Chemistry Support Services

Final Report
Report No. 951581-9

PRELIMINARY EVALUATION OF A THIN ORGANIC FILM COATING

Prepared for:

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA 91103

Attention: L. R. TOTH, TECHNICAL REPRESENTATIVE

CONTRACT 951581
UNDER NAS7-100
(TDM 68 X 08700)

STANFORD RESEARCH INSTITUTE
Menlo Park, California 94025 · U.S.A.

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By: N. H. G. DANIELS and M. C. GARRISON

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SRI Project PRD-6063

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FOREWORD

This report was prepared by Stanford Research Institute under JPL Contract Number 951581, TDM 68X 08700 for Jet Propulsion Laboratories. The work was carried out by N. H. G. Daniels and M. C. Garrison under the supervision of E. S. Wright, Manager, Metallurgical Processing with overall supervision by M. E. Hill, Director of Physical Sciences (Chemistry).

L. R. Toth is the designated technical representative for JPL and H. E. Patterson the cognizant contract negotiator.

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PRELIMINARY EVALUATION OF A THIN ORGANIC FILM COATING

Introduction

Thin siloxane films applied to a metal substrate by electron beam bombardment techniques[1] appeared to offer some promise as a surface finish having potential application as a dry lubricant or as a protection against natural environments or propellants.

Sixteen coated steel specimens, each approximately ½ in. wide by 3 in. long by 0.112 in. thick, and two blank control specimens were provided by JPL. The coatings had been produced by Systems Groups of TRW, Inc. using a single monomer material, Dow Corning oil DC-704. Details of the preparation and history of the specimens are given in Appendix A[2] which also includes results of evaluation of some similar samples by TRW. The specimens received by SRI were examined by optical and electron microscope techniques. Tests of the frictional characteristics of the coatings and of their resistance to high temperatures and humidities were performed as detailed below.

Preliminary Examination and Selection of Test Groups

The sixteen coated specimens consisted of the following four groups:

Group I	Specimen Nos. 13, 14, 15, 16
Group II	Specimen Nos. 17, 18, 19, 20
Group III	Specimen Nos. 21, 22, 23, 24
Group IV	Specimen Nos. 33, 34, 35, 36

One specimen from each group was to be selected for each of the following evaluation tasks:

A. Examination of "as received" coatings. This task included optical and electron microscope examination, and a film stripping test.

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[1]TRW Systems report 06641-6014-R000, Vol I, Section VI, under NASA Contract 7-436.

[2]Appendix A. Letter from R. J. Salvinski (Systems Group of TRW, Inc.) to L. R. Toth (JPL) dated 9/10/69, refer. 69.4712.5-20.

B. Temperature cycling between 0°F and 400°F.

C. Exposure to air saturated with water vapor at 140°F.

D. Friction test.

Examination of the specimens under a low power stereo microscope showed that, while the steel substrates appeared polished to the unaided eye, the actual surface finishes were scratched and not at all uniform in texture. The films were transparent and the film surface could not readily be observed in reflected light at moderate or low magnifications.

A photograph of the group I specimens is shown in Fig. 1, and typical surface areas for each of the four specimens at X200 magnification are shown in Fig. 2. Corresponding series of photographs for group II (Figs. 3 and 4), group III (Figs. 5 and 6), and group IV (Figs. 7 and 8) follow.

It will be seen that the substrates have a variety of surface finishes and textures. Considerable differences exist both within and between groups. Group I generally shows polishing scratches of somewhat random direction with deep, generally longitudinal furrows; specimen 15 of this group has areas of pitting (Fig. 2). In this group of specimens, the coating was applied in two sections, and there is a gap of uncoated, or very thinly coated, substrate between the two sections. Group II (Fig. 4) specimens showed the most uniform finish, characterized by rather deep, randomly oriented polishing scratches. Group III (Fig. 6) shows a wide variation from the pitted surface of specimens 21 and 23 to the highly directional, transverse ground finish of specimen 22, which also showed some deep voids. The coatings of the group IV (Fig. 8) specimens appeared less uniform than those of the other groups, particularly at the ends of the coated area, and showed a range of interference colors. This group also shows wide variations of substrate surface texture.

Since variations in the substrate surface texture were most likely to affect the friction measurements, one specimen from each group was first selected for friction testing to provide test samples with as nearly similar surface textures as possible. The specimens for the other tasks were allocated after these choices were made, also on the basis of the closest similarity of surface texture. The specimen allocations are summarized below.

Test	Group I	Group II	Group III	Group IV
Friction test	13	19	23	35
Microscopic examination and stripping test	14	18	22	36
Temperature cycling	15	20	21	34
Humidity test	16	17	24	33

Task A - Examination of As-Received Coatings

Specimens 14, 18, 22, and 36 were used for this task.

Stripping Tests

In order to test the adherence of the coating, a simple stripping test was used employing small pieces of Scotch tape approximately 2 mm by 5 mm. These were applied to the surface adjacent to the specimen edge, one short edge of the tape being left free. The tape was smoothed and pressed to remove entrapped air, then removed without jerking by pulling the free edge of the tape with forceps in a direction normal to the specimen surface.

Specimens 18, 22, and 36 showed good adhesion, the Scotch tape stripping cleanly from the coating surface, but the coating on specimen 14 was detached by this test. Fig. 9 shows an area of the surface where the film was detached, leaving an island of the coating still adhering to the substrate. While scratches on the substrate can generally be seen through the transparent film, the film itself appears to have a smoothing or leveling effect. Sharp surface features of the substrate are reproduced on the film surface, but with somewhat rounded edges so that they present a less distinct appearance. Fig. 10 is a stereo electron micrograph of the step between the substrate and the island of unstripped film. The film thickness at the step was measured by optical interference techniques as approximately 1500 Å.

Optical and Electron Microscope Examination

Because of the transparent nature of the coating, optical microscope examination of the surface was found generally unsatisfactory except in one respect. Observations of the shift in interference colors in the vicinity of scratches or pits in the substrate confirmed that the film was thicker in these regions, thus accounting for the apparent leveling or smoothing effect of the coating.

Electron microscope examination of replicas was therefore adopted as a more satisfactory technique for studying the coating. Specimen 14 could not be studied by this method, as the coating was stripped by the replica, in the same manner as occurred during the Scotch tape stripping tests. Stereo electron micrographs of specimens 18, 22 and 36 are shown in Figs. 11, 12, and 13. The coatings in the as-received condition generally show a smooth, fine-grained surface free from major defects, but marked differences between the films are very apparent from the micrographs. The coating on specimen 18 has a slightly textured appearance not found in the other two samples. The coating on specimen 36 has a rather uniform distribution of fine hemispherical cavities or pits. In neither case were these features related to the appearance of the substrate.

Taper Section Studies

Taper sections of specimens 14, 18, 22, and 36 were prepared and examined by high magnification optical microscopy and by electron microscope replica techniques. The organic film was preserved during sectioning by the deposition of a copper film. The initial copper coating was achieved by vacuum evaporation; this was followed by an electrolytic copper strike, and the coating thickness was then built up to approximately 0.004 in. using a cyanide electrolyte bath. A 15^{o} taper angle was employed in sectioning the mounted, copper-plated samples. Careful examination showed that in all samples except that from specimen 36, the coating had apparently been destroyed or removed during the plating operation. Fig. 14 shows a stereo electron micrograph of the steel substrate /film/ copper coating interface. The organic film, estimated to be about 1000 Å thick, can be detected between the steel and copper.

The taper sectioning technique proved to be generally difficult, and the observations made possible by its use were not of sufficient interest to justify further developmental effort.

Task B - Temperature Cycling

Specimens 15, 20, 21, and 34 were subjected to temperature cycling between 400^{o}F and 0^{o}F for ten cycles. Each cycle consisted of 20 hrs. at 400^{o}F and 4 hrs at 0^{o}F ambient air temperature. No attempt was made to control humidity.

At the end of the test period all the films appeared to be intact and in good condition (Fig. 15); all specimens passed the Scotch tape adhesion test. The interference colors of the films became more intense after the first heating cycle, indicating some change in reflectance at the film substrate interface. However, since the orders did not alter, it can be concluded that the film thickness did not change. The reduction of reflectance at the interface might suggest that oxidation of the metal surface was occurring, which in turn would imply either that there was some reaction between the film and the substrate or that the film was not an effective oxygen barrier at 400^{o}F.

Electron microscope replicas showed that a variety of changes in the films had occurred as a result of exposure to the temperature cycling test. Specimen 15 (Fig. 16) showed a wrinkled surface from which discrete areas appeared to have defoliated, exposing a lower layer of coating; it does not appear that the coating has separated from the substrate. This specimen also showed small white particles generally of circular or annular form. The distribution and size of these particles is reminiscent of the pits or bubble-like features of specimen 36 (group IV) in the as-received condition (Fig. 13). Specimen 20 (group II, Fig. 17) showed surface features suggestive of thermal etching. Small checks or fissures were found in the surface of specimen 34, and were associated with small blisters apparently caused by gas entrapped or liberated at the film/metal interface (Fig. 18).

Task C - Humidity Test

Specimens 16, 17, 24, and 33 were placed in a humidity test chamber at 140°F and 100% relative humidity for a period of ten days. The samples were mounted in a rack with the coated surfaces vertical to prevent the formation of standing water drops on the surface.

After 24 hours exposure, the coatings had already begun to lift from the steel substrate, and corrosion of the base metal was starting in the areas where the film had become detached. After 10 days, all coatings had peeled and disintegrated and the substrate metal had rusted. The specimens were removed from the humidity chamber and allowed to dry in air. Their appearance is shown in Fig. 19. In this condition, many areas of the coatings flaked off readily at the lightest touch; the Scotch tape stripping test removed the coatings completely.

Task D - Friction Tests

A simple friction device that could be mounted in an Instron testing machine (Figs. 20 and 21) was designed and built for these studies. The specimen was held by double-sided adhesive tape onto a block which was attached to the movable crosshead of the Instron. A saddle, carrying a hardened steel rider provided with two cylindrical contacts, was pulled over the specimen surface by a nylon thread that passed over a pulley and was attached at its upper end to the load cell of the Instron machine. The thread was attached to an extension arm projecting from the saddle so that the thread was pulling in the plane of the specimen surface in a direction parallel to the specimen length. The detail of the rider surfaces is shown in Fig. 22.

The choice of rider geometry was dictated by the requirement of relatively low contact stress, and the practical matter of obtaining smooth, even rider motion. Spherical contact surfaces, within suitable limits of spherical radius, impose very high contact stresses. A cylindrical contact surface was therefore preferred. A triple cylindrical contact rider was tried, but found not to give uniform loading, because the rider tended to "nose over" when pulled over the specimen surface. A double cylindrical contact was found to provide the best stability. The two cylindrical contact surfaces were made with a radius of 0.125 in., a width of 0.029 in., and a spacing of 0.033 in. between their inner edges. The saddle weight was 88.94 gm, resulting in a calculated Hertzian contact stress of 17,188 psi on a steel surface. The modifications to the Hertzian area of contact produced by the thin organic film would of course imply that the actual stress on the film is significantly less than 17,000 psi, but no attempt was made to estimate a probable value.

The test procedure adopted was to draw the rider over the specimen surface by driving the testing machine crosshead, and to record the force in the nylon thread on the strip chart recorder of the machine. Two crosshead speeds, giving rider travel velocities of 0.002 in. and 0.2 in. per min, were selected. The slower speed may be taken as providing a quasi-static friction condition.

The frictional resistance of the pulley under which the nylon thread passed could be a significant part of the total force measured by the recorder. Since the pulley friction will also increase with the load in the thread, comparison of the frictional behavior of the four specimens at the two rider speeds required the determination of the frictional resistance of the pulley as a function of the total recorded force. This measurement was made by comparing the force required to stretch a rubber band attached at one end to the nylon thread when the other end was attached to the specimen support block (so that the thread passed under the pulley as in the normal test arrangement) with the force required to stretch the band when its free end was attached directly to the crosshead of the machine (so that the thread did not pass around the pulley). Load extension plots for the two cases were approximately linear and showed that the pulley friction varied from 22% to 28% of the total force, for a total force range of 5 to 20 gm. An average correction of 25% of the total force measured was therefore deducted from the total force to obtain the net friction force resisting the movement of the rider over the specimen surface. This value was then divided by the rider load of 88.9 gm to obtain the coefficient of friction, according to the relationship:

$$\mu = \frac{\text{friction force}}{\text{normal load}}$$

The results obtained are summarized in Table I. The range of friction coefficients is generally lower than those obtained for clean dry steel-against-steel contact and is more typical of rather poor boundary lubrication. No special treatments were used to clean the uncoated areas of the specimens, because of the danger of also affecting the coated areas, so that the nominally uncoated surfaces could well have been contaminated. The friction traces also indicated that a thin, sometimes invisible film often extended outside the margins of the actual coated areas, by distances of up to 0.2 in.

Table I

FRICTION TEST RESULTS

(Rider load, 88.9 gm)

Specimen No.	Surface	Rider Speed, in./min	Friction Force, gm	Corrected Friction Force, gm	Coeff. of Friction	Comments
13 (Group I)	Uncoated	0.002	16	12	0.13	Sawtooth trace of amplitude ~ 5 gm.
	Coated	0.002	14-15	10.5-11.2	0.12-0.13	Trace sometimes sawtoothed, with amplitude ~ 2 gm; sometimes wavy, amplitude ~ 0.5 gm.
	Uncoated	0.2	13-16	9.8-12	0.11-0.13	Generally sawtooth trace, amplitude 2-5 gm. One wavy trace, amplitude ~ 1 gm.
	Coated	0.2	16-20	12-15	0.13-0.17	Generally sawtooth trace, amplitude 2-8 gm.
19 (Group II)	Uncoated	0.002	15-16	11.2-12	0.13	Sometimes sawtooth with amplitude 3-6 gm; sometimes wavy with amplitude ± 0.5 gm.
	Coated	0.002	16-18	12-15.5	0.13-0.15	Variable; generally sawtooth with amplitude 2-3 gm, but with occasional wider excursions. One wavy trace, amplitude ± 1 gm.
	Uncoated	0.2	12-15	9-11.2	0.10-0.13	Generally smooth, wavy trace, amplitude ± 0.5 gm.
	Coated	0.2	15-17	11.2-12.7	0.13-0.14	Generally sawtooth trace, with amplitude ~ 2-3 gm.

Table I (Concluded)

Specimen No.	Surface	Rider Speed, in./min	Friction Force, gm	Corrected Friction Force, gm	Coeff. of Friction	Comments
23 (Group III)	Uncoated	0.002	11-14	9.2-10.5	0.10-.12	Smooth, with some waviness, amplitude ± 0.5 gm.
	Coated	0.002	20-25	15-19	0.17-0.21	Variable sawtooth, amplitude ~ 3-8 gm with some wide excursions up to gm.
	Uncoated	0.2	13-15	9.8-11.2	0.11-0.13	Smooth, wavy trace with amplitude ~ ± 1 gm.
	Coated	0.2	20-30	15-22	0.17-0.25	Sawtooth, with amplitude 3-10 gm.
35 (Group IV)	Uncoated	0.002	14-18	10.5-13.5	0.12-0.15	Variable, from wavy trace with amplitude ± 1.0 gm to sawtooth, amplitude ~ 6-8 gm.
	Coated	0.002	16-20	12-15	0.13-0.17	Variable. Wavy trace with amplitude ± 2 gm to sawtooth trace with amplitude 3-8 gm.
	Uncoated	0.2	12-16	9-12	0.10-0.13	Wavy trace with amplitude ~ ± 1 gm.
	Coated	0.2	12-20	9-16	0.10-0.18	Irregular wavy trace with amplitude ± 0.5 gm. At lead-in to coated area, transient high forces ~ gm maximum.

Inspection of the results of Table I shows that the most general form of friction trace obtained was sawtooth in form, indicative of stick-slip motion of the rider. Results generally did not demonstrate any lubricating effect of the coating and suggested that friction over the coated area of a given specimen was slightly higher than over uncoated areas of the same specimen. Specimen 13 (group I) gave a friction coefficient slightly lower for the coated areas at a rider speed of 0.002 in./min, but somewhat higher at a rider speed of 0.2 0.2 in./min. Specimen 19 (group II) gave significantly higher values for the friction over the coated areas at both speeds, the uncoated surface showing a smoother, wavy friction trace, as compared with the more typical sawtooth pattern. Wavy traces also characterized the rider motion at both speeds over the uncoated areas of specimen 23 (group III), as contrasted with a sawtooth form of trace over the coated areas. The values of the friction coefficient over the coated areas approached double the corresponding values for the uncoated areas. Specimen 35 (group IV) also gave higher friction coefficients for the rider motion over the coated versus the uncoated areas at both speeds.

The friction tracks on the samples were examined by both optical and electron microscopy. Although the surface damage as viewed at low power appeared minor, the surface disturbance was too gross to be studied effectively by electron microscope methods, and use of this technique was therefore abandoned. Figures 23-29 show typical areas of the friction tracks on the coated areas of the various samples. Surface damage did not generally occur over the full width of the rider track, as can be seen from the relatively narrow damage tracks displayed in the photomicrographs; the full track width at the magnification used would be more than 4 in. wide.

Comparison of Figs. 23 and 24 (specimen 13) indicates that the higher speed of rider travel, 0.2 in./min, produced more film damage than the slow speed (0.002 in./min). The white areas on the micrographs are regions in which the film has been detached from the steel substrate.

A similar comparison of the effect of speed can be made in the two micrographs of the friction tracks on specimen 19 (Figs. 25 and 26). In spite of the somewhat higher friction coefficients measured for the coated areas of specimen 23, damage to the coating (Figs. 27 and 28) was less pronounced than for specimens 13 and 19. The coating of specimen 23 showed scratching along the friction track, but no evidence of actual disruption or detachment of the film from the substrate. Examination of the friction tracks for specimen 35 showed areas in which the coating had been removed at both rider speeds; Fig. 29 shows the track at 0.2 in./min rider speed.

Conclusions

The conclusions that can be drawn from this preliminary examination of silicone polymer coatings are summarized below.

1. The coatings are generally free from flaws and are well bonded to the steel substrate. The coating on specimen No. 14, however, did not pass a stripping test. The coating thickness was measured as approximately 1500 Å on this specimen.

2. The coatings are resistant to cycling between temperatures of 400°F and 0°F in air.

3. The coatings are not resistant to hot, humid atmospheres, and failed extensively after exposure to an environment of 140°F and 100% relative humidity.

4. Friction tests at a relatively light load did not reveal any lubricating behavior of the film. Friction coefficients tended to be higher for the coated than for the uncoated areas of the specimens. The coatings suffered damage during a single pass of a rider carrying a load of 89.9 gm at speeds of 0.002 and 0.2 in./min.

