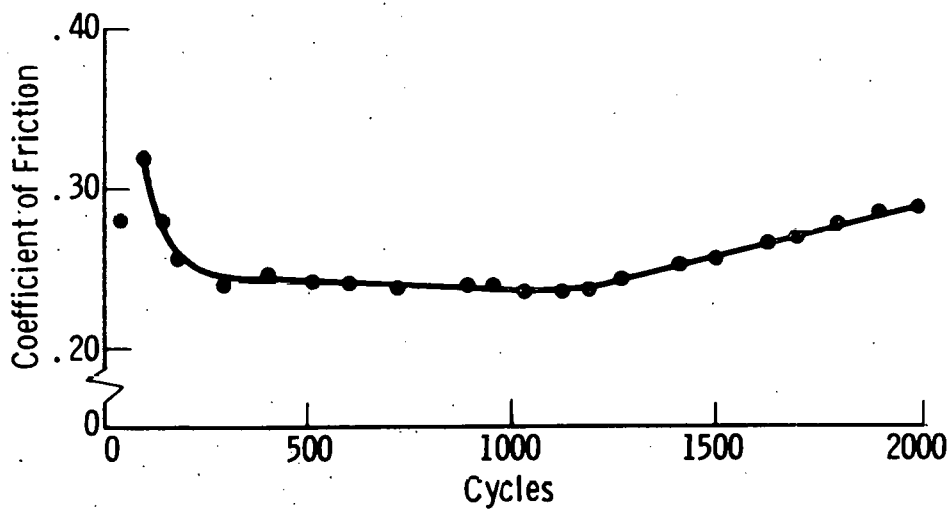
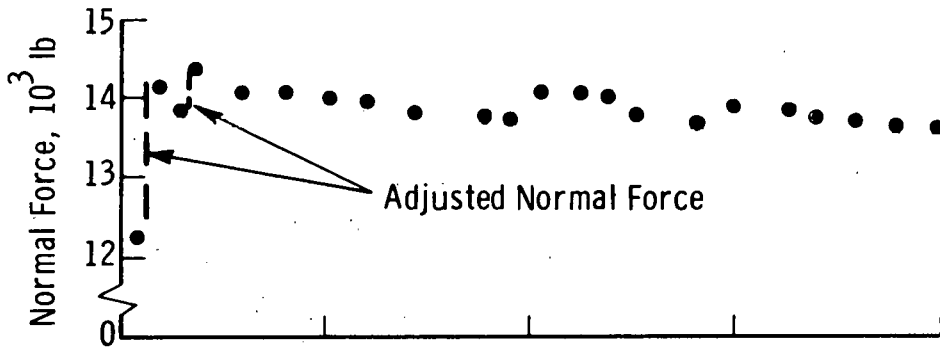
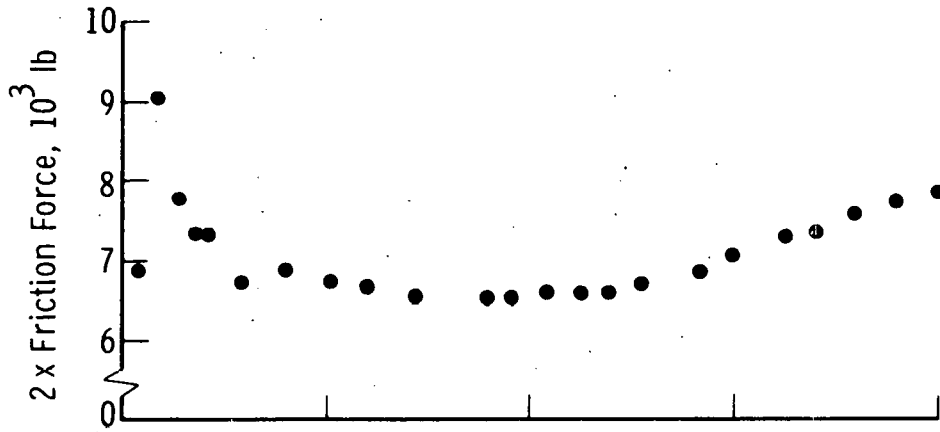


Fig. 32-I-7 resin + 20 vol % WSe₂. Samples exposed 30 days at 100% RH and 150°F. Film against film

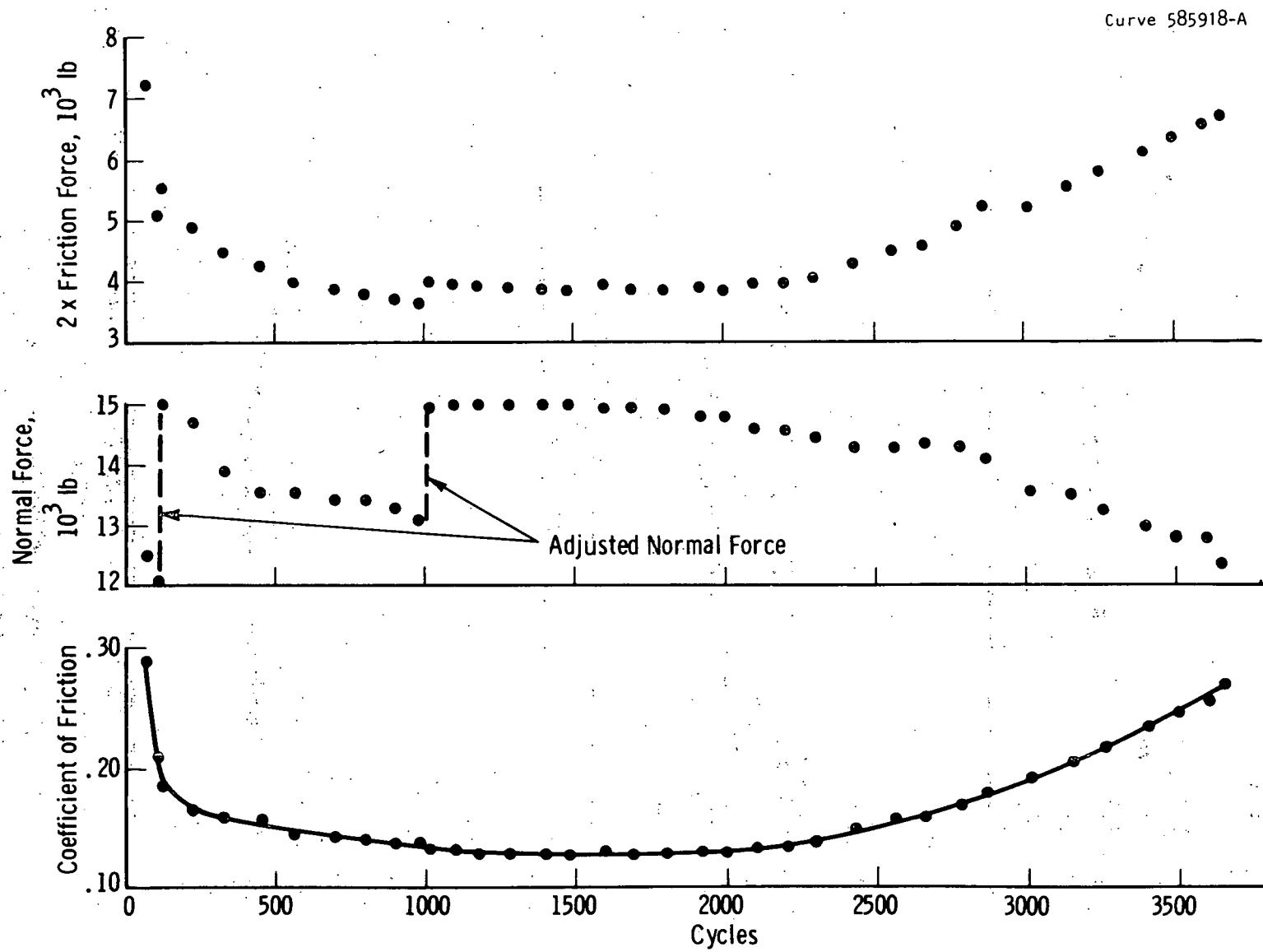


Fig. 33-AI-131 resin + 20 vol % WSe₂. Samples exposed 30 days at 100% RH and 150°F. Film against film