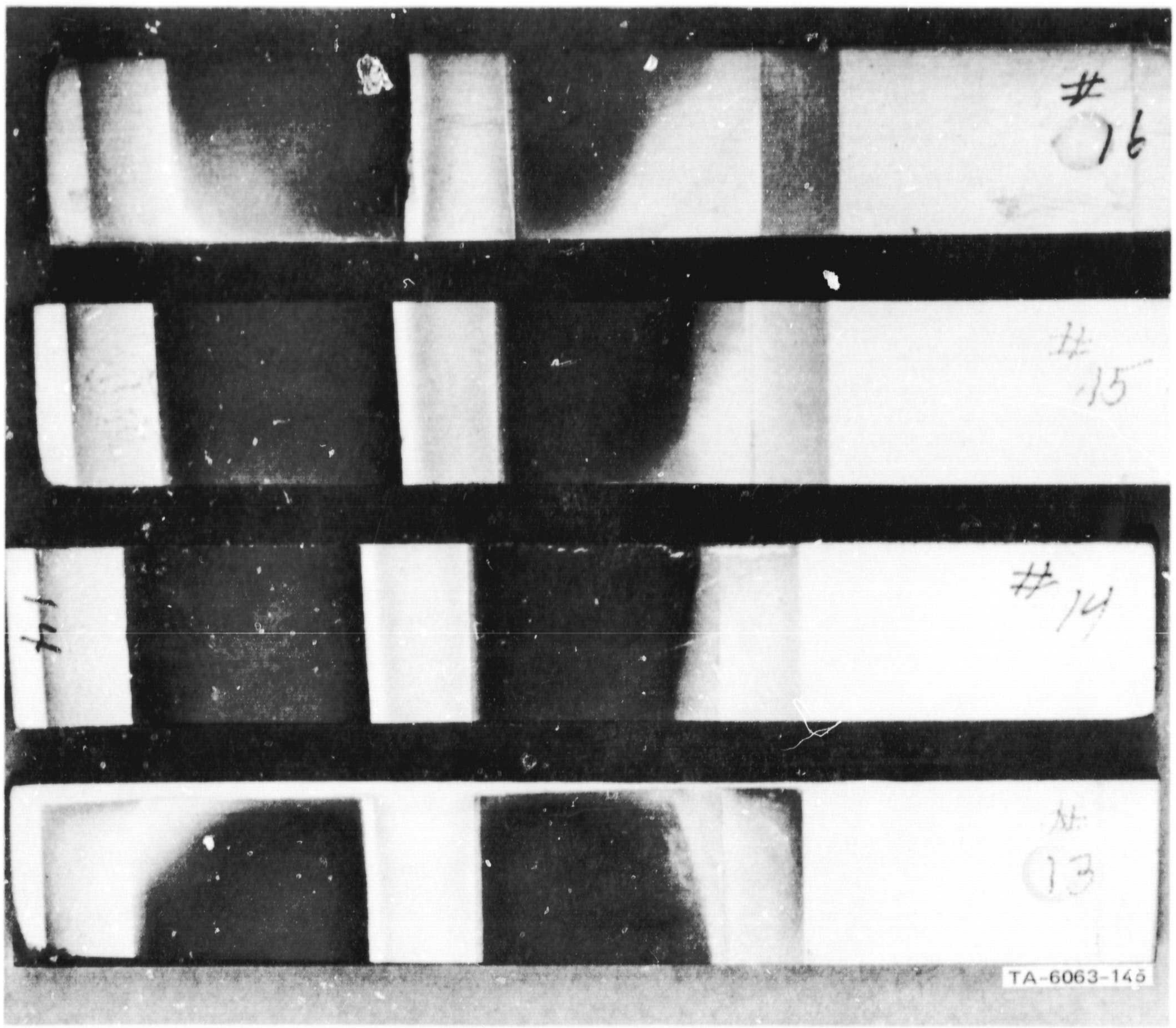
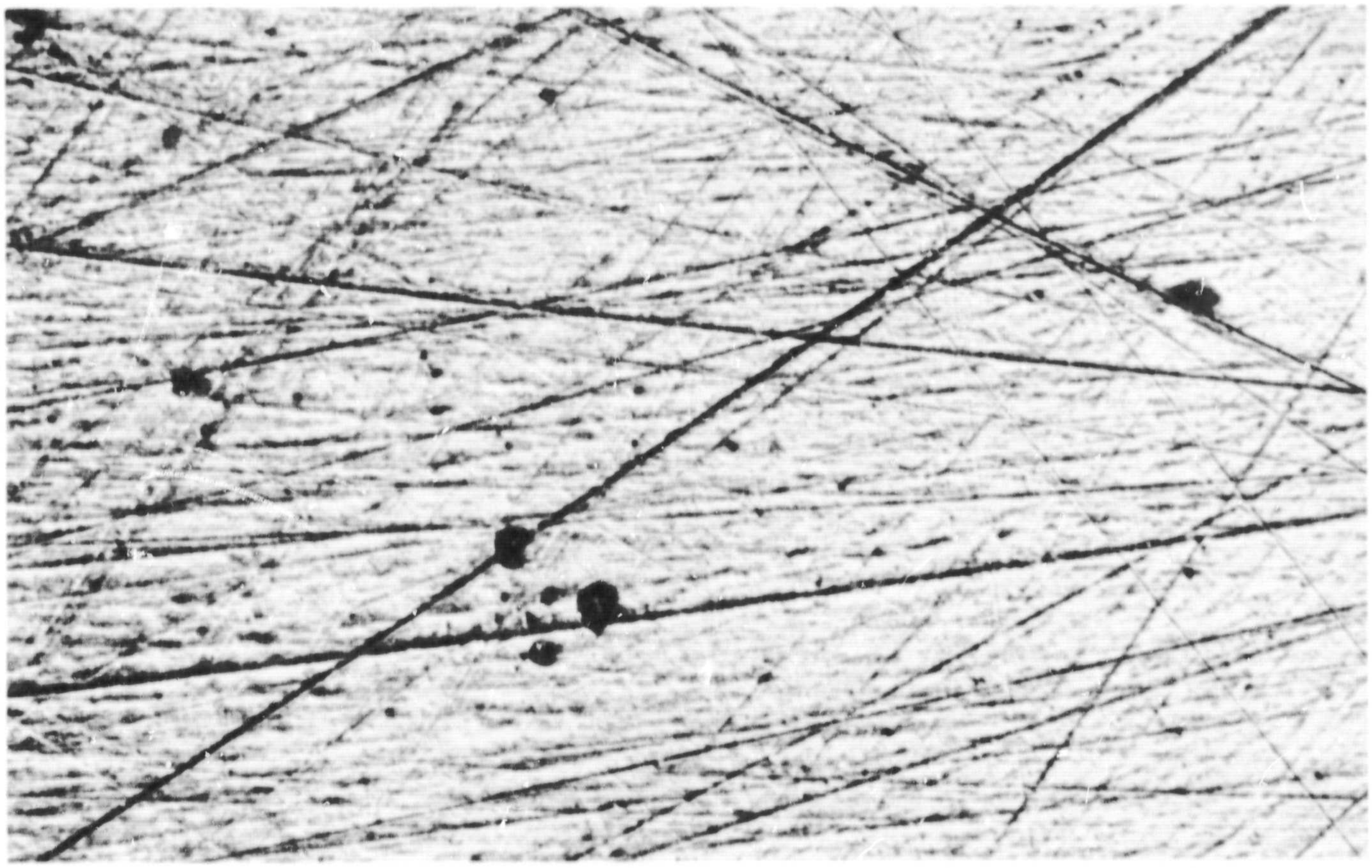
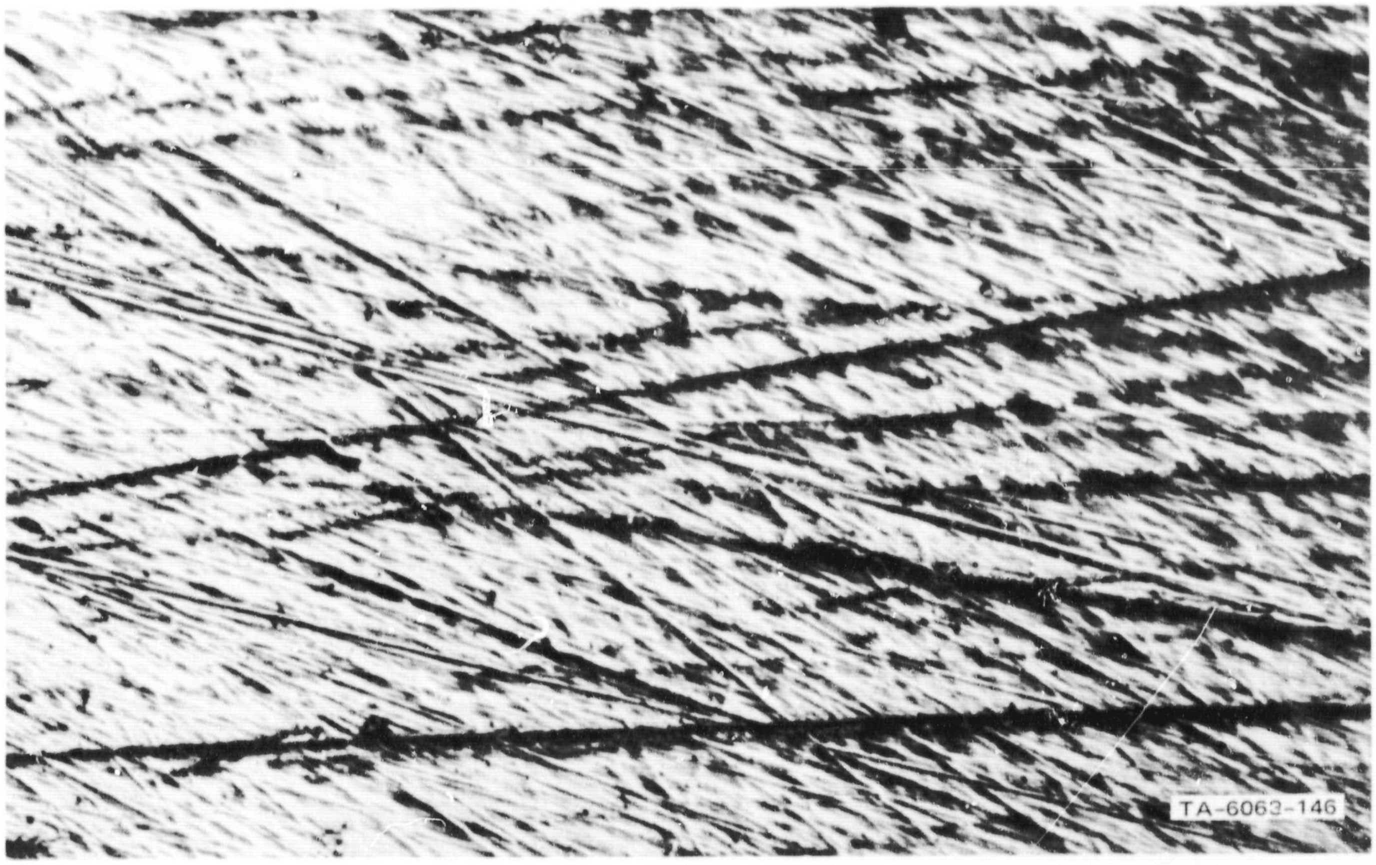