Curve 585919-B

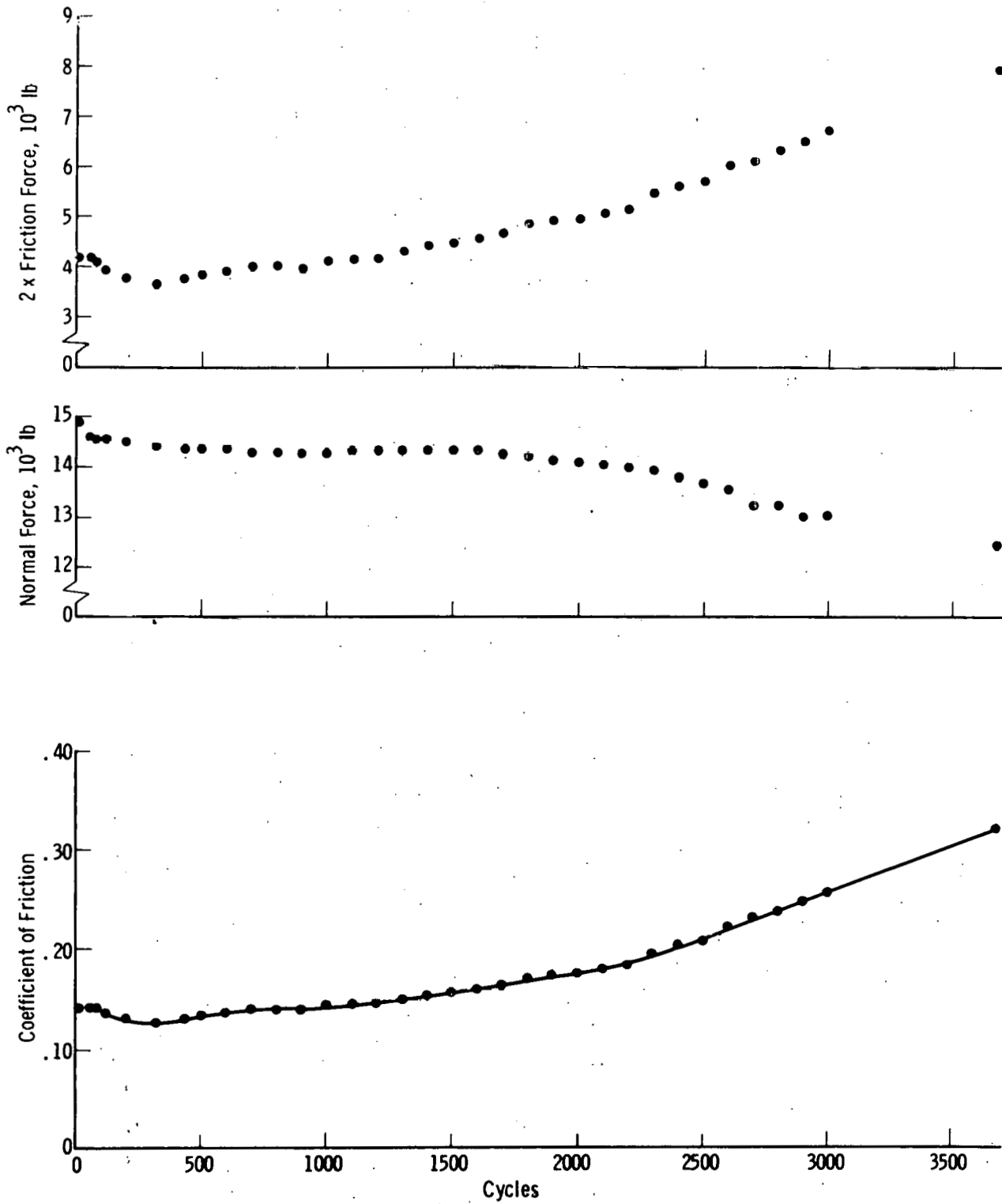


Fig. 34-AI-131 resin + 40 vol % WSe₂. WSe₂ powder rubbed in on sample surfaces. Film against metal

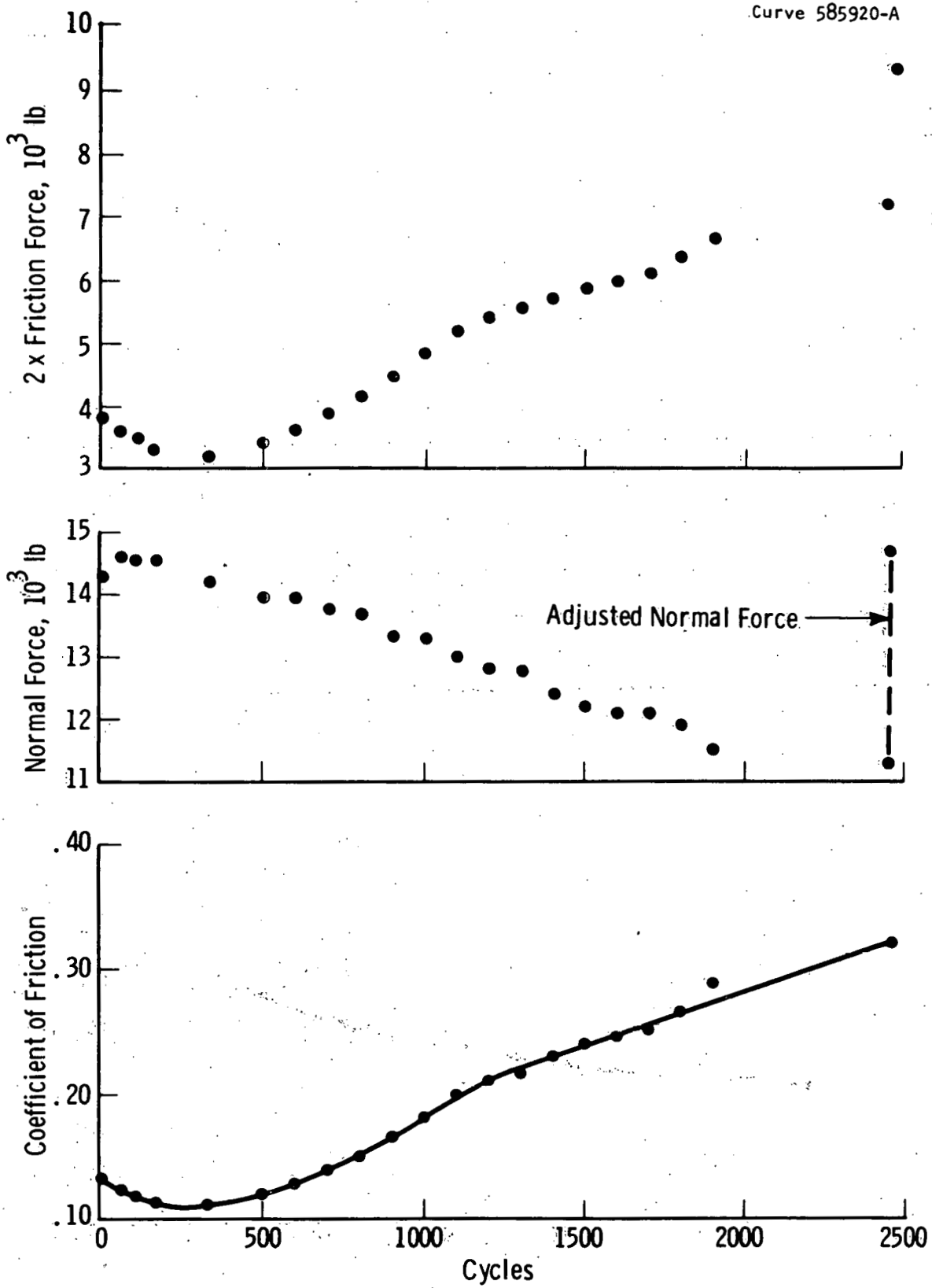


Fig. 35-AI-131 resin + 40 vol % WSe_2 . Film against metal

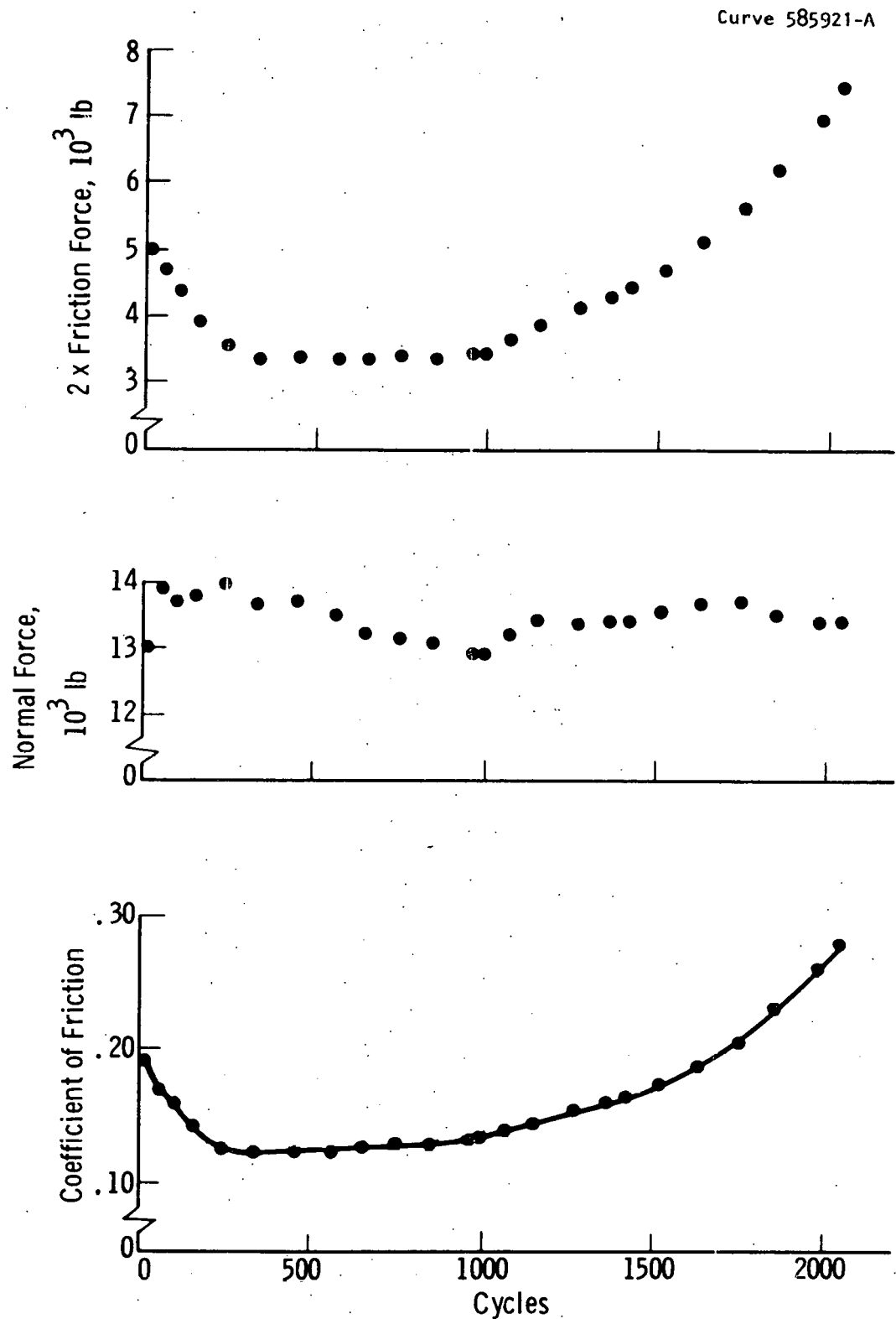


Fig. 36-AI-131 resin + 90 wt % WSe₂.
Film against metal