FIGURE 1 GROUP I SPECIMENS, AS RECEIVED

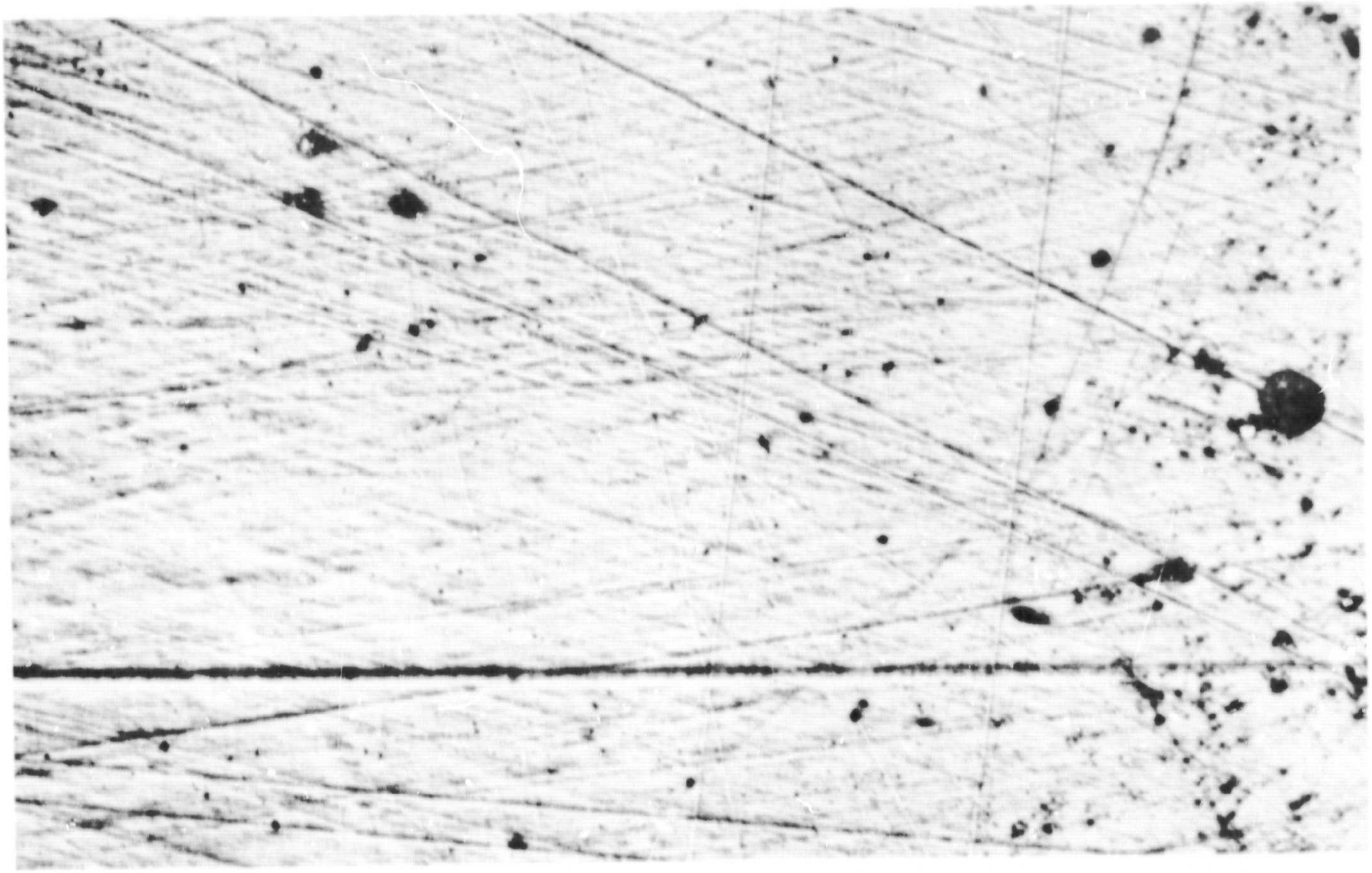


SPECIMEN 13

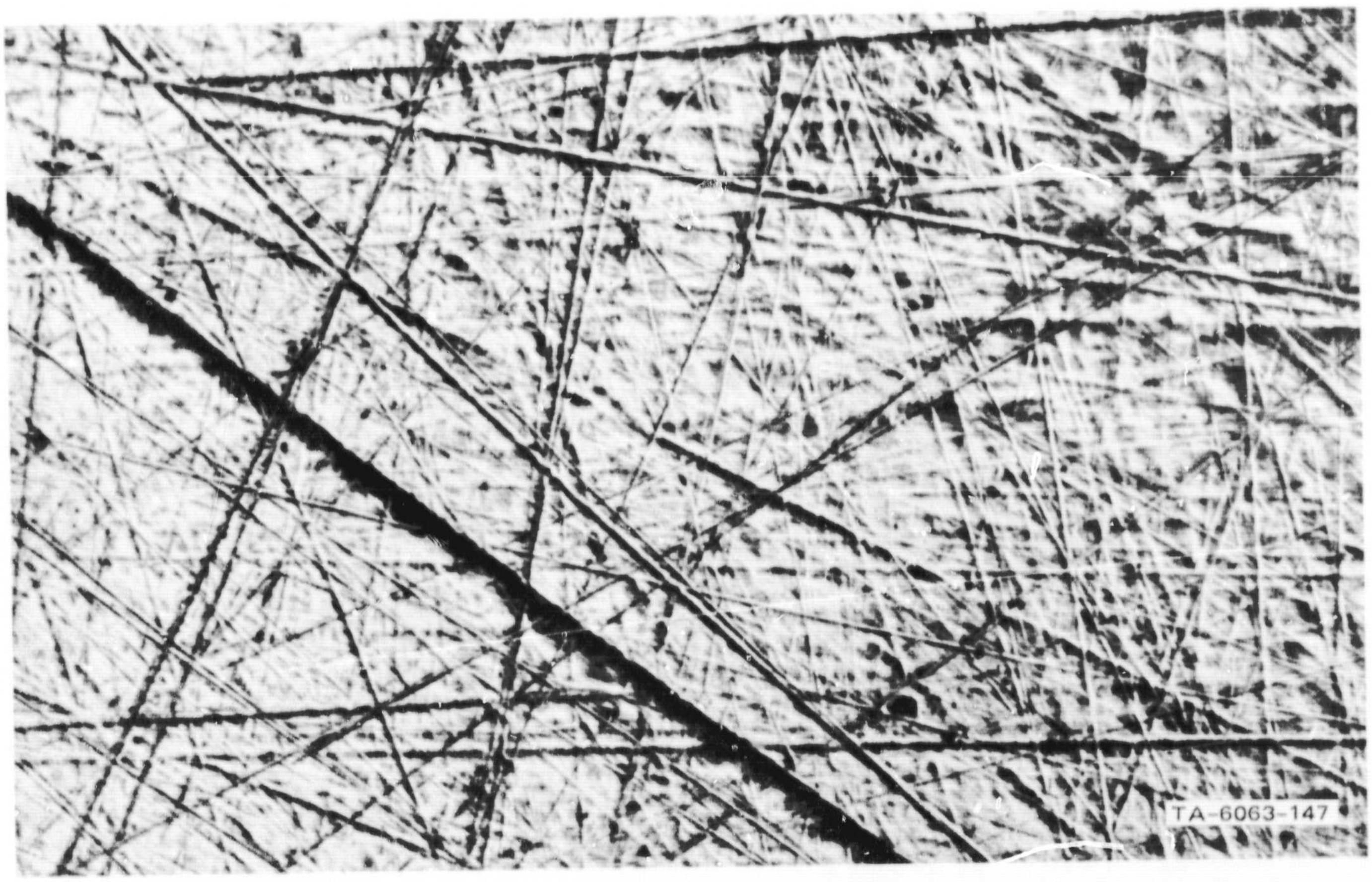


SPECIMEN 14

FIGURE 2 TYPICAL SURFACES OF GROUP I SPECIMENS X200



SPECIMEN 15



SPECIMEN 16

FIGURE 2 Concluded

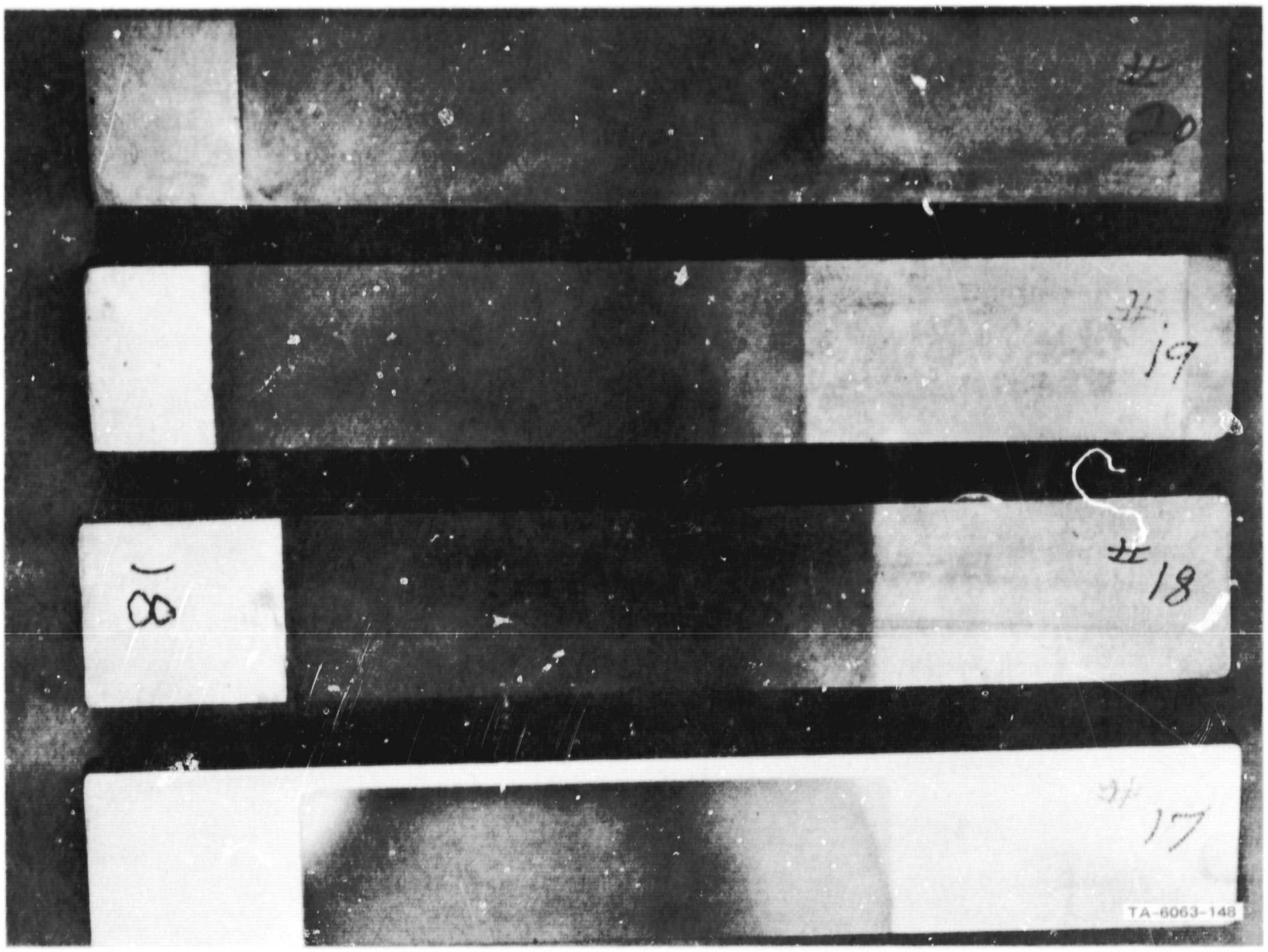
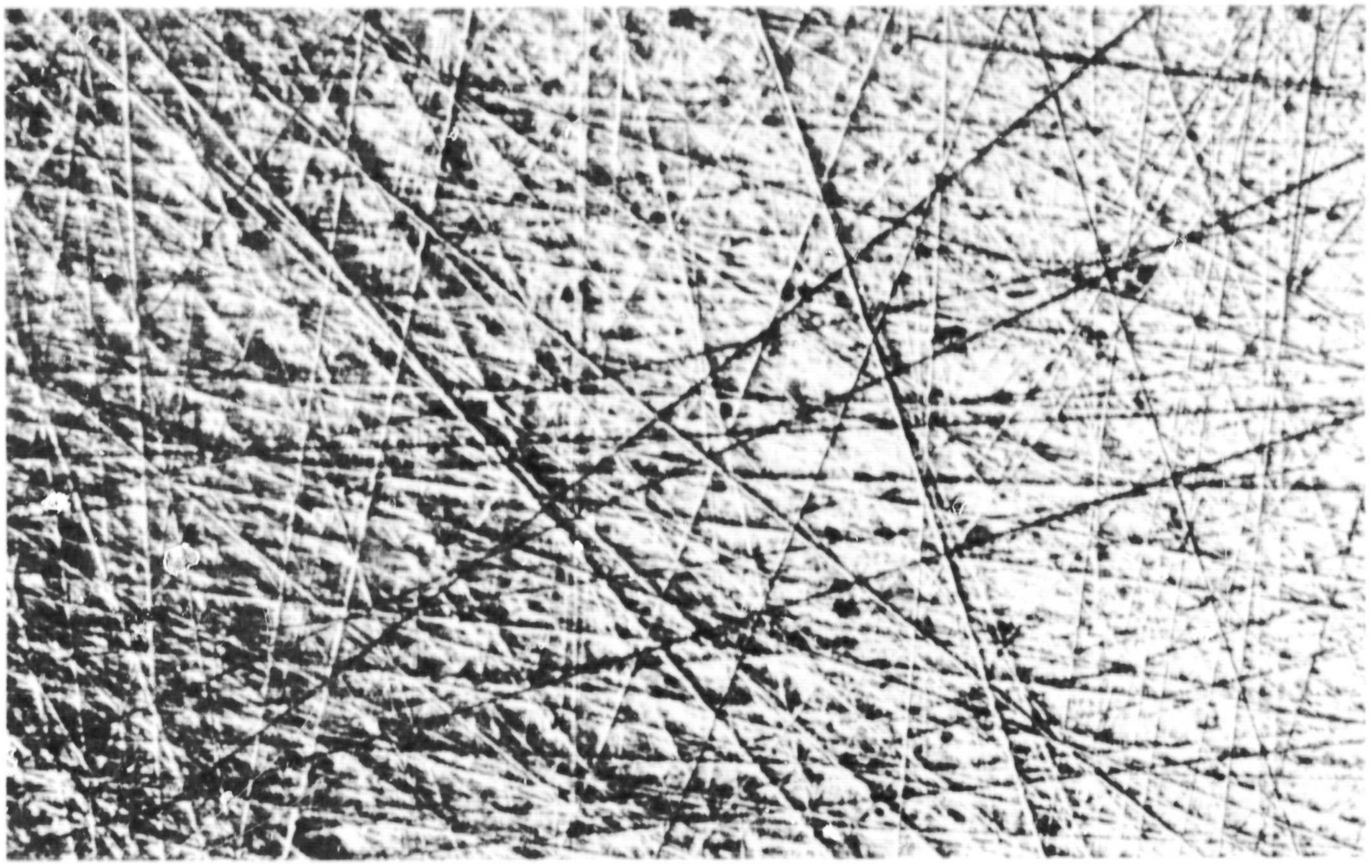
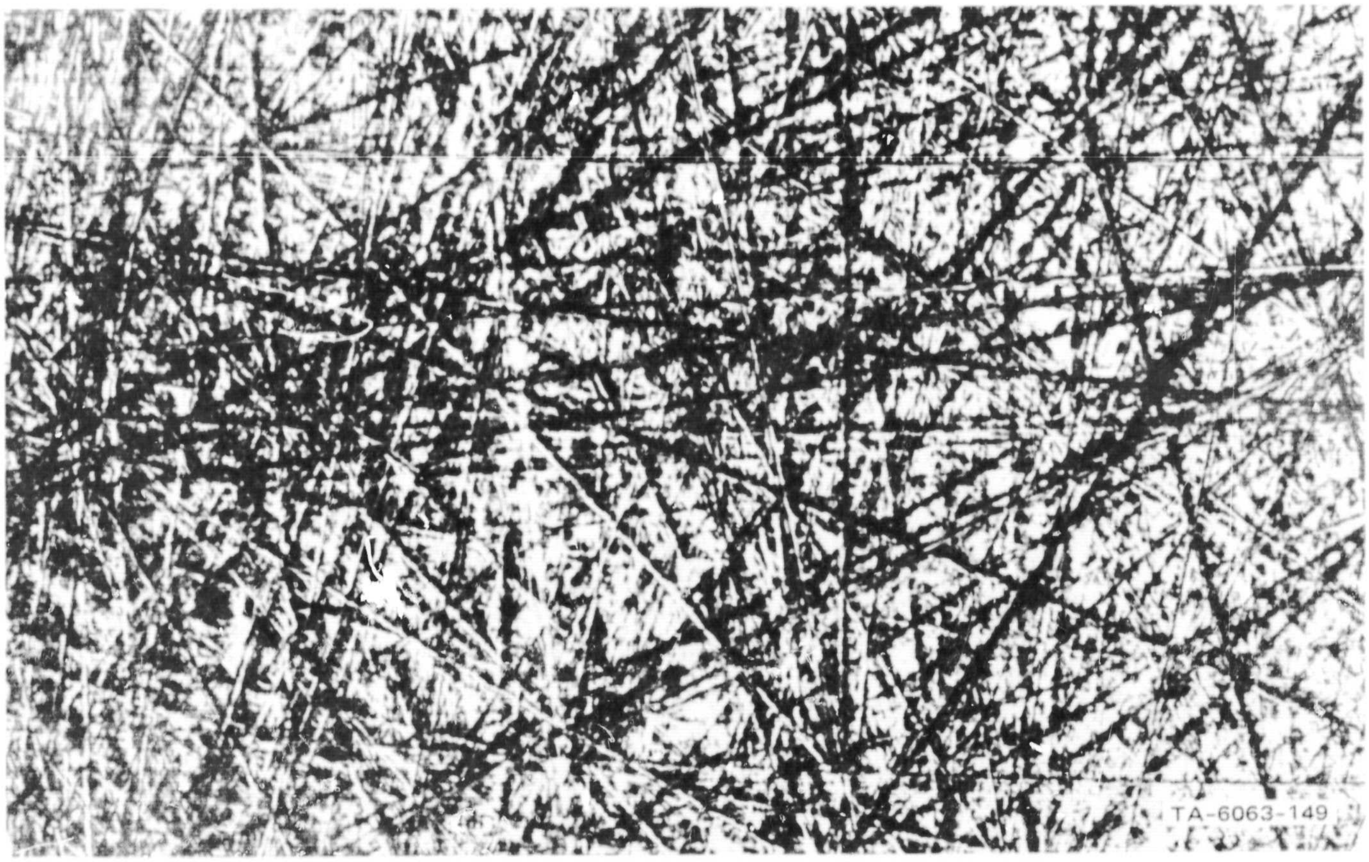