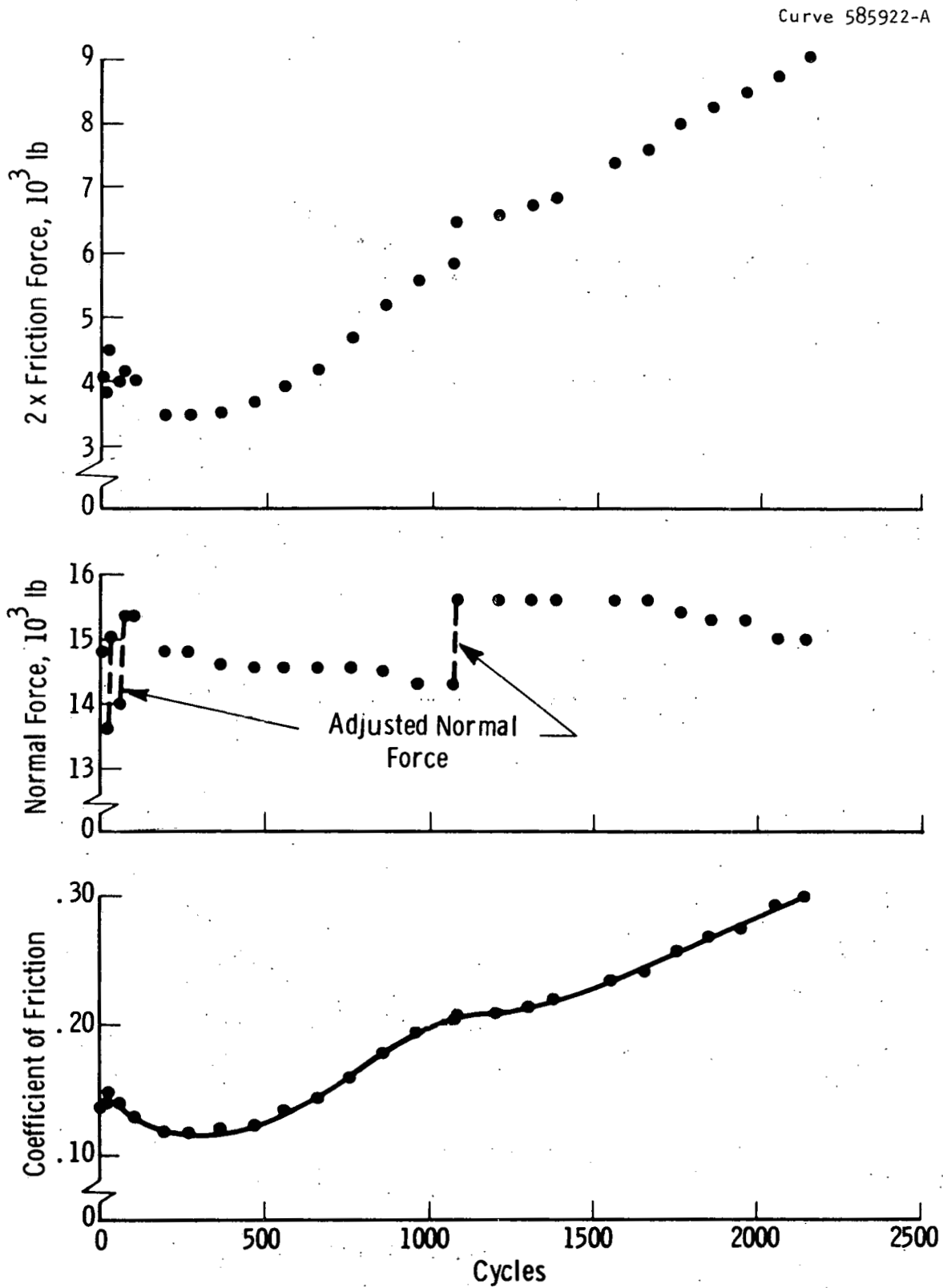


Fig. 37-AI-131 resin + 90 wt % WSe₂. Film against film

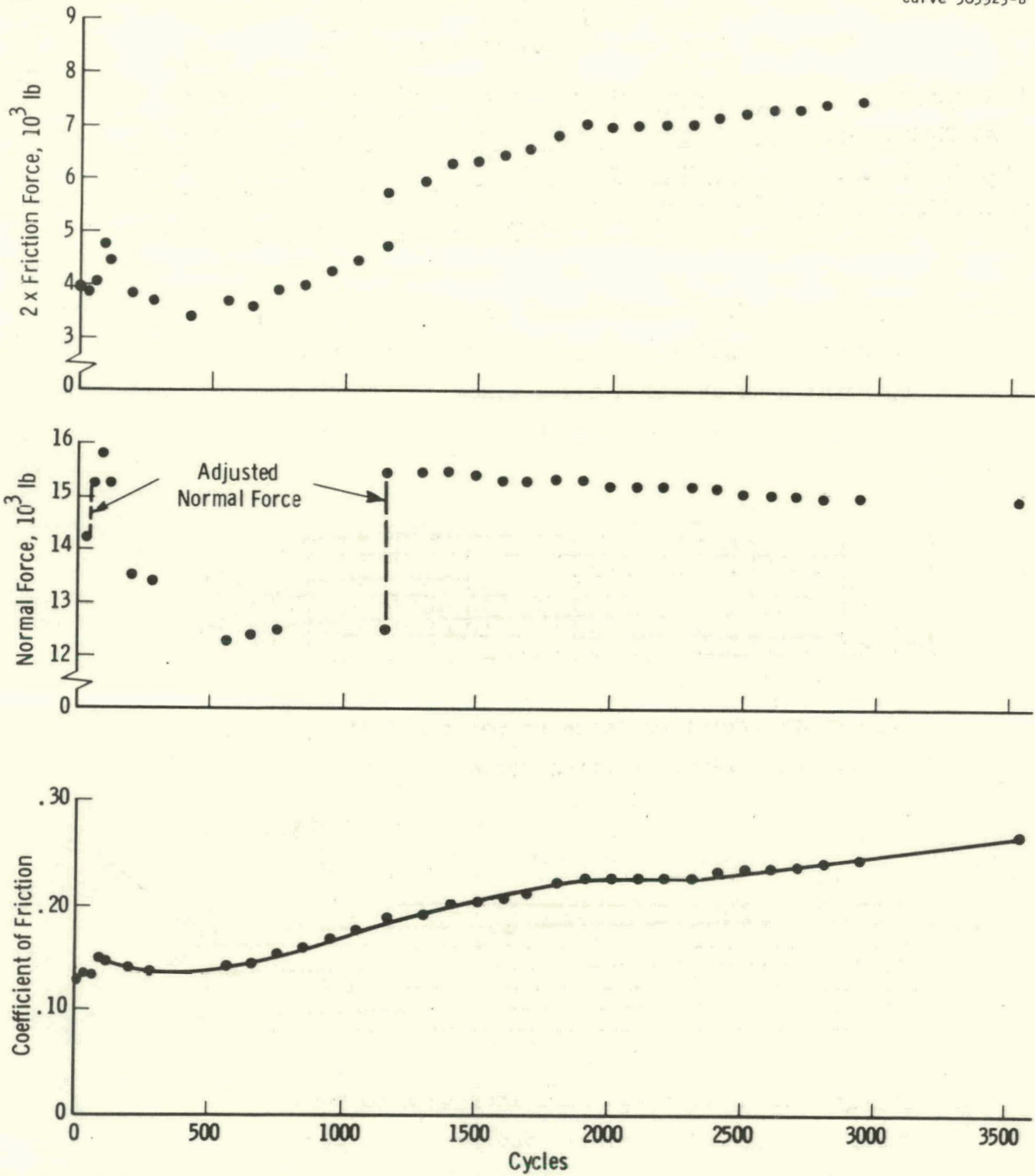
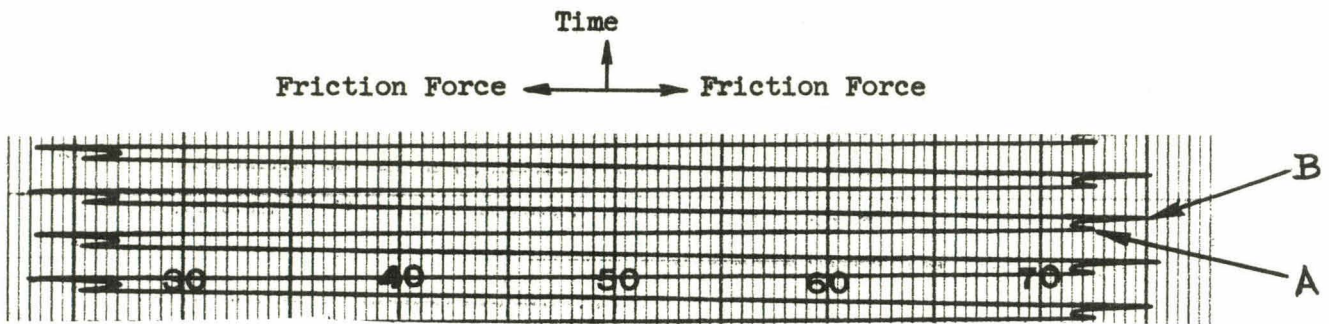
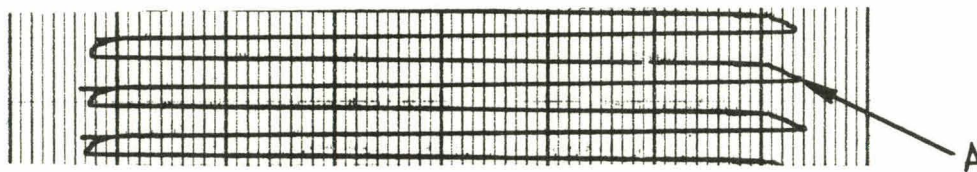