FIGURE 3 GROUP II SPECIMENS, AS RECEIVED

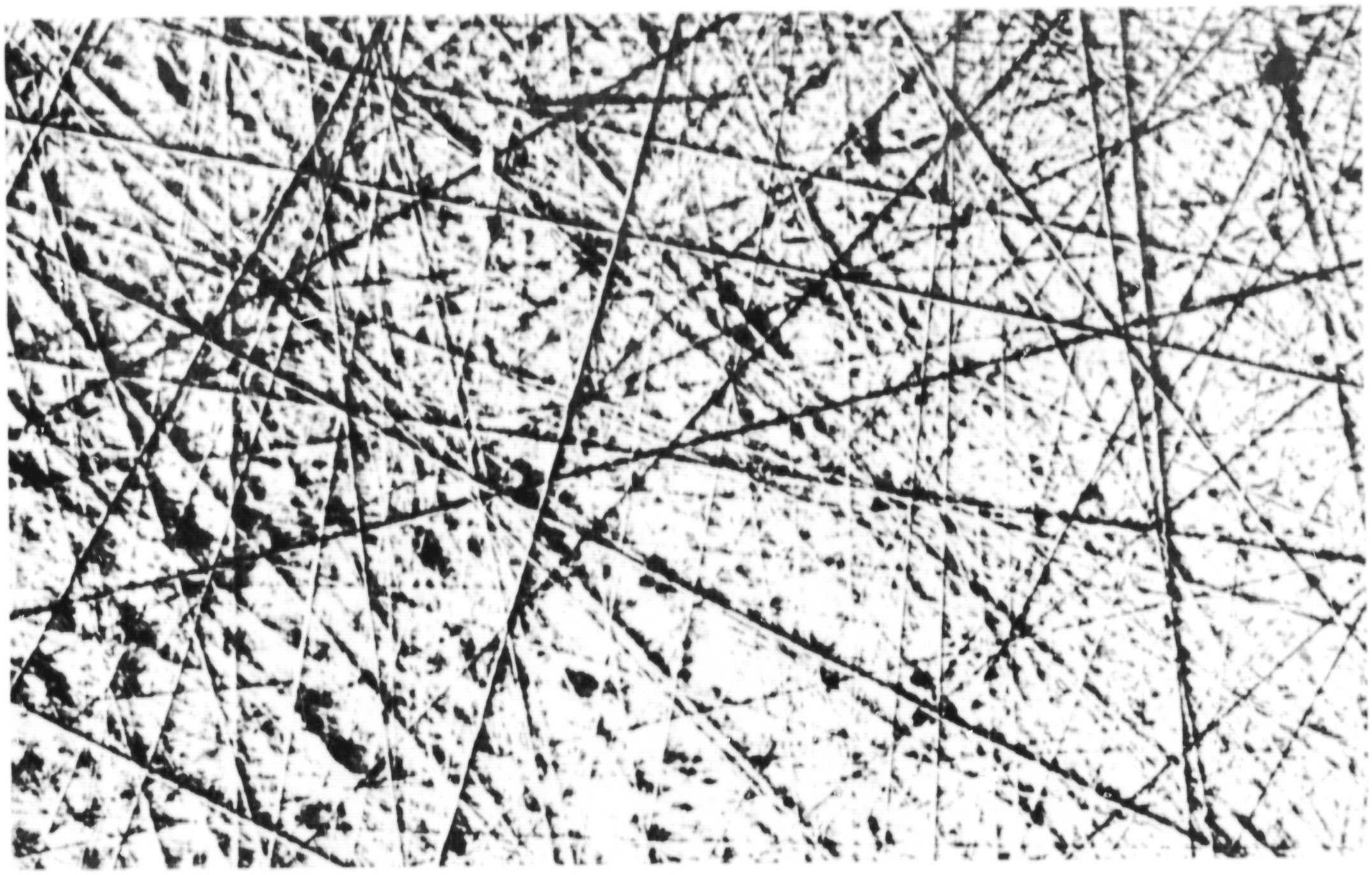


SPECIMEN 17

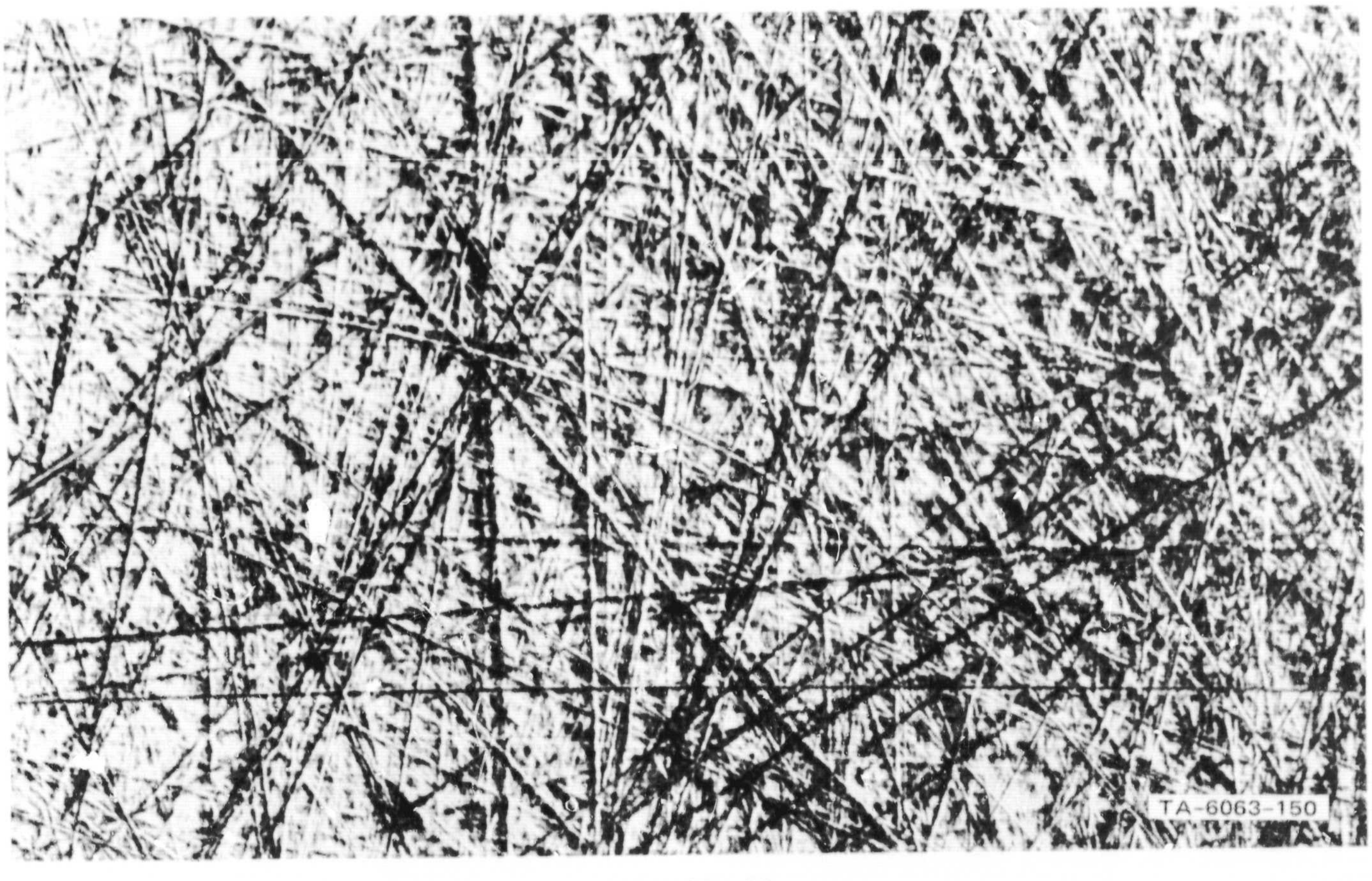


SPECIMEN 18

FIGURE 4 TYPICAL SURFACES OF GROUP II SPECIMENS X200



SPECIMEN 19



SPECIMEN 20

FIGURE 4 Concluded

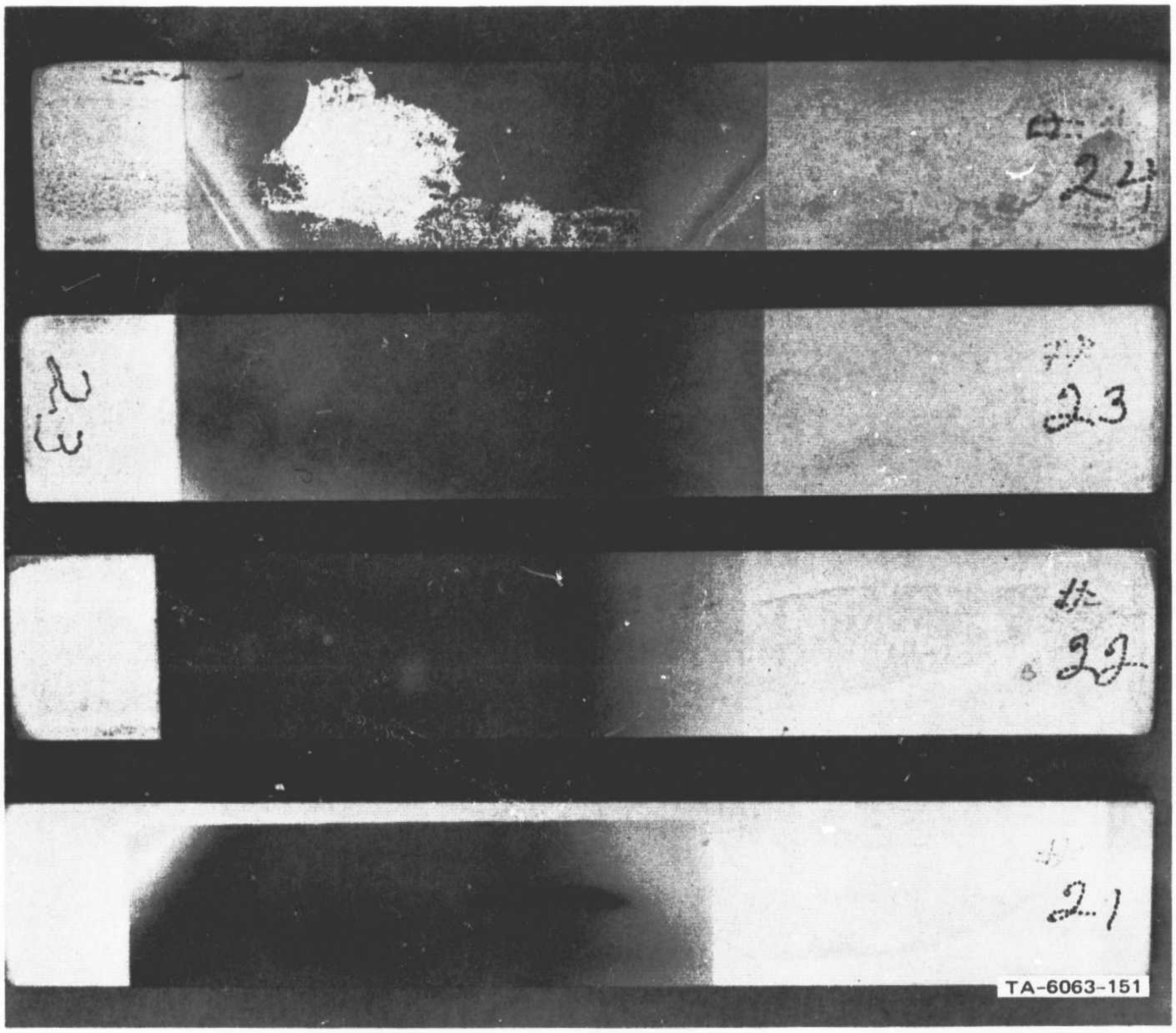
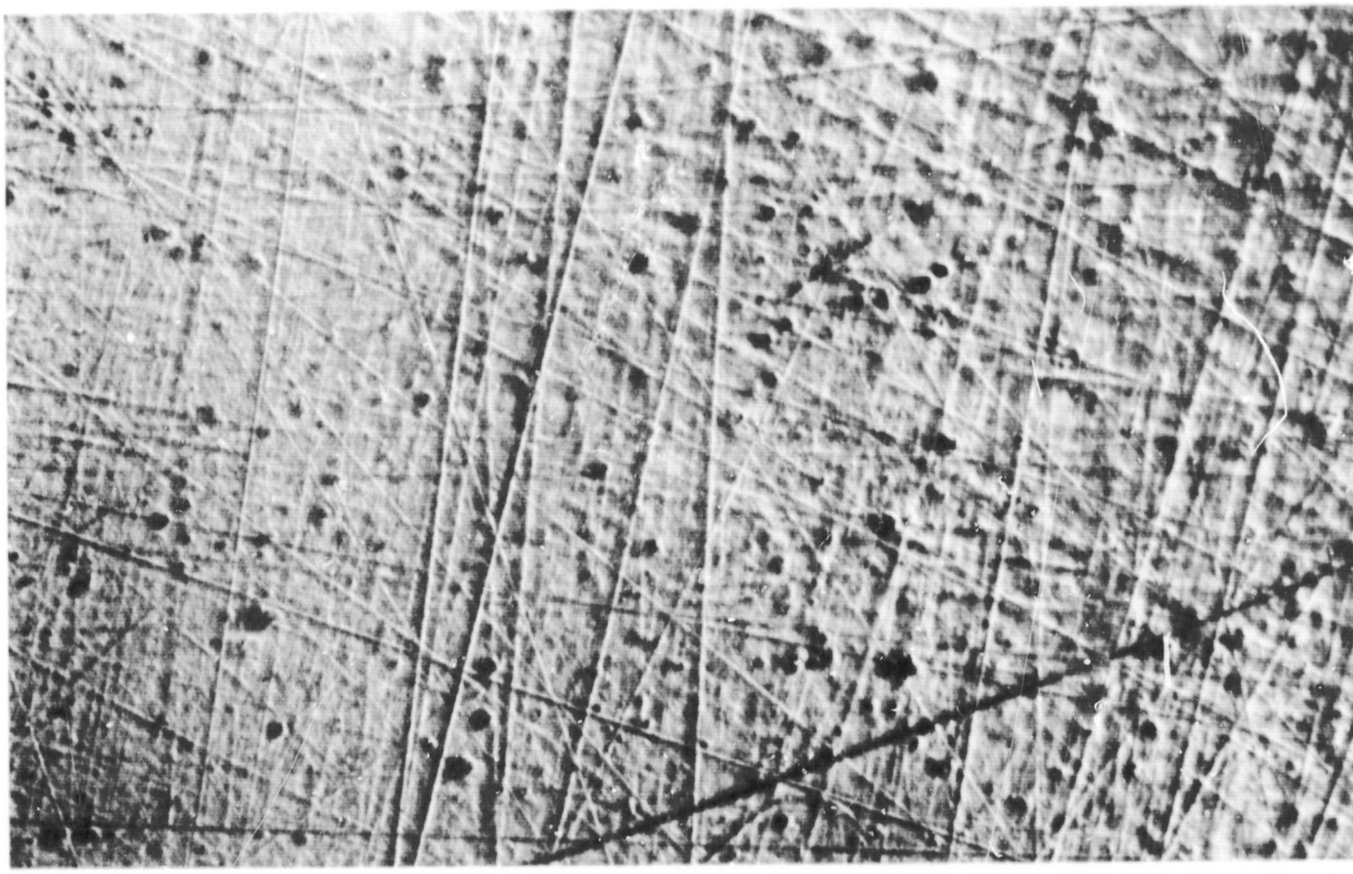
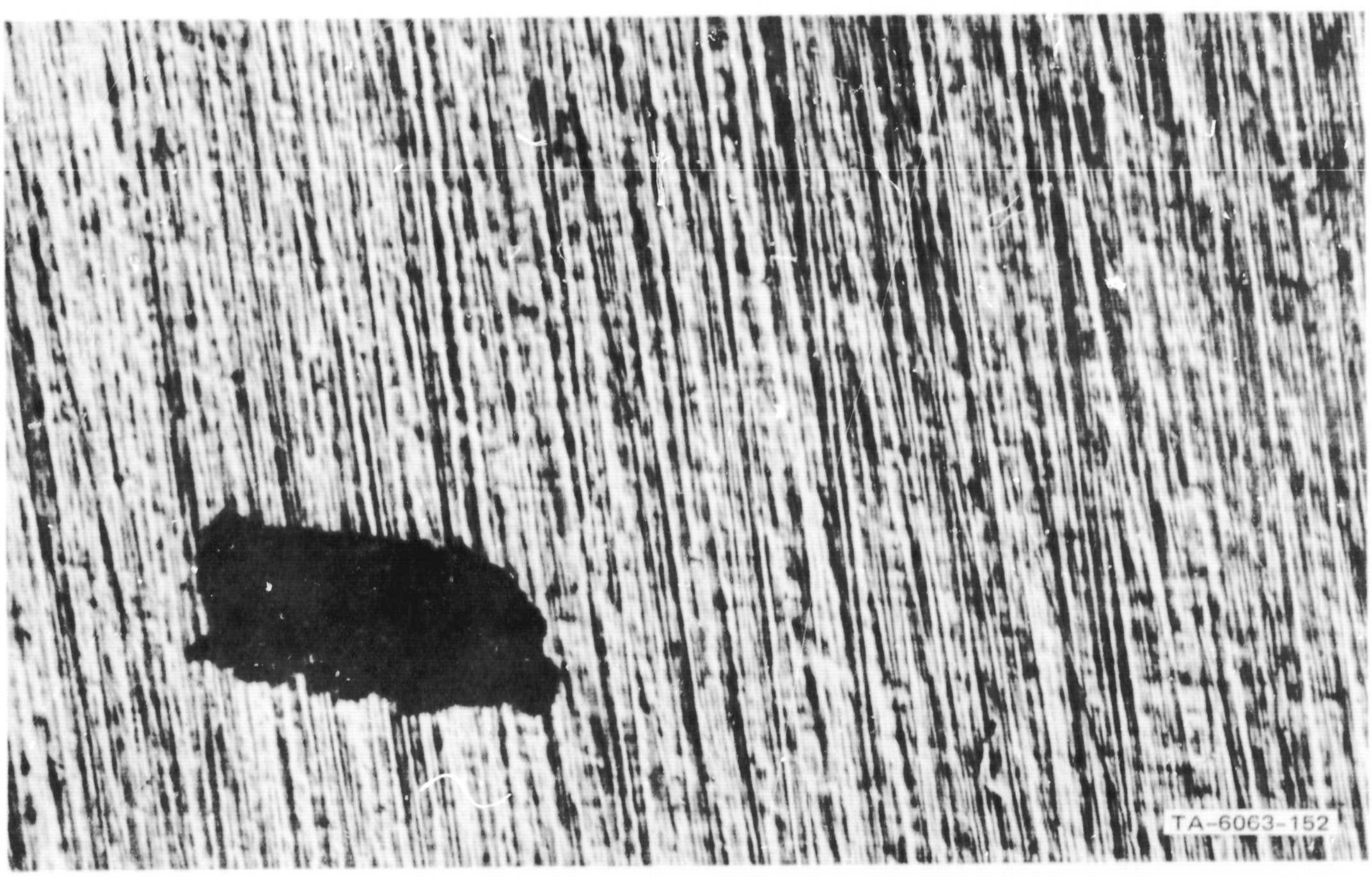


FIGURE 5 GROUP III SPECIMENS, AS RECEIVED

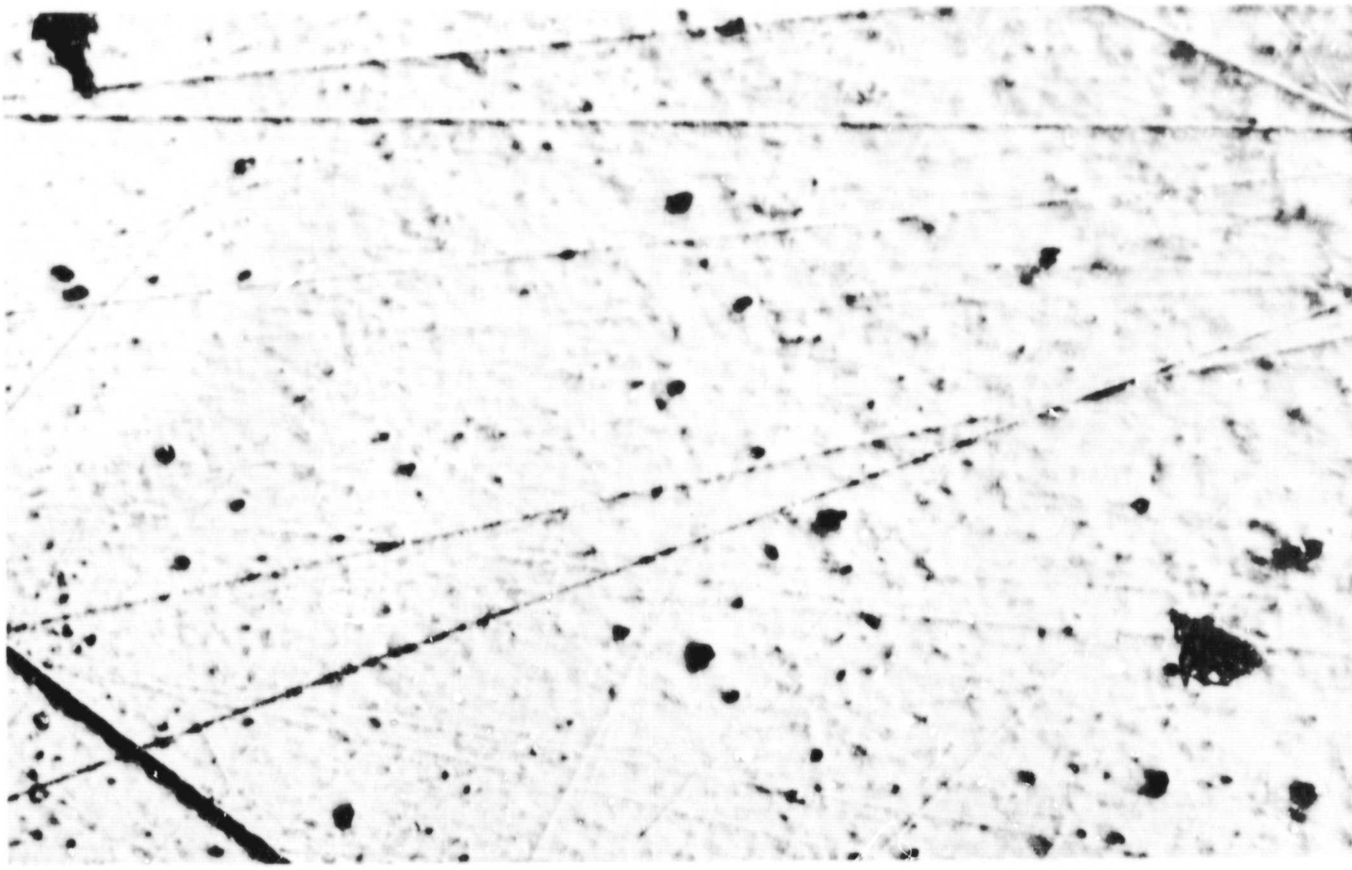


SPECIMEN 21

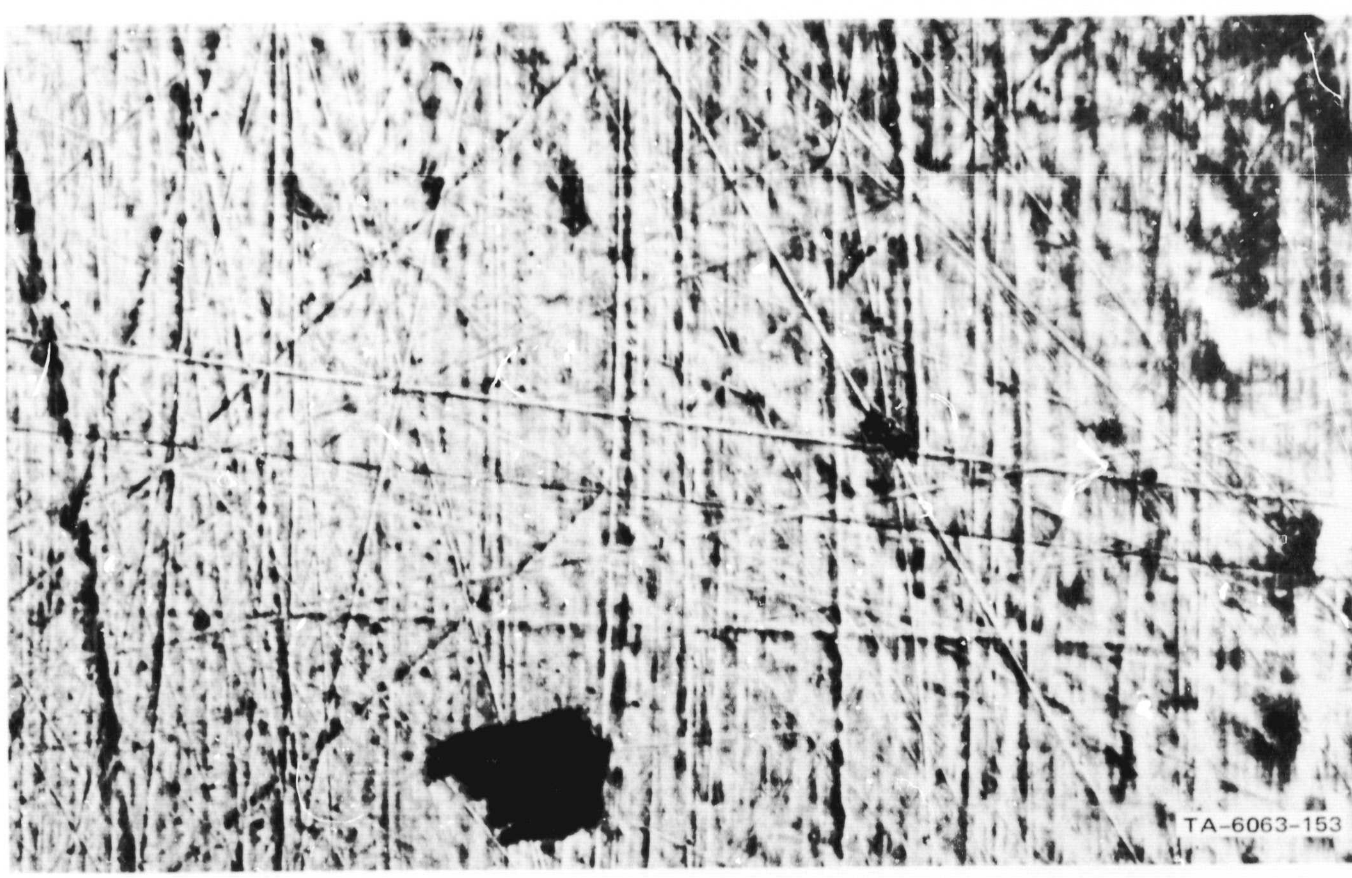


SPECIMEN 22

FIGURE 6 TYPICAL SURFACES OF GROUP III SPECIMENS X200



SPECIMEN 23



SPECIMEN 24

FIGURE 6 Concluded

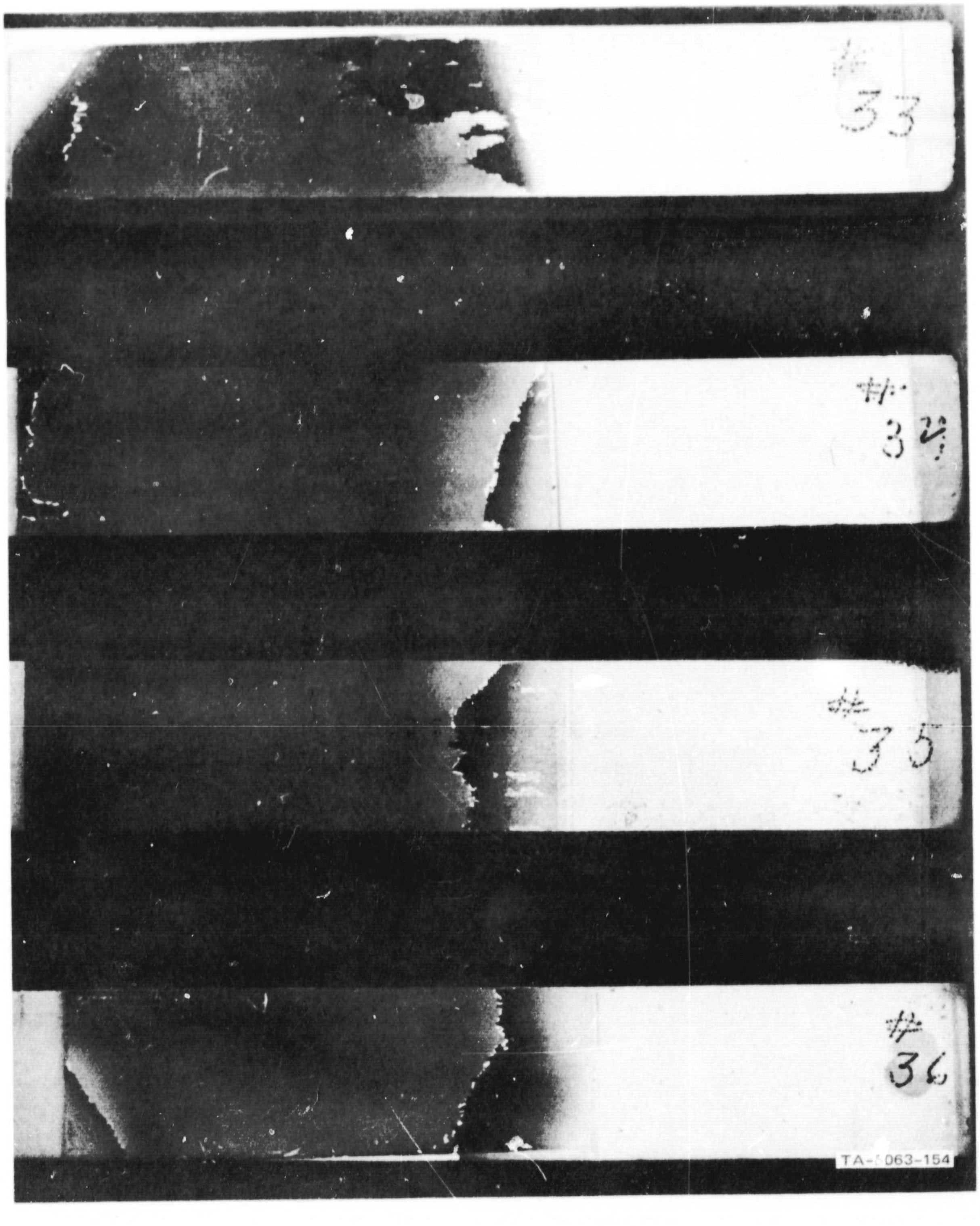
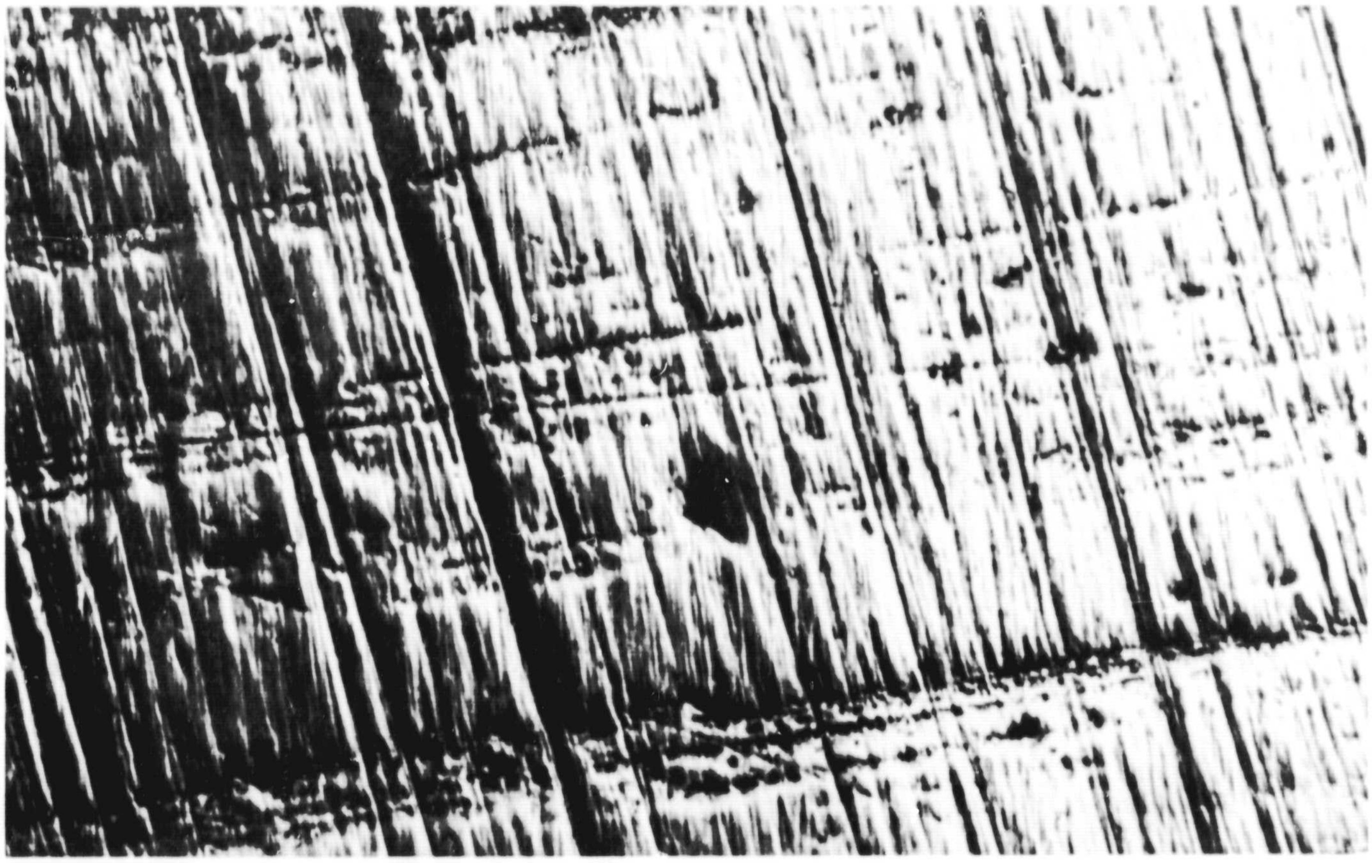


FIGURE 7 GROUP IV SPECIMENS, AS RECEIVED

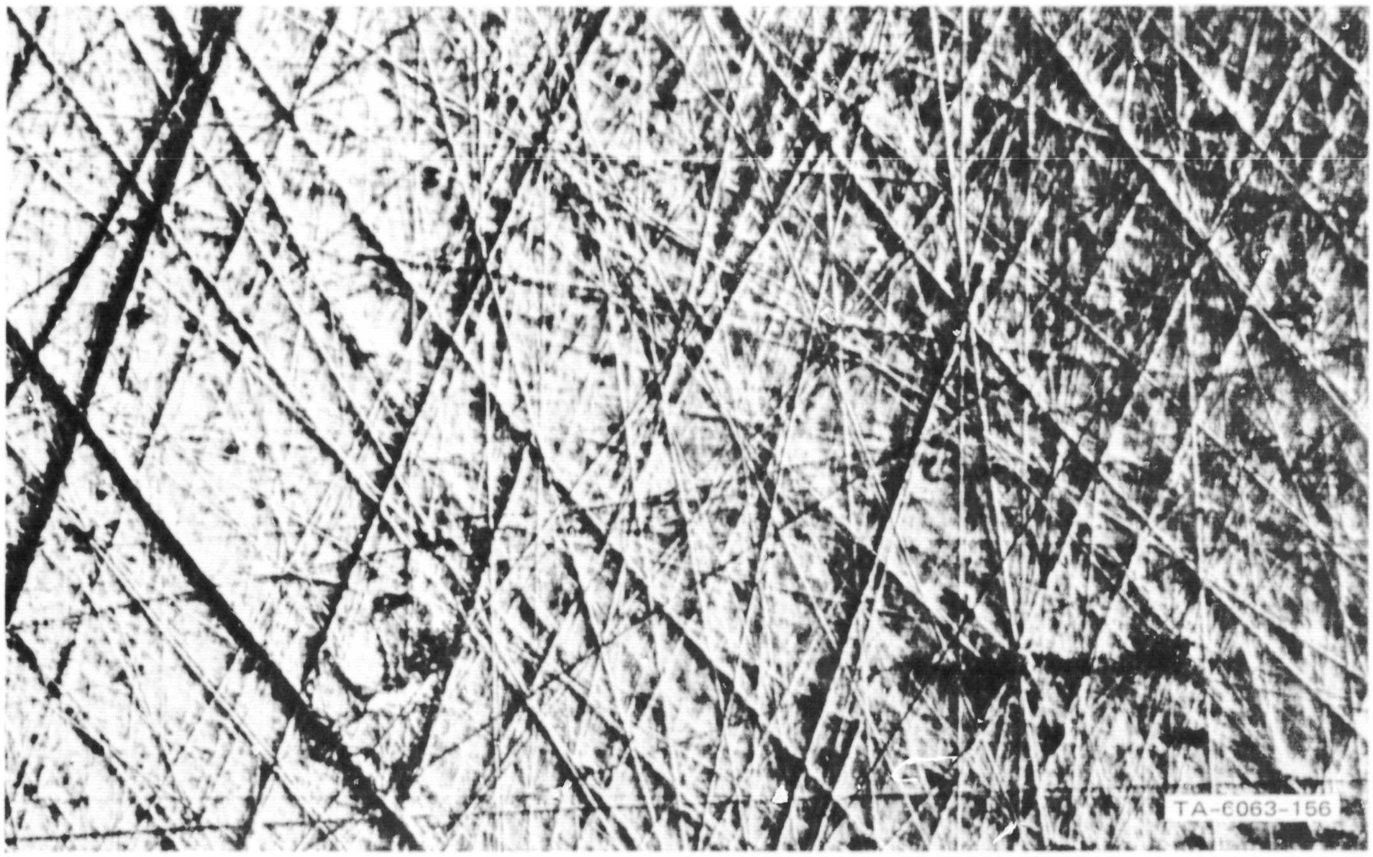


SPECIMEN 33

SPECIMEN 34

FIGURE 8 TYPICAL SURFACES OF GROUP IV SPECIMENS X200

SPECIMEN 35



SPECIMEN 36

FIGURE 8 Concluded

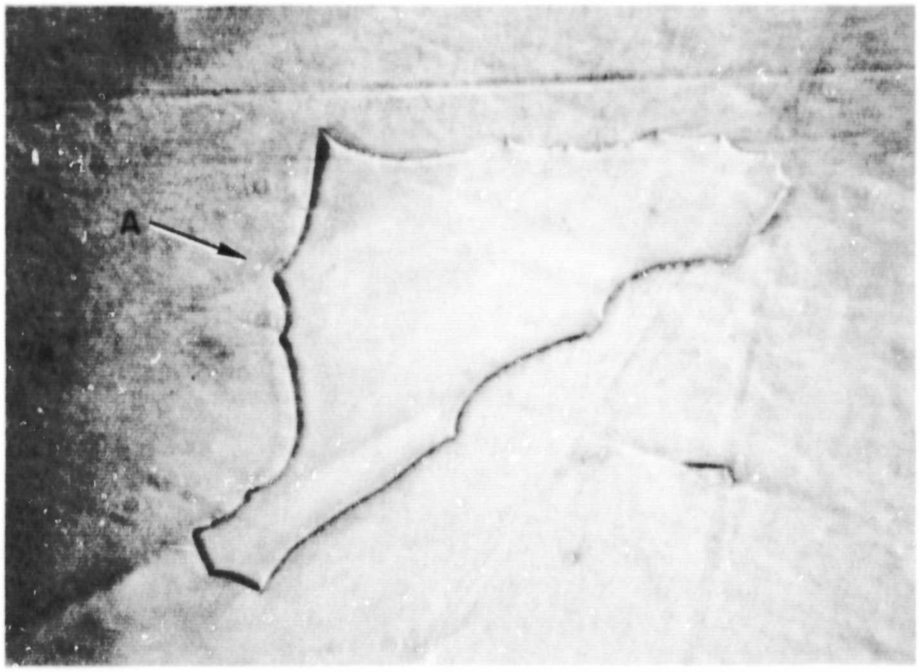


FIGURE 9 SPECIMEN 14, AFTER STRIPPING TEST, SHOWING ISLAND OF ADHERING COATING X440

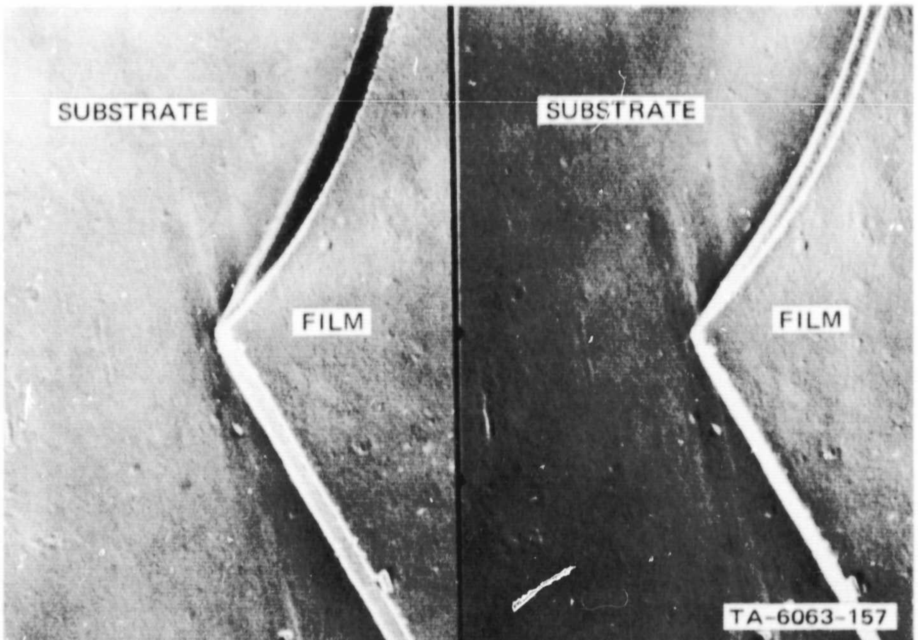


FIGURE 10 STEREO ELECTRON MICROGRAPH OF AREA 'A' OF FIGURE 9 X2000

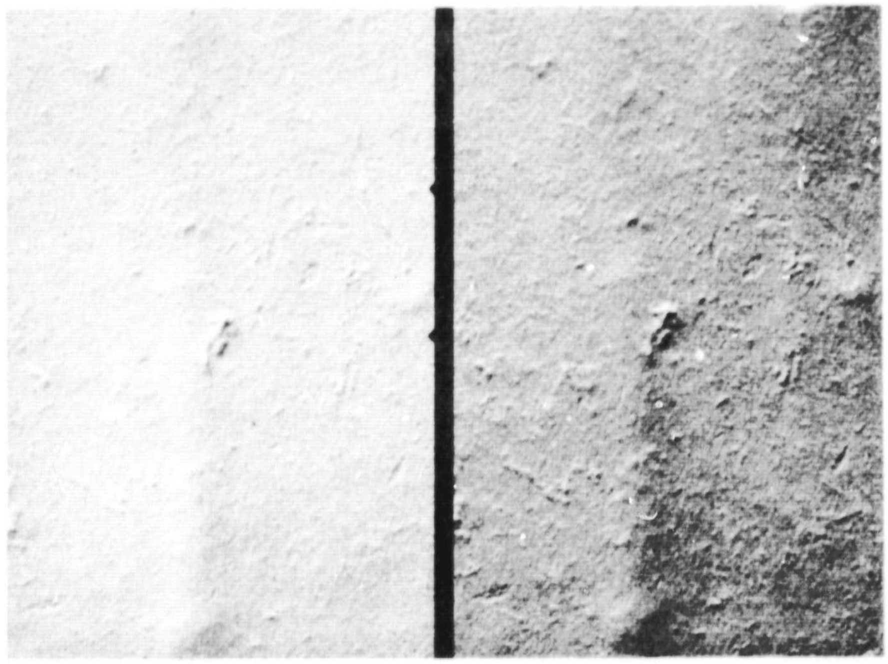


FIGURE 11 STEREO ELECTRON MICROGRAPH OF COATING SURFACE, SPECIMEN 18 (Group II)

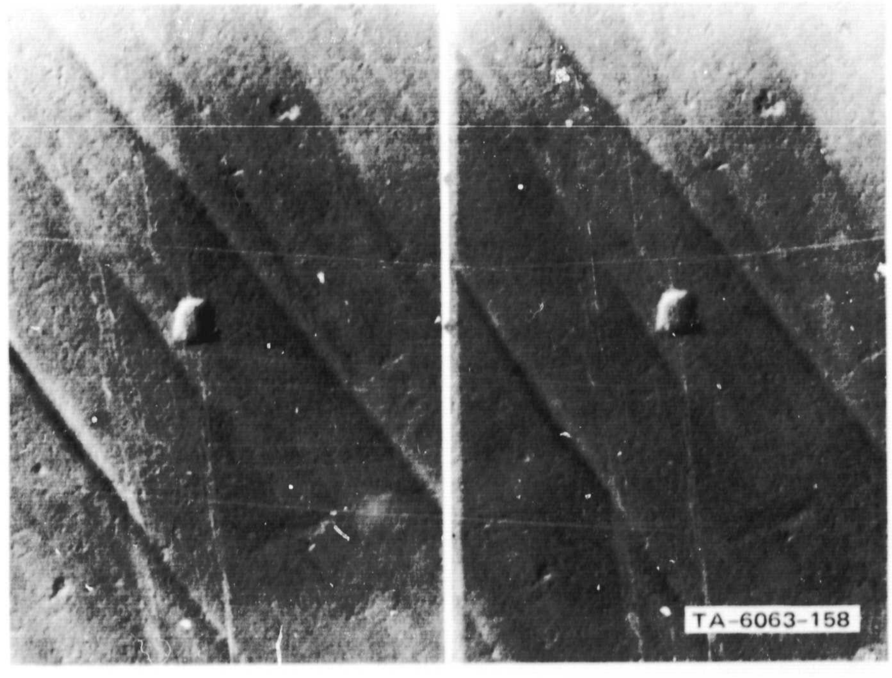


FIGURE 12 STEREO ELECTRON MICROGRAPH OF COATING SURFACE, SPECIMEN 22 (Group III)