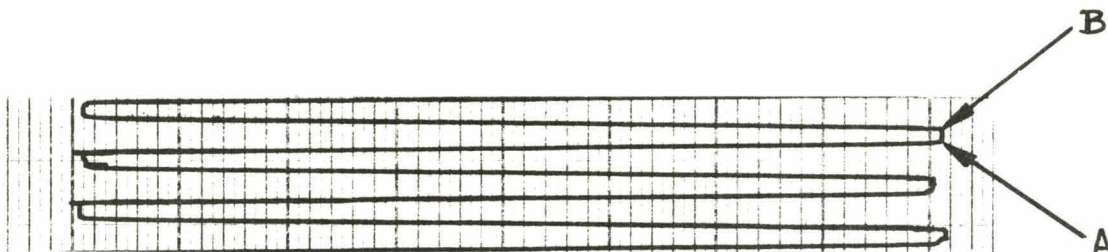
Fig. 38-AI-131 resin +40 vol % WSe₂. WSe₂ powder rubbed in on sample surfaces.
Film against film



- c - High static friction force at point A followed by a decreasing sliding friction force and rising again to point B as abrasion takes place.



- b - High static friction force at point A followed by a decreasing sliding friction force.



- a - Steady sliding action. Load increases to point A where sliding motion starts and continues at the same load to point B. Here the sliding motion stops and the testing machine reverses direction.

Fig. 39- Examples of friction force records showing three types of sliding mechanisms.

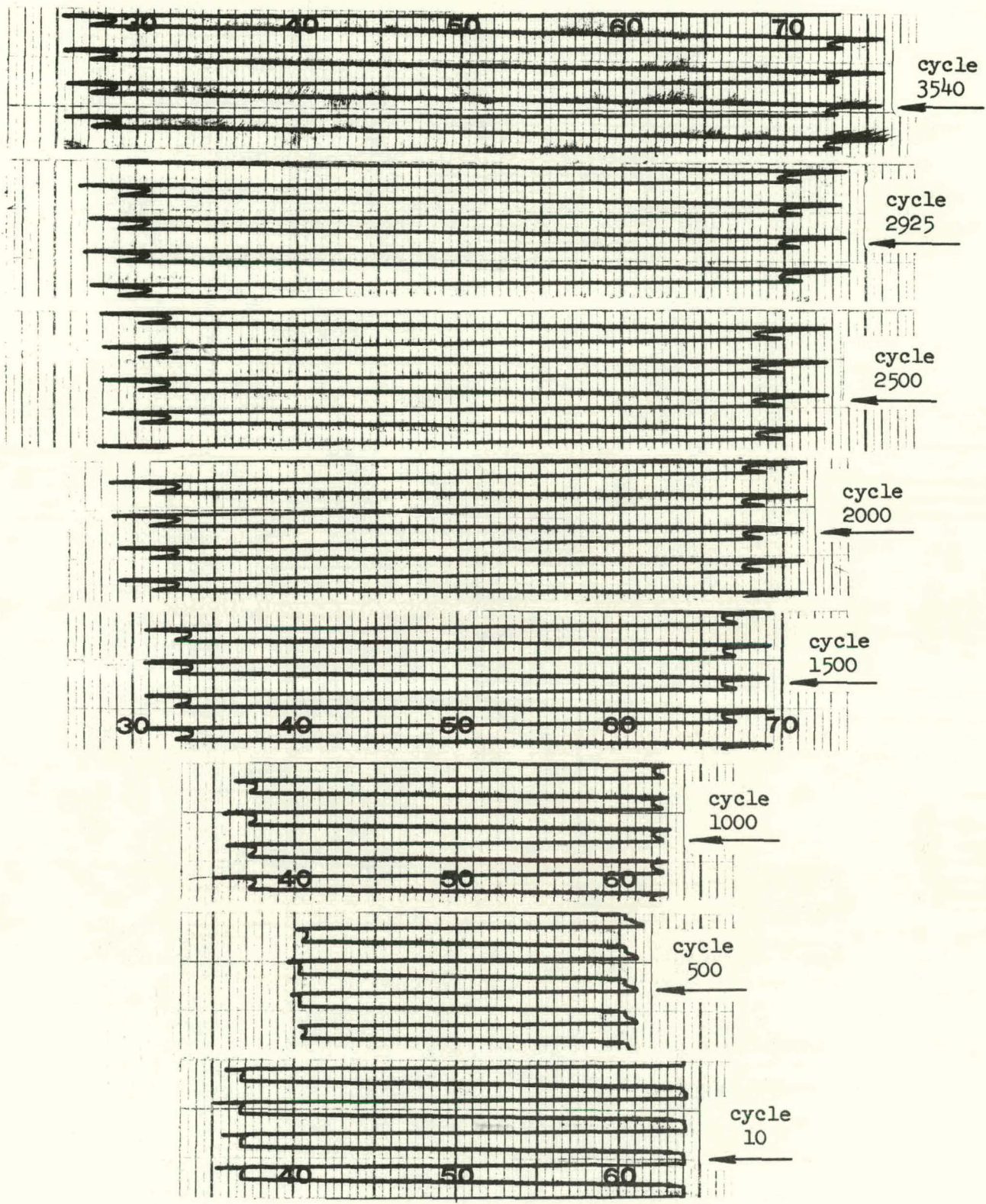


Fig. 40 - Sections of friction force record. AI-131 resin with 40% filler by volume, film on film with WSe_2 rubbed in on film surfaces.

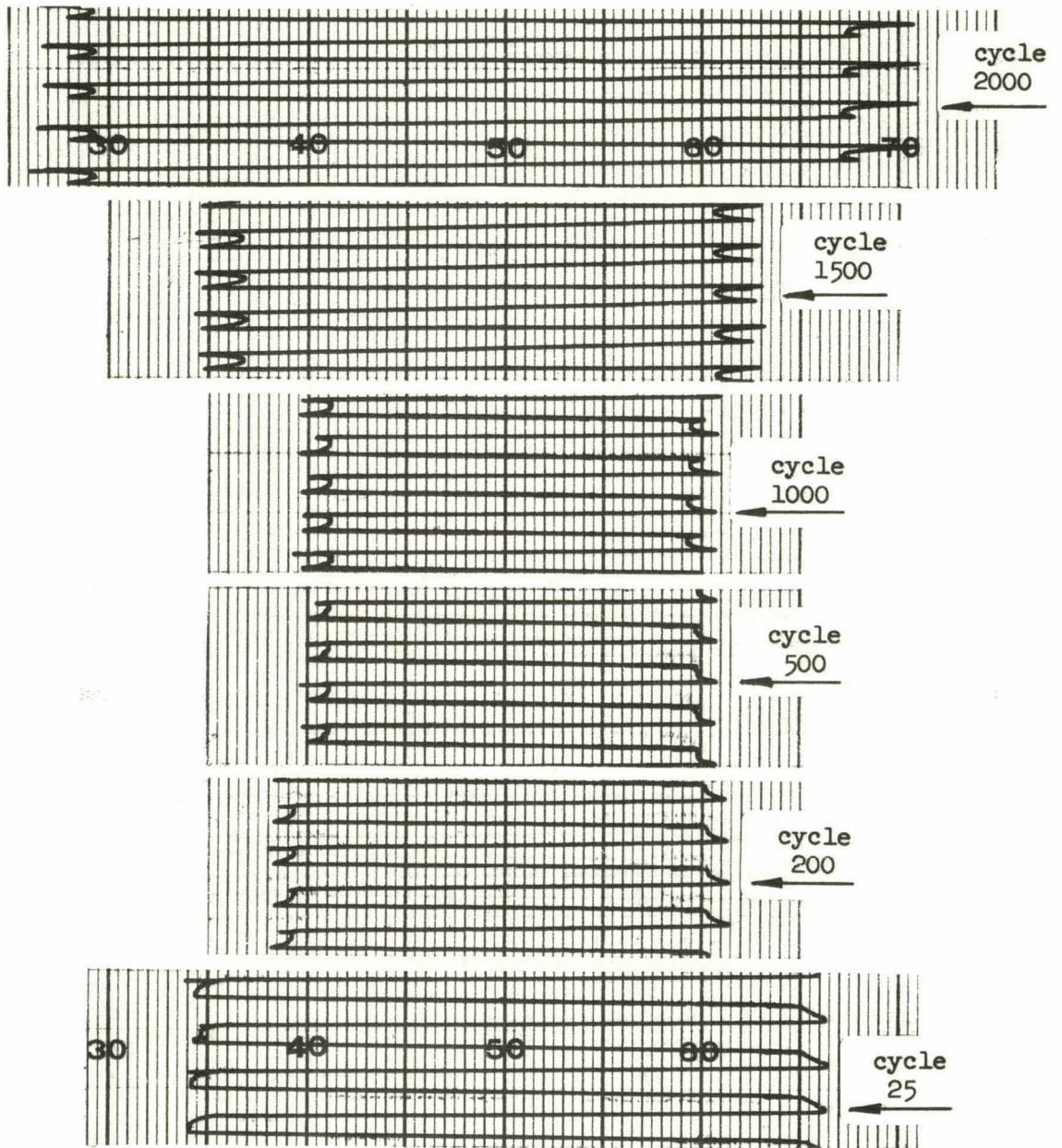


Fig. 41- Sections of friction force record. AI-131 resin with 90% filler by weight, film on bare metal.

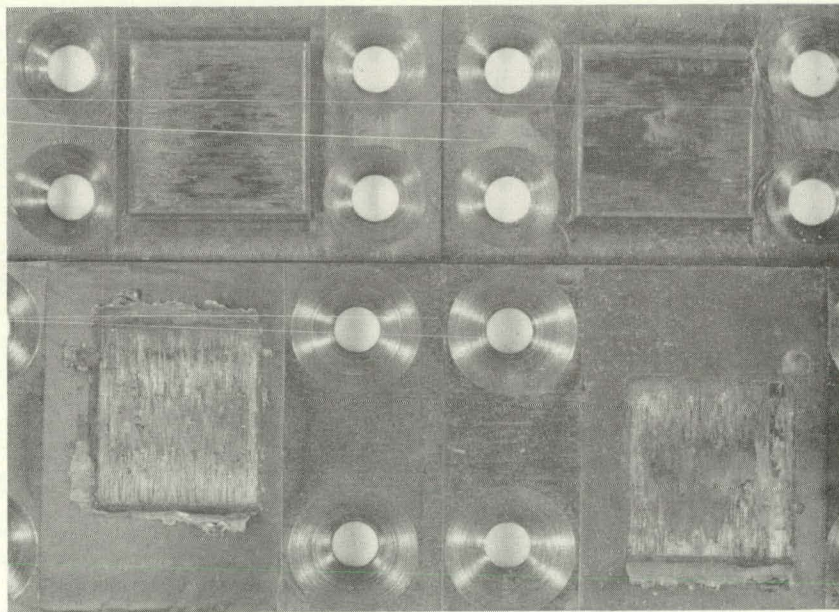
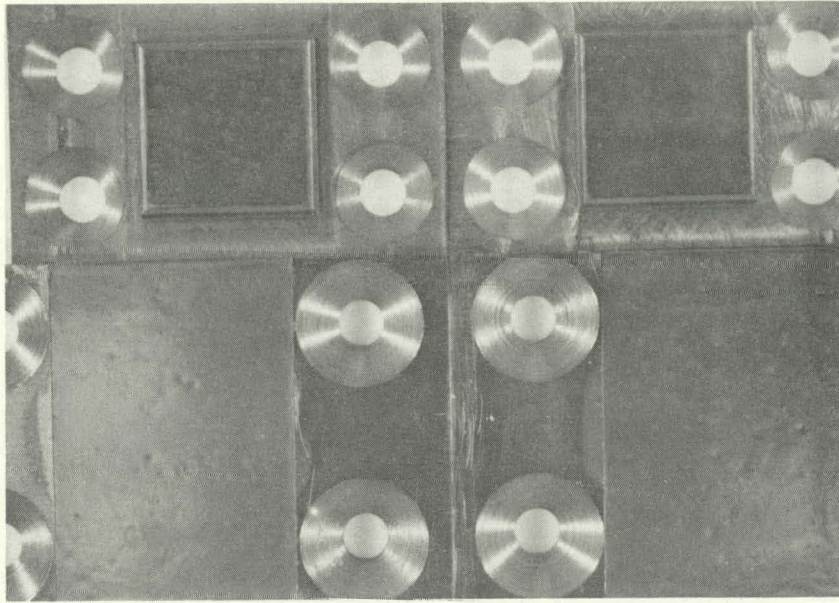


Figure 42 Irradiation Samples (AI-131 + 40 vol % WSe_2) before and after friction testing. Film against film.