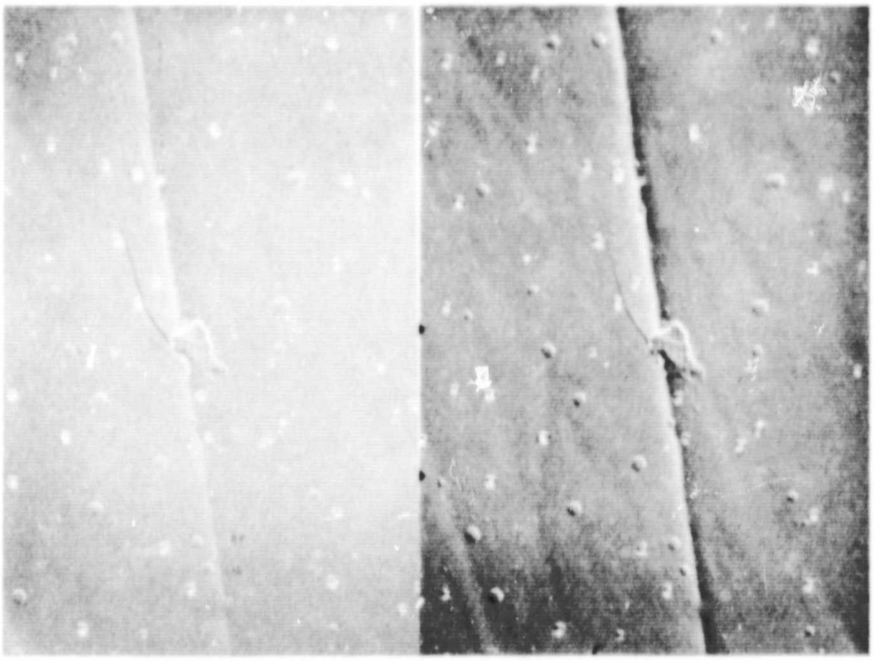


FIGURE 13 STEREO ELECTRON MICROGRAPH OF COATING SURFACE, SPECIMEN 36 (Group IV) X2000

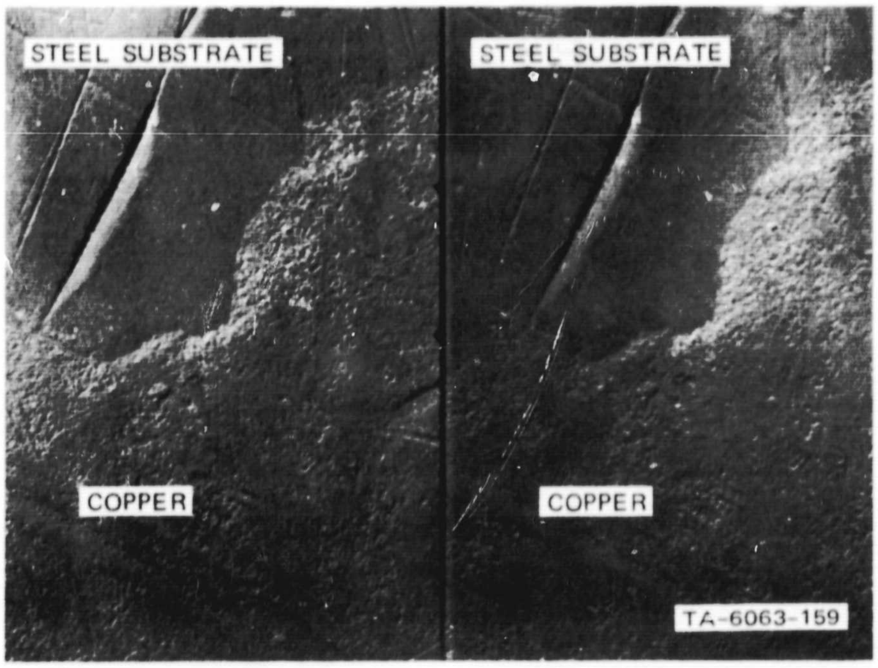


FIGURE 14 STEREO ELECTRON MICROGRAPH OF TAPER SECTION, SPECIMEN 36 (Group IV) X2000

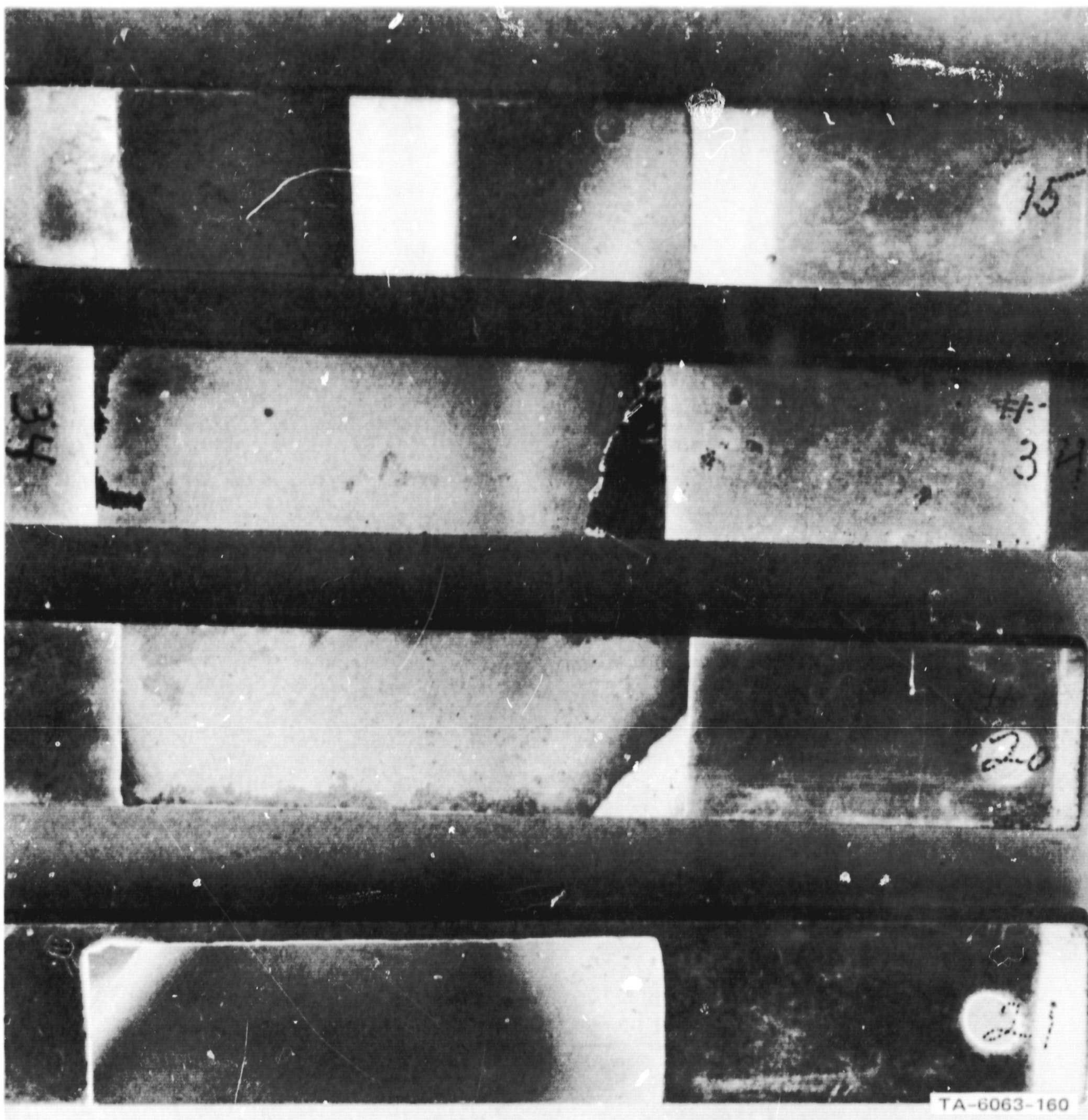


FIGURE 15 TEST SPECIMENS AFTER EXPOSURE TO THERMAL CYCLING

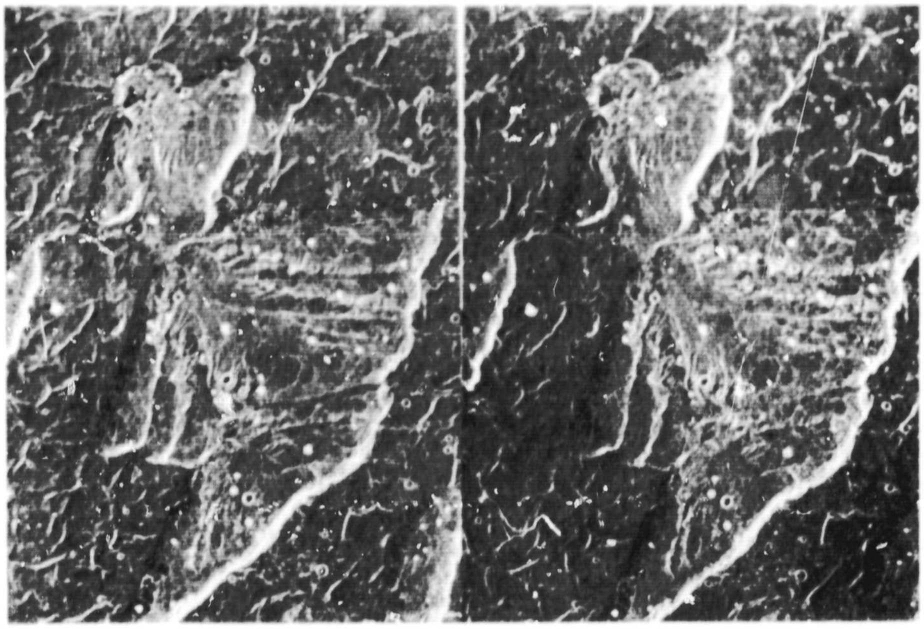
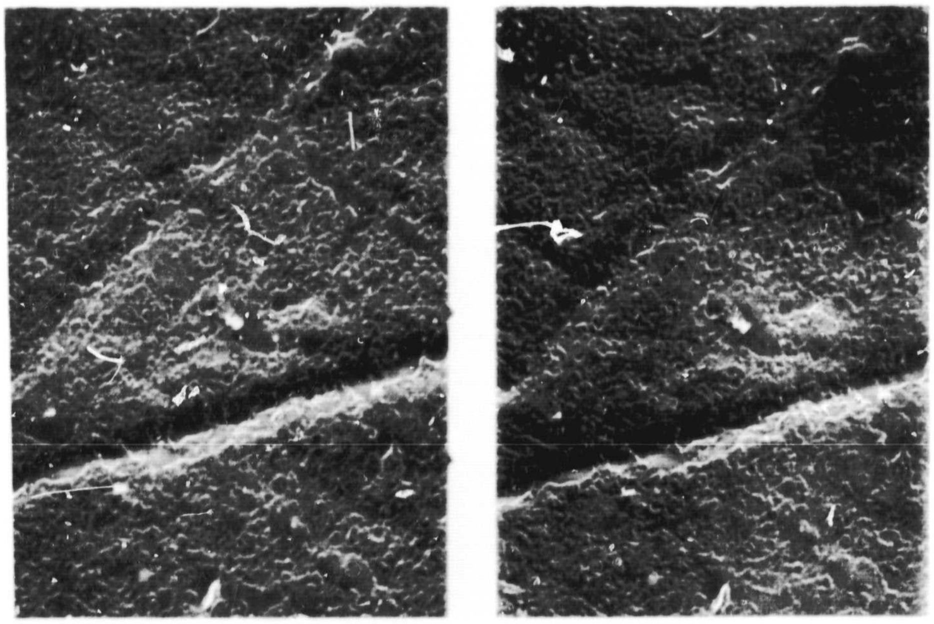