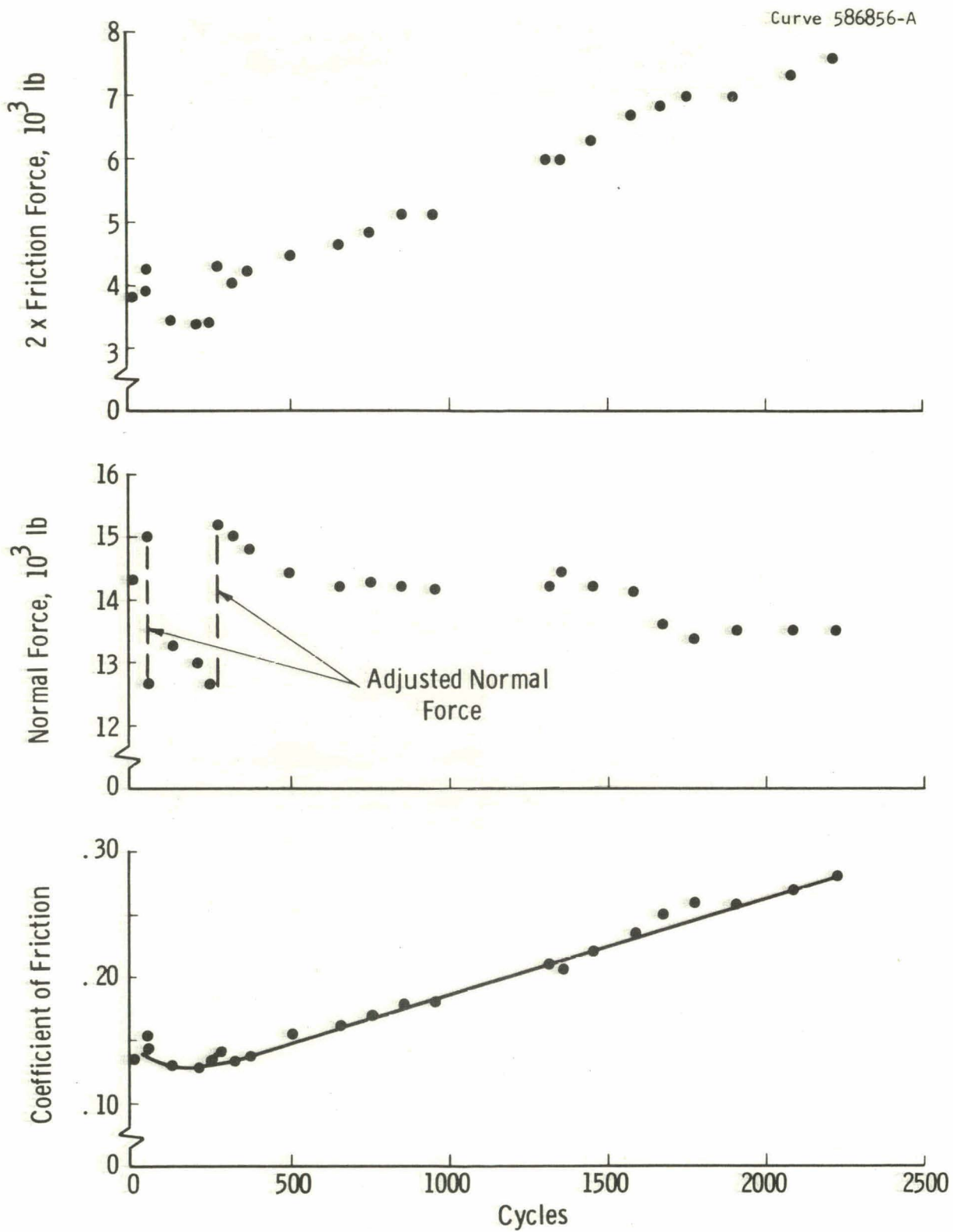


Fig. 43-AI-131 resin + 40 vol % WSe₂. Film against film (irradiated samples)

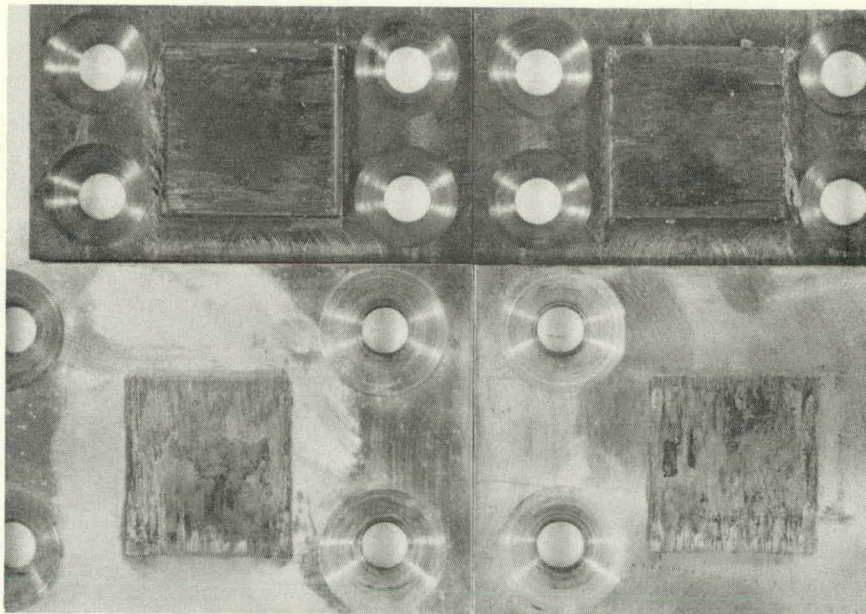
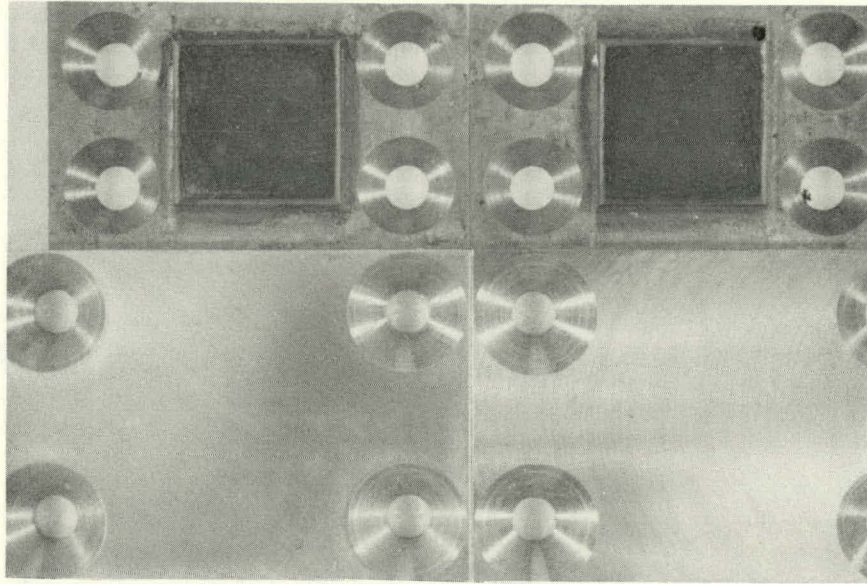


Figure 44 Inconel friction test samples before and after testing (AI-131 + vol % WSe_2). Film against bare Inconel.

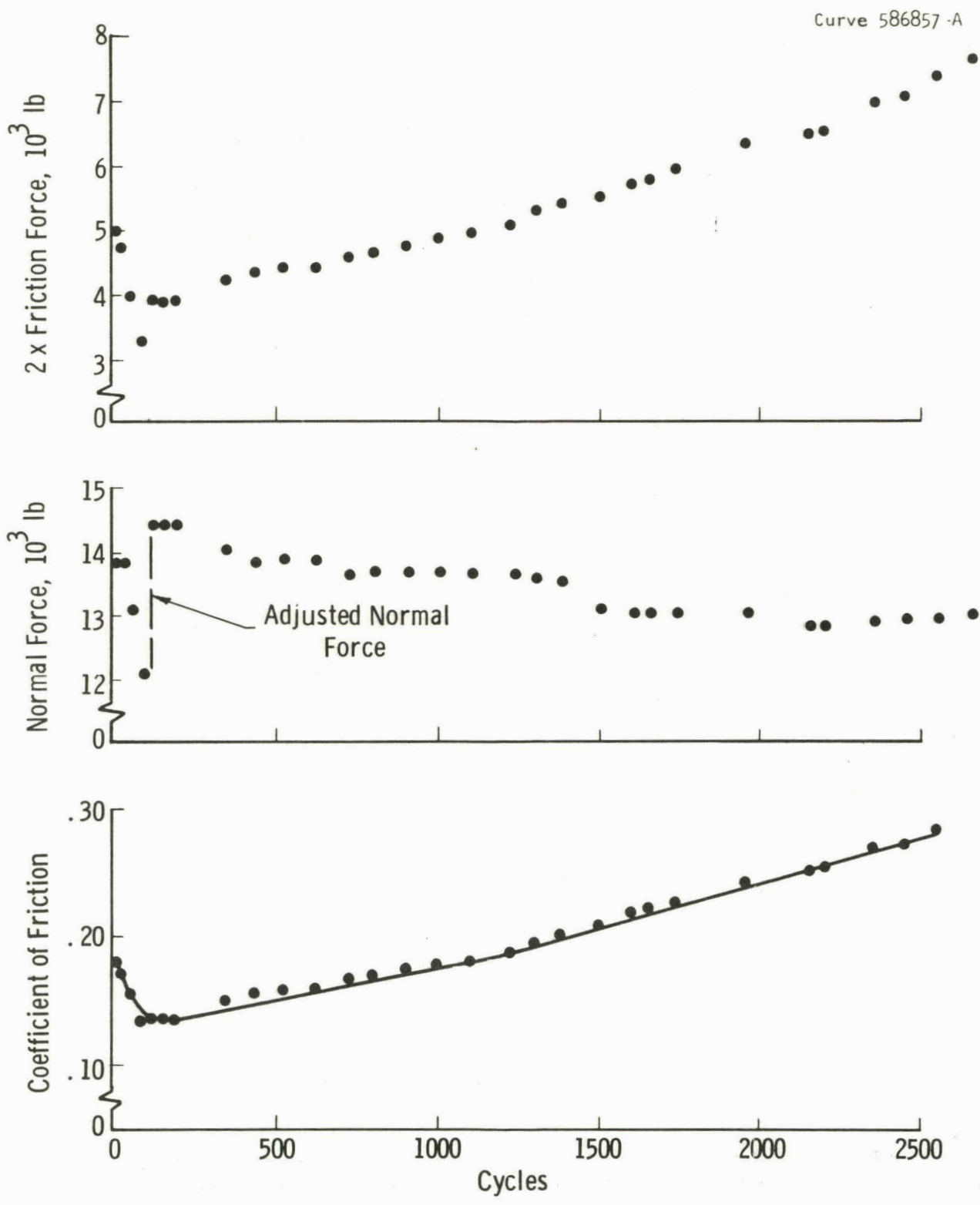


Fig. 45-AI-131 resin + 40 vol % WSe₂. Film against bare Inconel

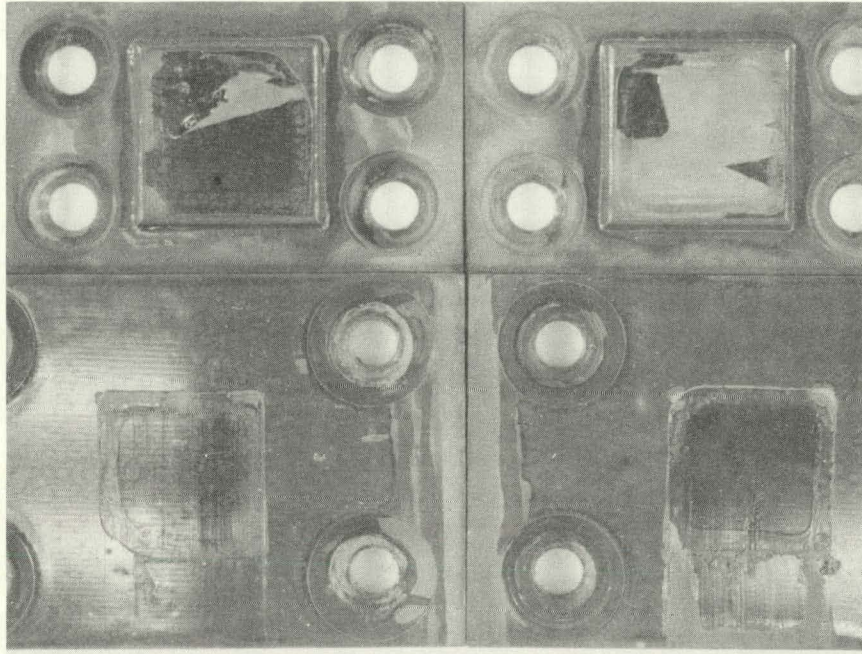


Figure 46 Static Load test sample after 90 days exposure at 550°F under 15,000psi (Al-131 + 40 vol % WSe₂).

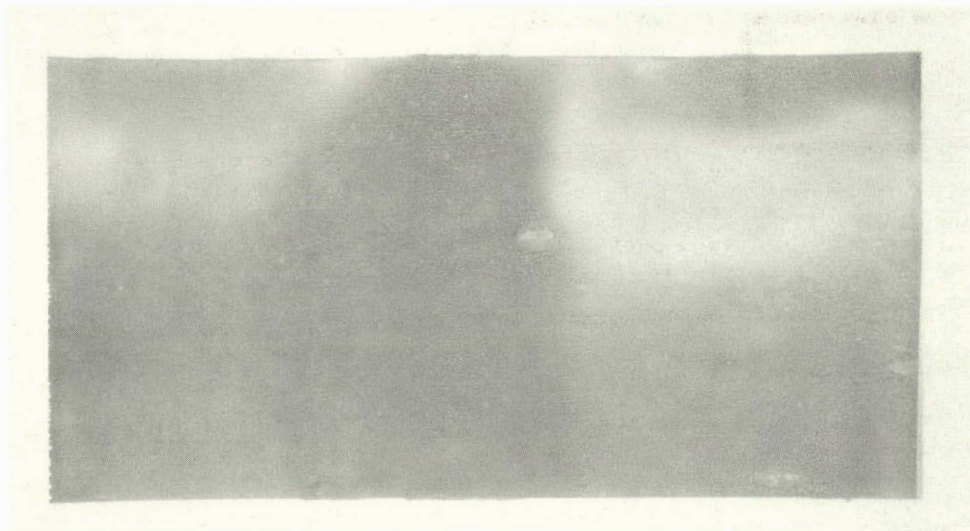


Figure 47 Illustration of free film (top) and the stainless steel coated shim (bottom). (AI-131 + 40 vol % WSe_2).

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DEVELOPMENT OF POLYIMIDE BONDED SOLID FILM LUBRICANTS -
W. M. Alvino and G. E. Rudd

Keywords - Imides, amides, films, lubricants, radiation, humidity, friction, coatings, fillers, properties.

The results of work conducted to develop heat and radiation resistant organically bonded solid film lubricants are described. These solid film lubricants are composed of an organic resin binder (polyimide and amide-imide resins) and several transitional element compounds such as the selenides of tungsten, molybdenum, niobium and tantalum. The approach, the data and the procedures necessary to mix, apply and cure these films are (over)

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outlined. Friction and wear properties of these films at elevated temperatures (550°F) under high loading (15,000 psi) in addition to those parameters affecting the aforementioned properties such as, resin type, filler and its concentration, film thickness, test surface, radiation, long term static bonding are also presented.

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A polyamide-imide resin designated as AI-131 and loaded with 40 volume or 79 weight percent WSe₂ was found to give the best combination of friction and wear properties under the severe conditions of the test. This composition is unaffected by humidity, gamma radiation and does not bond significantly under conditions of long-time heat and pressure. Coefficients of friction between 0.13-0.27 have been maintained under test conditions for 3700 sliding motion cycles. Procedures for applying this coating composition onto large metal plate sections using either the dry bar or spray coating technique are also described.

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