FIGURE 16 STEREO ELECTRON MICROGRAPH OF SPECIMEN 15 (Group I) AFTER THERMAL CYCLING

FIGURE 17 STEREO ELECTRON MICROGRAPH OF SPECIMEN 20 (Group II) AFTER THERMAL CYCLING



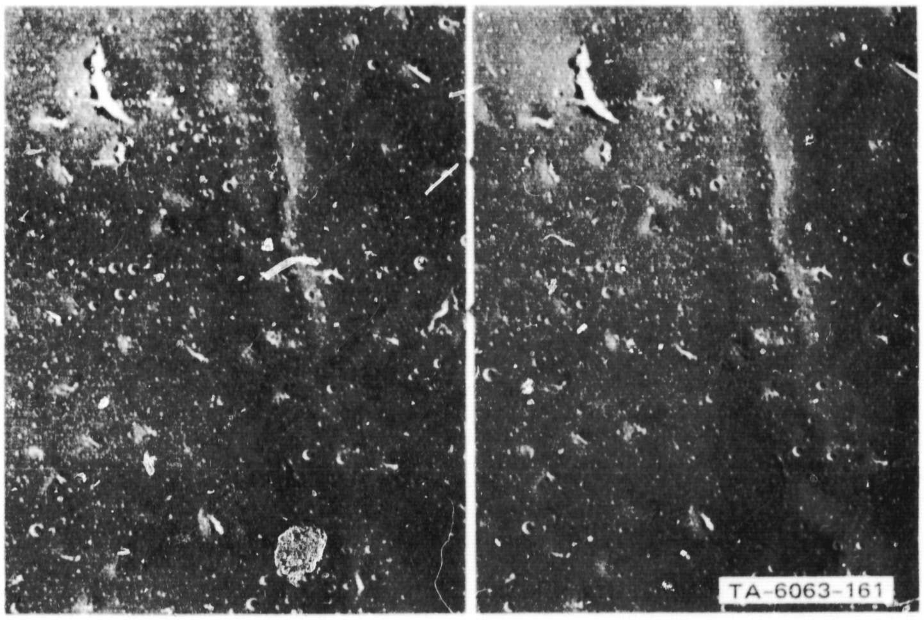


FIGURE 18 STEREO ELECTRON MICROGRAPH OF SPECIMEN 34 (Group IV) AFTER THERMAL CYCLING

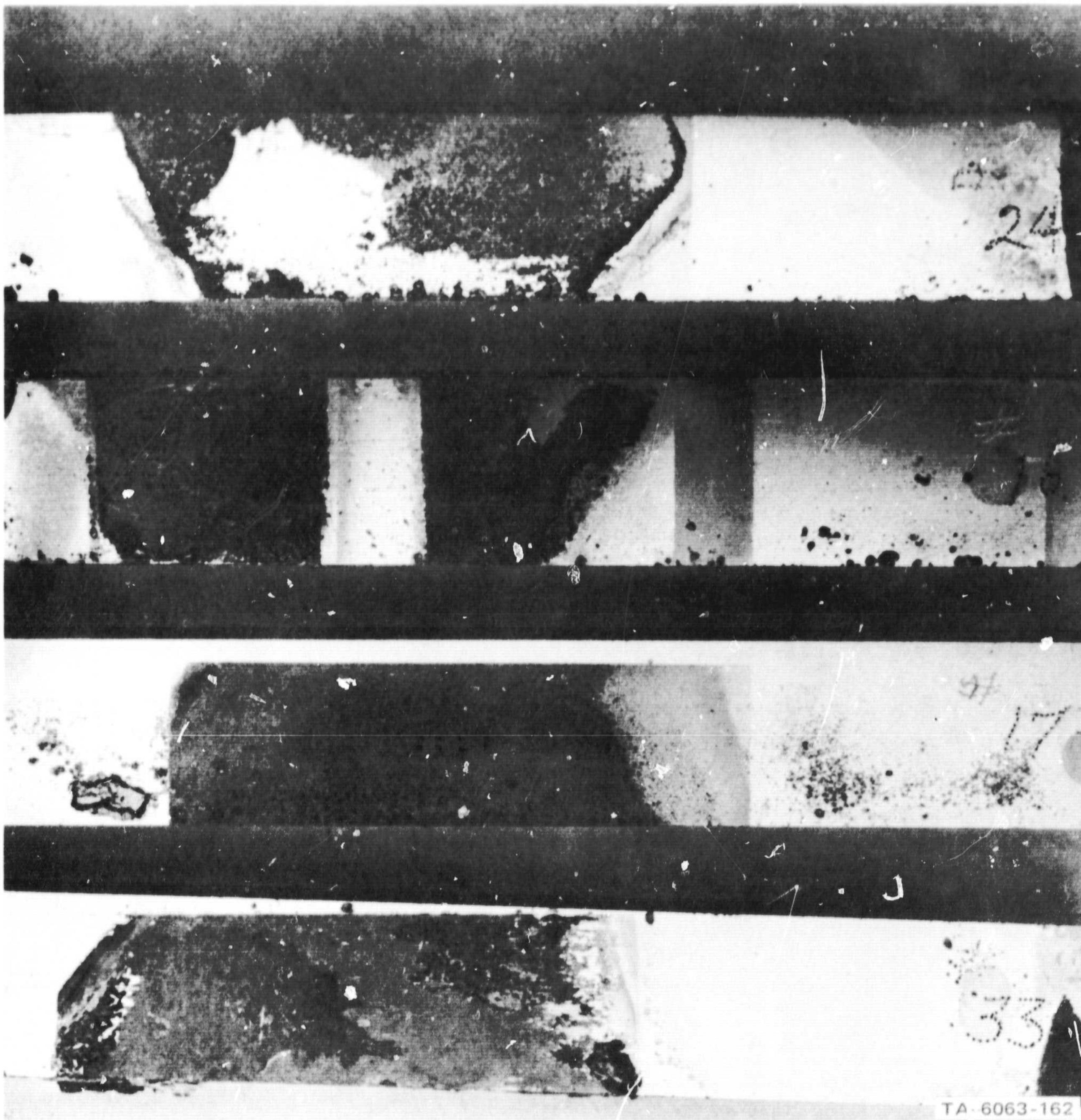


FIGURE 19 SPECIMENS 16, 17, 24, AND 22 AFTER HUMIDITY TEST

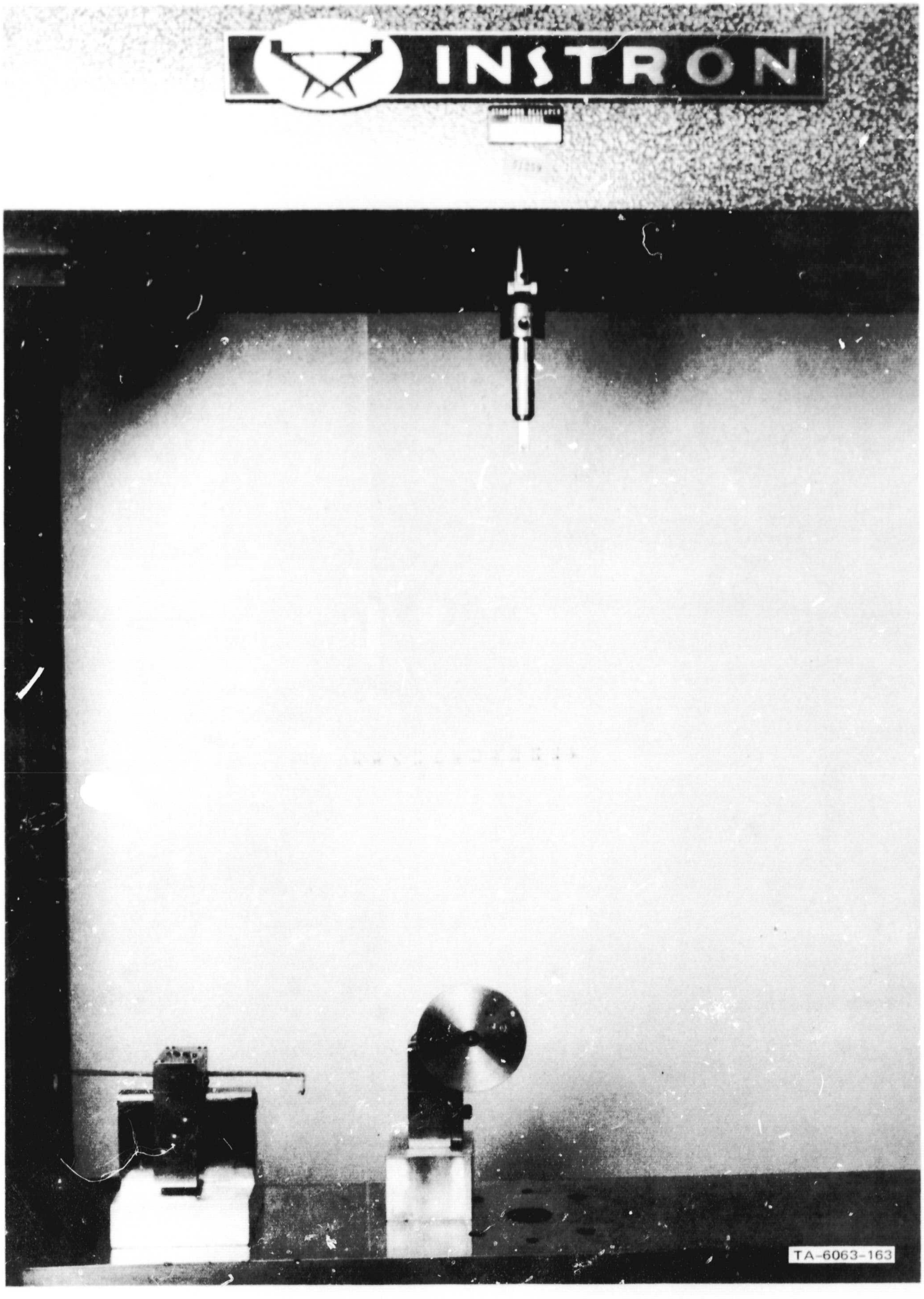


FIGURE 20 FRICTION DEVICE IN INSTRON TESTING MACHINE

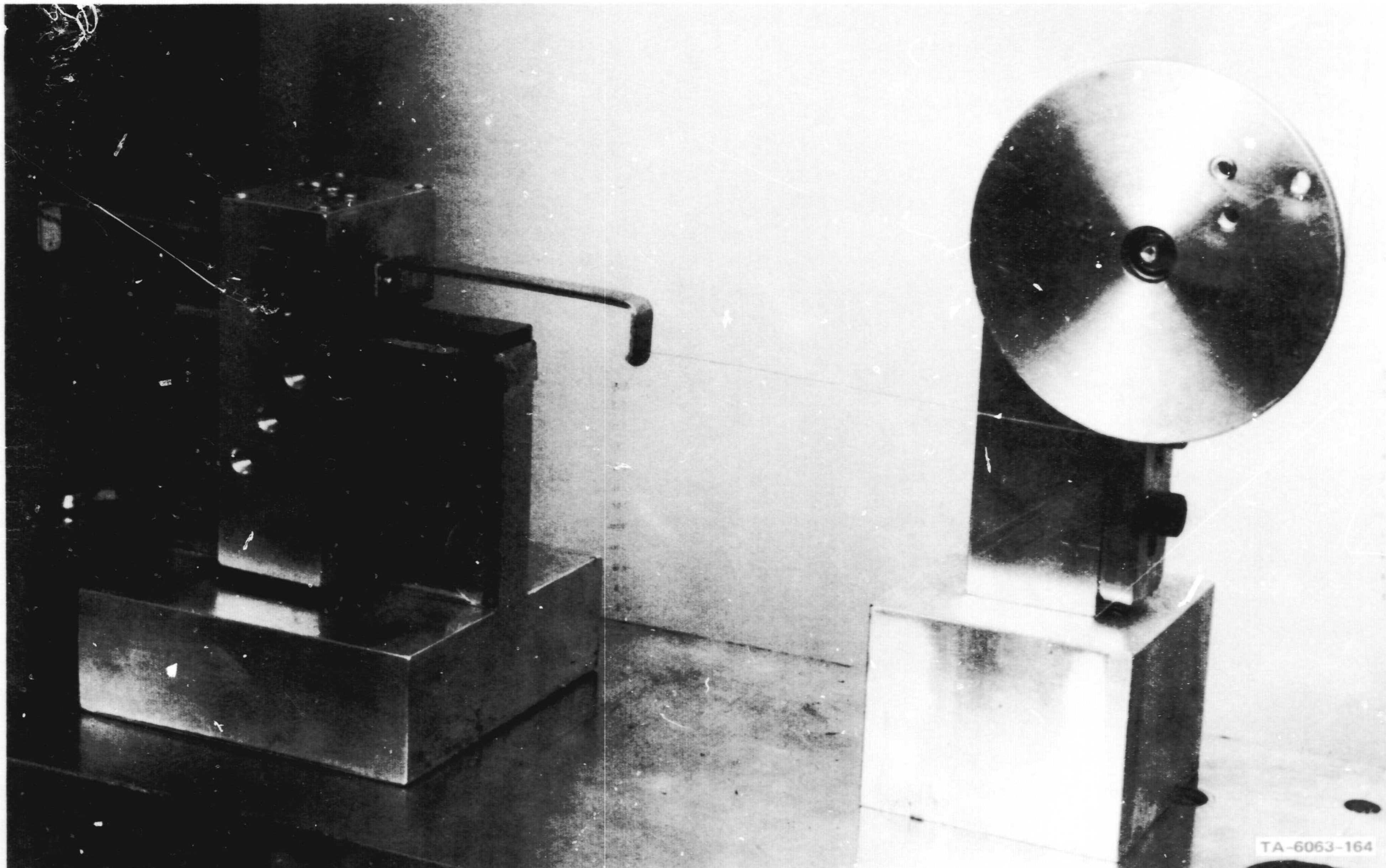


FIGURE 21 FRICTION DEVICE

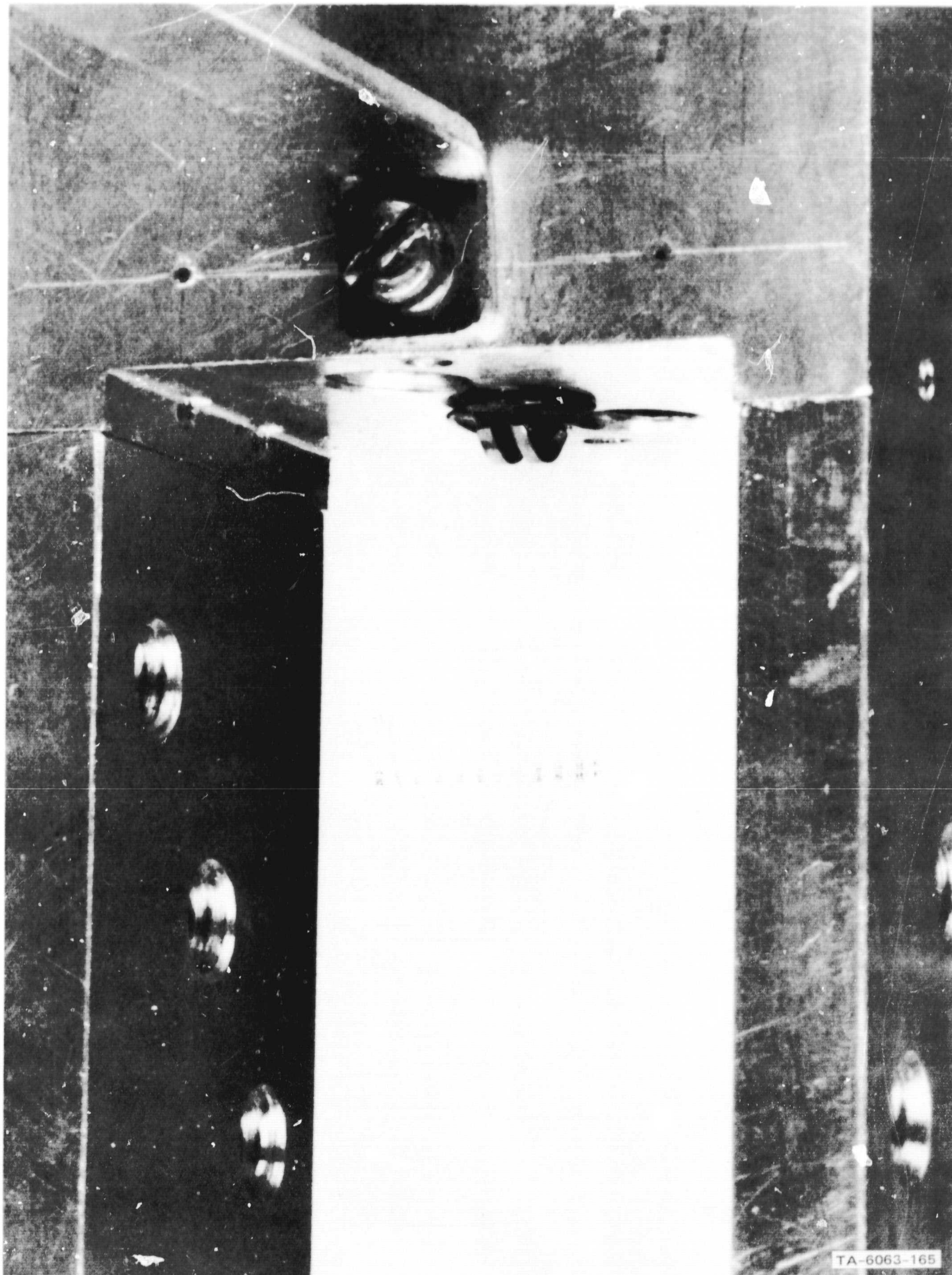


FIGURE 22 FRICTION DEVICE. Rider Detail.

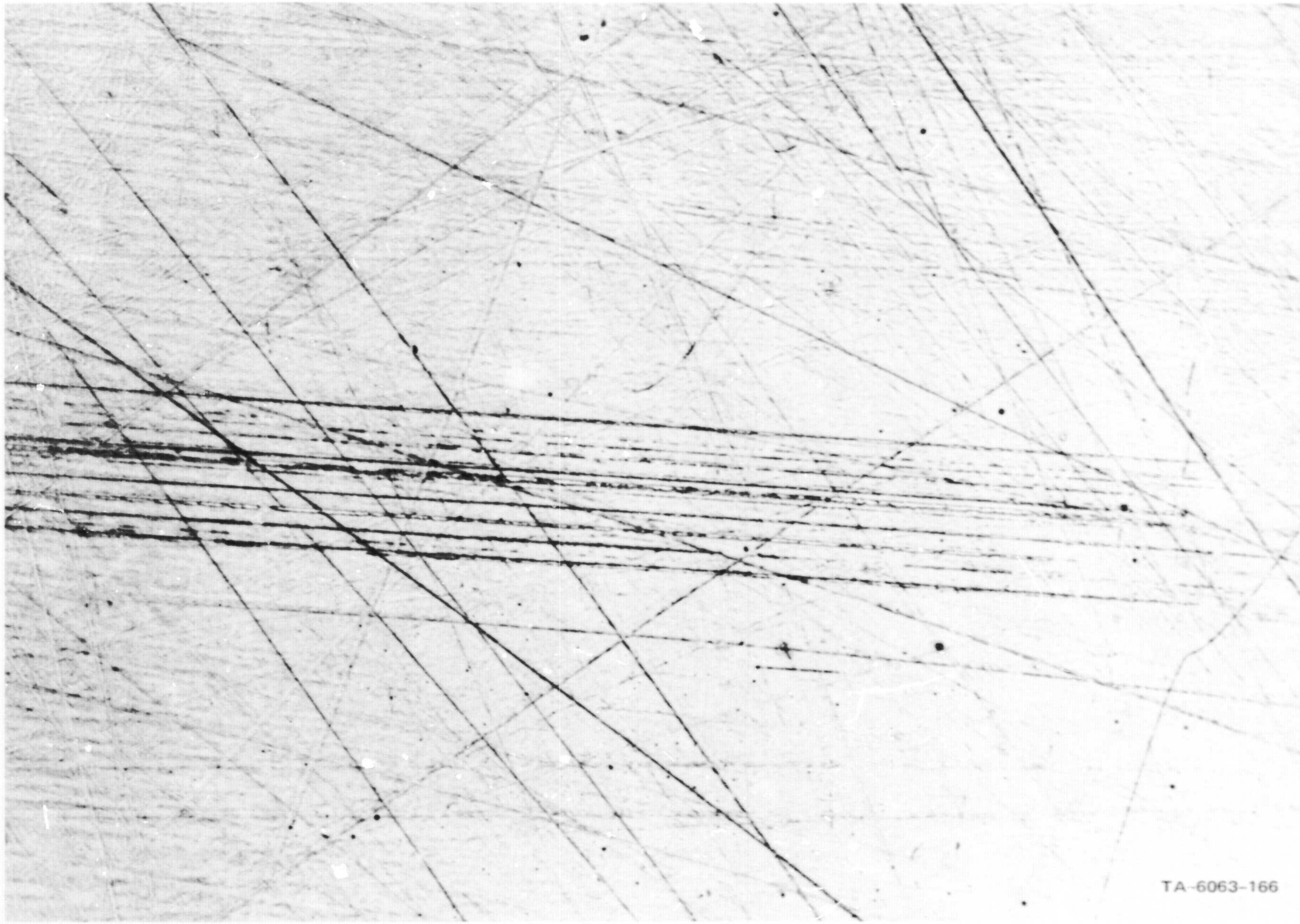


FIGURE 23 FRICTION TRACK ON SPECIMEN 13 (Group I). Rider speed 0.002 in./min. X150.

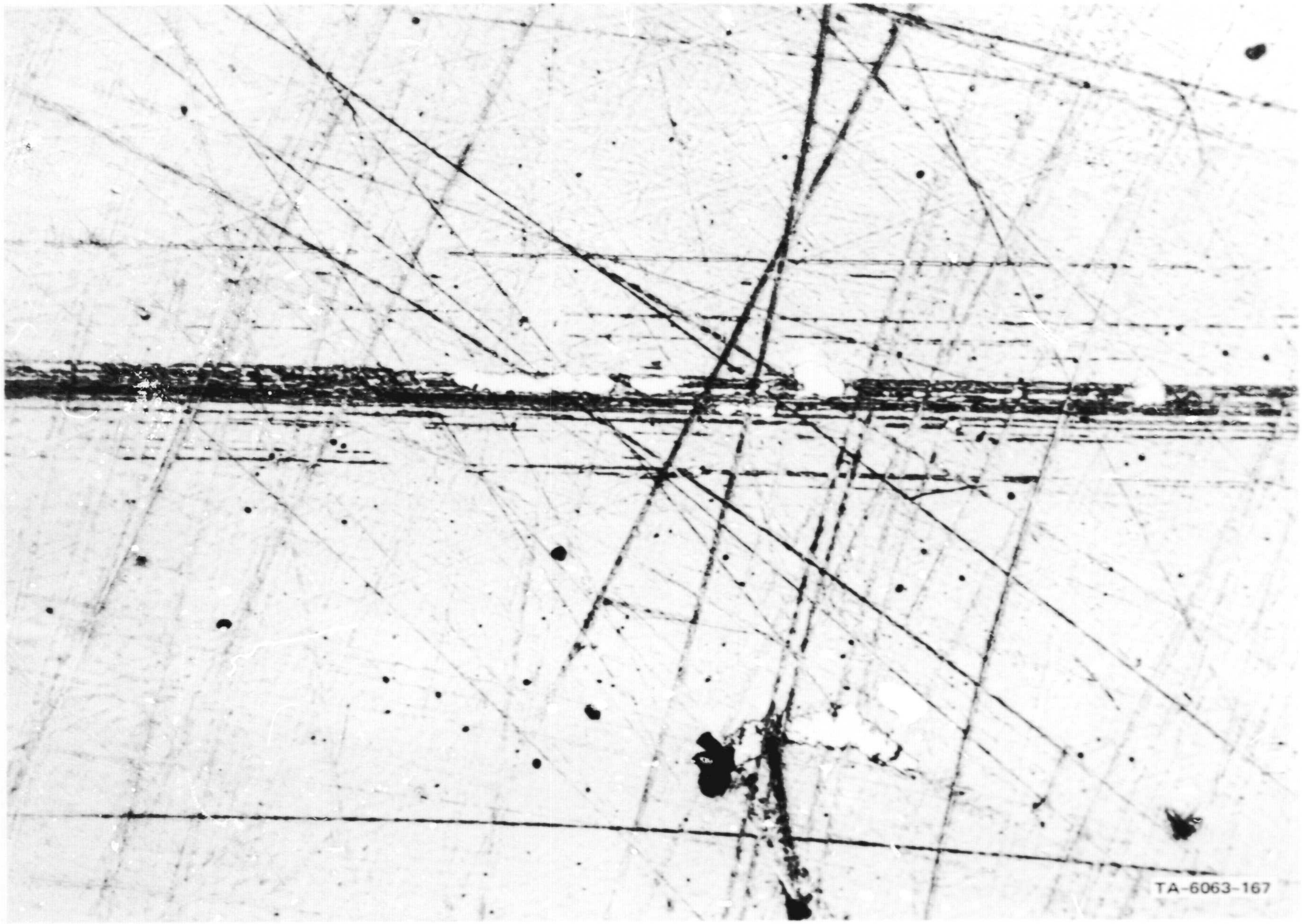


FIGURE 24 FRICTION TRACK ON SPECIMEN 13 (Group I). Rider speed 0.2 in./min. X150

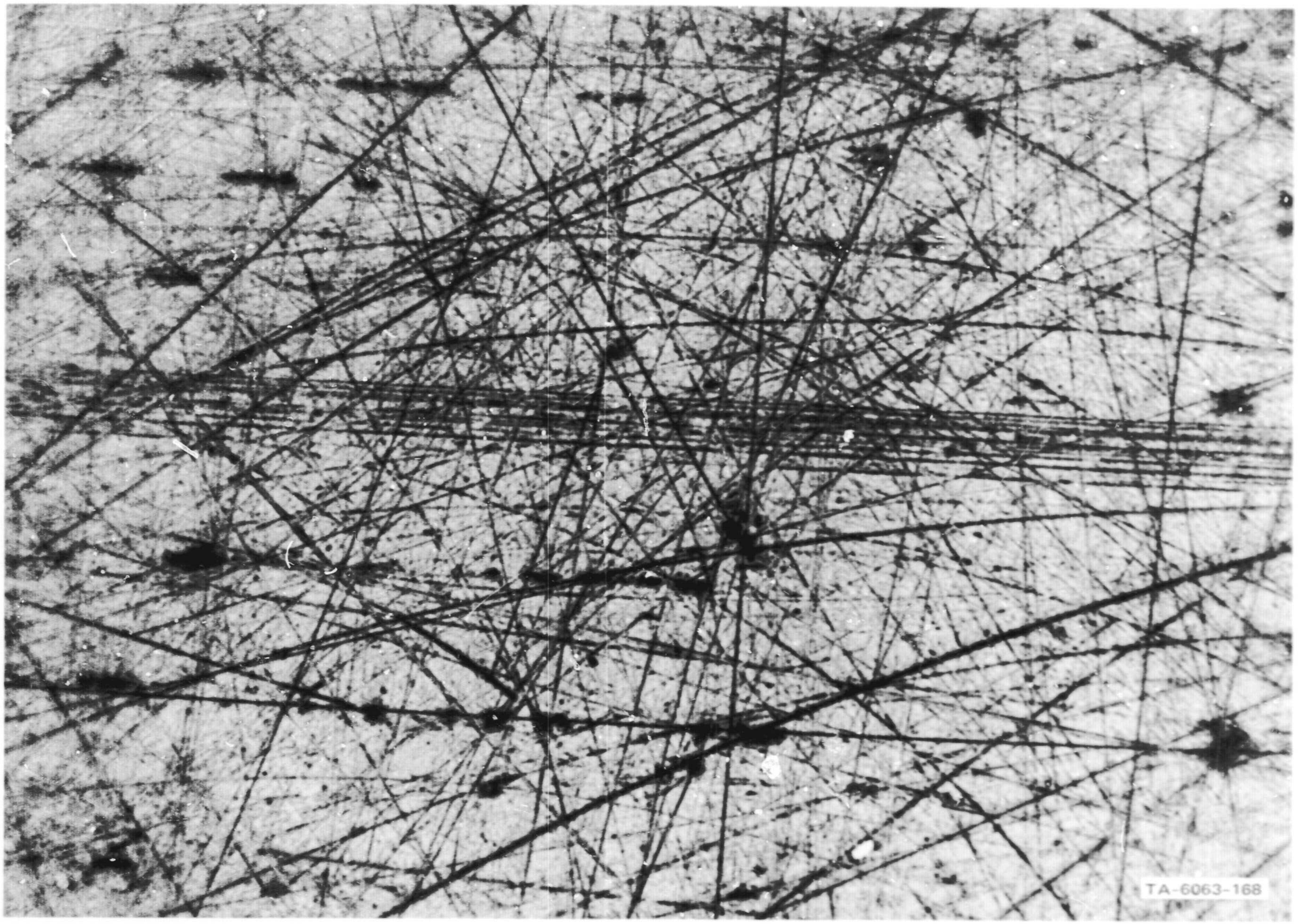


FIGURE 25 FRICTION TRACK ON SPECIMEN 19 (Group II). Rider speed 0.002 in./min. X150

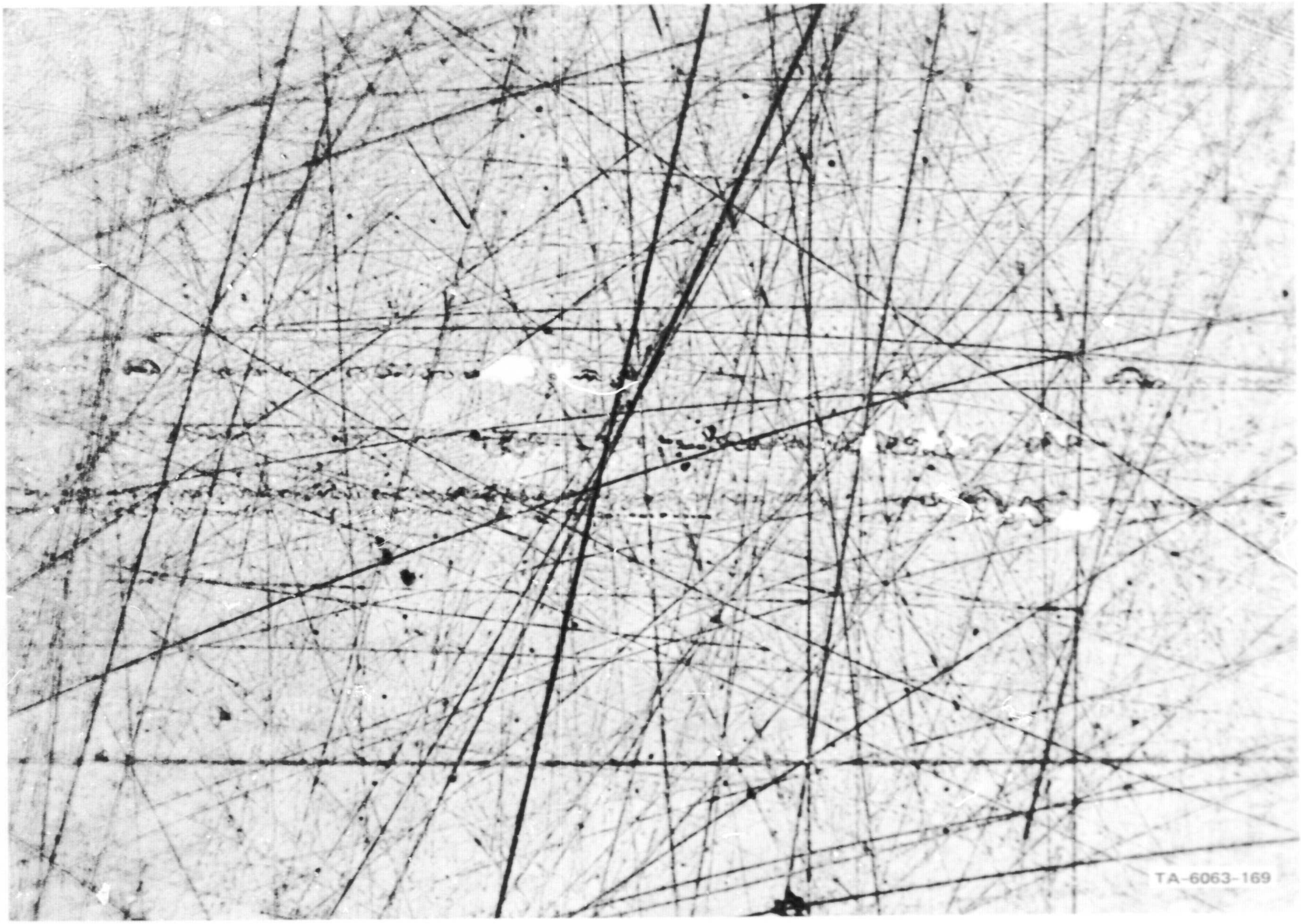


FIGURE 26 FRICTION TRACK ON SPECIMEN 19 (Group II). Rider speed 0.2 in./min. X150

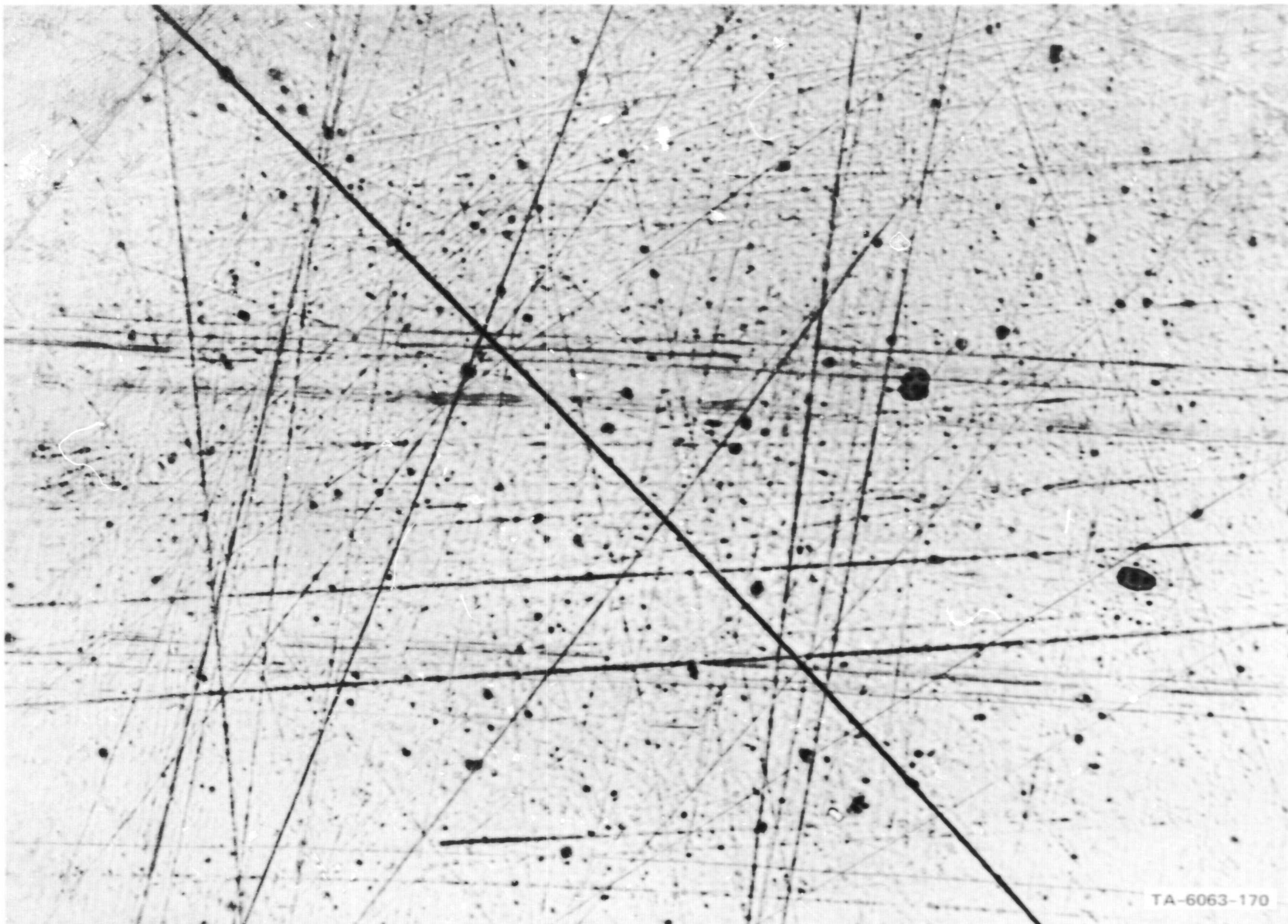


FIGURE 27 FRICTION TRACK ON SPECIMEN 23 (Group III). Rider speed 0.002 in./min. X150

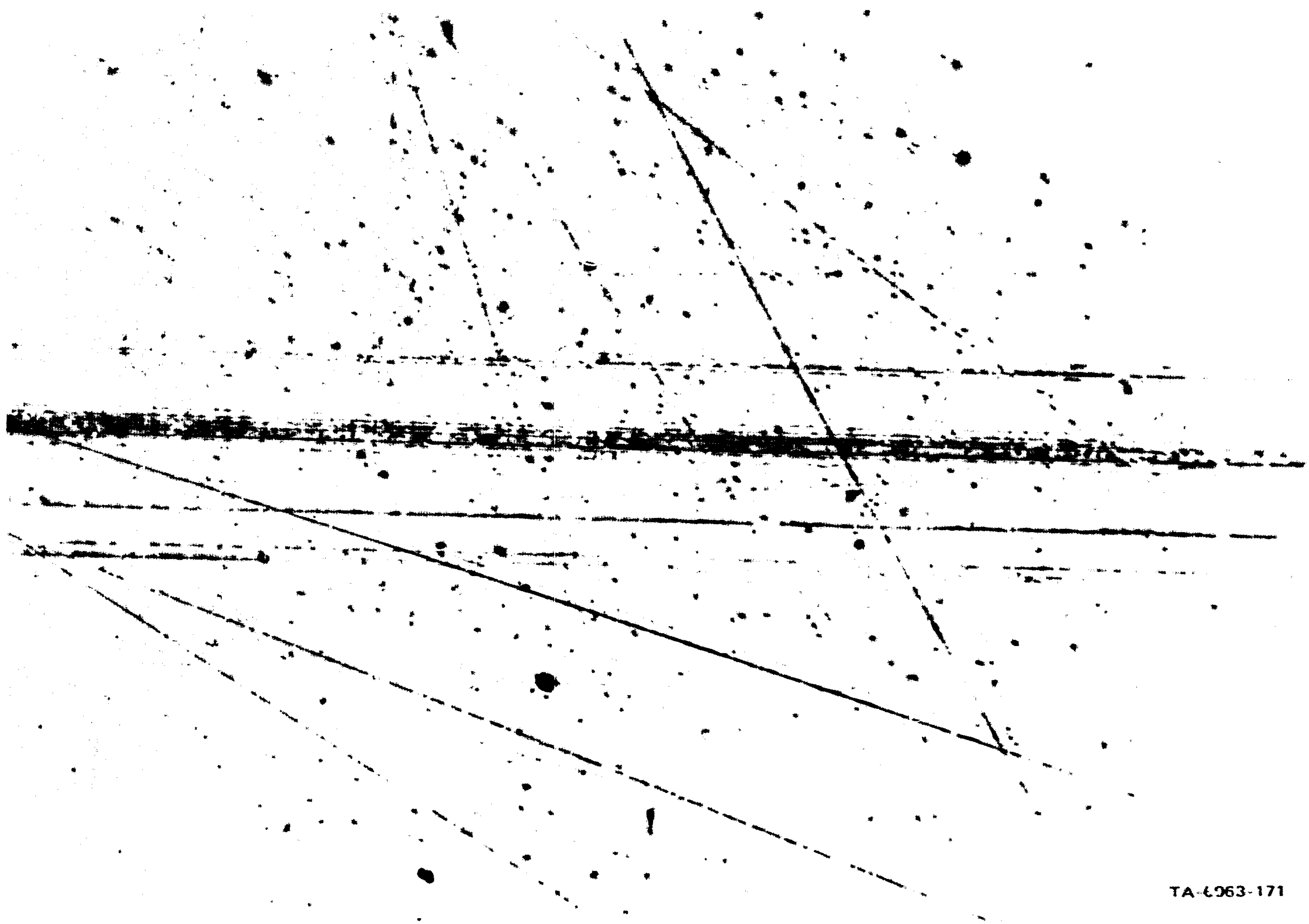


FIGURE 28 FRICTION TRACK ON SPECIMEN 23 (Group III). Rider speed 0.2 in./min. X150

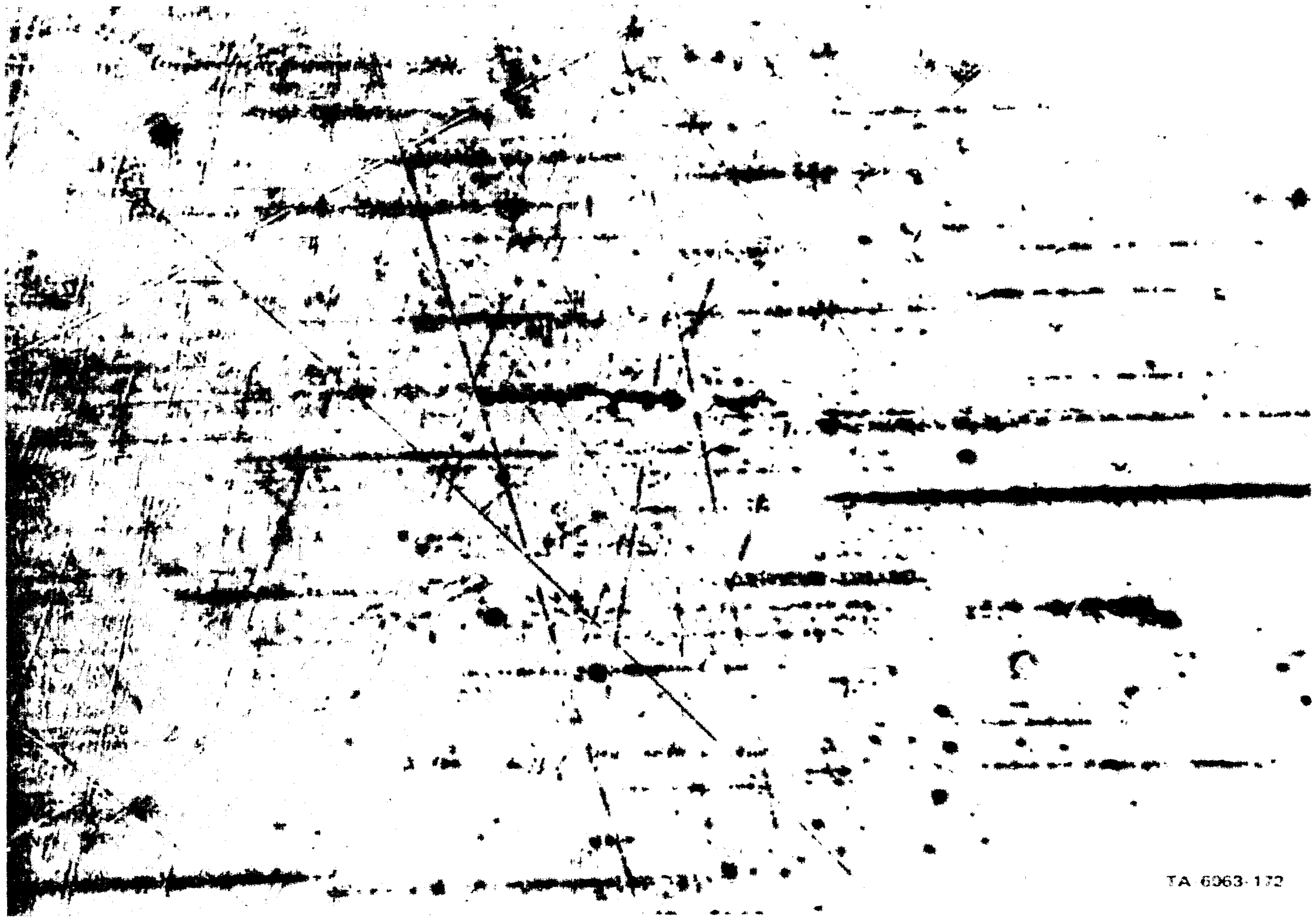


FIGURE 29 FRICTION TRACK ON SPECIMEN 35 (Group IV). Rider speed 0.2 in./min. X150

APPENDIX A

69.4712.5-20

10 September 1969

Mr. L. R. Toth
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, California 91103

Subject: Thin Film Evaluation Tests,
Advanced Spacecraft Valve Technology, NAS 7-717

Gentlemen:

Siloxane coated steel specimens were evaluated at TRW Systems Group in response to your request made at our last progress review. The specimens evaluated were a part of eight groups delivered to you in May 1968. Four groups (I-IV) consisted of 16 coated specimens which were submitted to SRI for evaluation. Four (V-VIII) sets of specimens were evaluated at TRW. Each group is defined as follows:

		Electron Beam		
		Current	Volts	Exposure Time
Group I	Specimens Nos. 13, 14, 15, 16	1 milliamp	3000	2 hrs
Group II	Specimens Nos. 17 through 20	2 milliam	3000	3 hrs
Group III	Specimens Nos. 21 through 24	2 milliamp	3000	3 hrs
Group IV	Specimens Nos. 33 through 36	2.4 milliamp	3000	7.5 hrs
Group V	Specimens Nos. 1 through 4	1 milliamp	2000	5 hrs
Group VI	Specimens Nos. 9 through 12	1 milliamp	2500	4 hrs
	followed by:	1 milliamp	5000	2.5 hrs
Group VII	Specimens Nos 25 through 28	2 milliamp	3000	1.5 hrs
Group VIII	Specimens Nos. 29 through 32	2.5 milliamp	3000	3 hrs

As the above table shows, the difference in specimens of each group was the electron beam current and voltage, and the exposure time. The longer exposure times should result in harder films. The odd numbered specimens were case hardened and all specimens were ground to a 32 rms finish. All the specimens were reportedly made of carbon steel.

Preliminary tests were made on the first batch coatings made in May 1968. These tests indicated poor coatings and it was believed the surface finish may be the cause. Specimens 13 through 36 were then lapped with 600 grit paper, cleaned and coated. During coating of samples 13 through 36, blistering and peeling were evident. The specimens were reprocessed and recoated. The specimens in Groups I through IV were not tested or evaluated prior to submittal to JPL.

TRW evaluated several of the specimens in Group V through VIII as per your instructions given at the last progress meeting. The tests made at TRW consisted of an adhesion test. The procedure used to determine adhesion consisted of applying scotch tape to the coating followed by removal of the tape. The coatings on specimens Nos. 28, 29, 30, and 31 pulled off during this test indicating the coatings were of poor quality.

Specimens Nos. 1 through 4 and 9 through 12 were used in the nitric acid exposure testing using interferometry holography, the results of which are reported in the last final report under Contract NAS 7-436.

In order to determine if the correct process was used in applying the coatings, a second set of specimens were coated. These coatings were applied to low carbon (CRS) steel, as rolled, 321 stainless steel lapped with 300 grit paper, and microscope cover glass substrates. These specimens were tested for adhesion by applying the scotch tape peel test. Also the specimens were exposed to ultrasonic cleaning in Toluene. No evidence of film removal was noted.

It is not known why the inconsistency of the test results between the different batches. The major difference may be the surface finish and method of fabrication. It is possible material from the grinding wheel or grit paper was embedded in the substrate and was not removed during the cleaning operation.

It is recommended a second set of specimens be prepared with adequate attention to surface preparation and re-evaluated. These samples should be selectively evaluated at TRW before delivery and should include the following evaluation.

1. Scotch tape peel test
2. Acid resistance
3. High temperature exposure
4. Ultrasonic in Toluene

An additional test should include the storage of thin film coated specimens (preferably stainless steel, aluminum and titanium) in liquid OF_2 and liquid B_2H_6 propellants. This test would be important in evaluating reactivity and protectivity of these films with the reactive propellants.

Sincerely,

R. J. Salvinski,
Program Manager
Contract NAS 7-717

RJS/prp

cc R. L. Hammel