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CYCLE TESTING OF ACS VALVES IN CLF_5
FINAL REPORT

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AUGUST 1972

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This report documents the complete test program conducted ~~by the AFRPL~~ to establish a seat and poppet material(s) for use in chlorine pentafluoride (ClF_5) attitude control system (ACS) valves. Test results of iron, nickel, tungsten, boron and zirconium based materials in the flat-on-flat sealing concept are discussed. Additionally, a sapphire/copper lip-seal concept and thin coatings of aluminum, tungsten and chromium on substrates, previously proved to be incompatible, were evaluated. In the tests conducted under this program, two carbides of tungsten and one of boron have successfully met the helium leakage requirement after 100,000 actuations in the ClF_5 environment.

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FOREWORD

This report was prepared by the Engine Components Branch, Liquid Rocket Division, Air Force Rocket Propulsion Laboratory. The subject tests were conducted under Project 305810RP, "Attitude Control Component Evaluation Program," from February 1968 to October 1971, with Sgt G. J. Gunderson as Project Engineer and Mr. R. R. Rittel as Test Engineer. The work described herein completes the current program whose objective has been to establish a seat and poppet material for use in chlorine pentafluoride (ClF_5, CPF) attitude control systems. The author wishes to specifically acknowledge and express his appreciation to Mr. Rittel and TSgt T. J. Fellows for their assistance in conducting the entire test program. Special thanks are also extended to Sgt G. S. Whiting for his pre- and post-test photographic support, and to Mr. H. Blazek of the China Lake Naval Weapons Center for the enthusiastic metallic coatings support that he provided.

This technical report has been reviewed and is approved.

HOWARD V. MAIN
Chief, Engine Components Branch

ABSTRACT

This report documents the complete test program conducted by the AFRPL to establish a seat and poppet material(s) for use in chlorine pentafluoride (ClF_5) attitude control system (ACS) valves. Test results of iron, nickel, tungsten, boron and zirconium based materials in the flat-on-flat sealing concept are discussed. Additionally, a sapphire/copper lip-seal concept and thin coatings of aluminum, tungsten and chromium on substrates, previously proved to be incompatible, were evaluated. In the tests conducted under this program, two carbides of tungsten and one of boron have successfully met the helium leakage requirement after 100,000 actuations in the ClF_5 environment.

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SECTION I
INTRODUCTION

AFRPL effort to develop the component technology needed for high-energy propulsion systems led to Air Force Contract F04611-68-C-0074, "Advanced ACS Valve Development Program," with the Marquardt Corporation in February 1968. Complementing this effort was the original AFRPL in-house test program (Project 305810RP) whose objective then was to provide general performance data on ACS valves and filter elements for use in chlorine pentafluoride (ClF_5), monomethylhydrazine (MMH) and hydrazine (N_2H_4), and to update design specifications for future attitude control system (ACS) valve procurements. Initial results of that in-house testing are documented in AFRPL-TM-69-14, "Hydraulic Research and Manufacturing Company Mono-Propellant (Post-Boost) ACS Valve Test," dated August 1969 (Reference 1). It was during this time period early in the AFRPL test program that the magnetically linked bipropellant valve under development by Marquardt encountered a severe seat/poppet leakage problem when cycled in ClF_5. A contract amendment requiring evaluation of several different seat and poppet materials failed to solve that problem. Table I in Section VI of this report summarizes the Marquardt test results.

Unreliable mechanical features that had resulted in past valve failures during engine development programs were eliminated with the current Marquardt design (Figure 1).* Whereas poppet/seat closure is commonly accomplished in other designs via thin-walled torque tubes or sliding fits, the only moving part in the Marquardt design is the armature/poppet assembly. Possibilities of failures from torque tube fracture or contamination problems associated with tight tolerances of sliding assemblies are virtually nonexistent.

*Figures are located sequentially on pages 45 through 84.

Cycle tests in MMH, N_2H_4 and N_2O_4 at Marquardt (Reference 2) demonstrated the valve's ability to meet all design objectives (Table II) except the internal helium leakage requirement after 100,000 cycles in ClF_5. The bipropellant valve was cycled successfully (100,000 times) in the MMH/N_2O_4 propellant combination without developing seat leakage in excess of the design goal. For these tests the original seat and poppet materials (Pyromet X-15 and Duranickel 301 on the fuel and oxidizer sides, respectively) were used. The Pyromet X-15 material had also undergone 100,000 cycles in N_2H_4 without excessive leakage. In addition, the valve has been subjected to a series of 10 engine tests at the 90-pound thrust level utilizing the cryogenic FLOX (80 percent F, 20 percent O) and methane propellant combination (Reference 2). Again, the Pyromet X-15/ Duranickel 301 original materials were used for these cryogenic engine firings.

Based upon the original valve failures in ClF_5, the objective of the AFRPL in-house Attitude Control Component Evaluation Program (ACCEP) has been to establish a seat and poppet material(s) and/or new sealing concept for use in ClF_5 ACS valves. This final report documents all valve cycle tests in ClF_5 conducted under the program.

SECTION II
APPROACH

Based upon the unsuccessful early test results at Marquardt (Reference 2), three potential approaches to the problem were investigated under this program. They included:

1. Continue testing seats/poppets fabricated from different materials, but still in the original flat-on-flat sealing configuration. The choice of candidate materials selected for testing depended initially upon the most promising of the original Marquardt tests and finally upon further Marquardt work being performed simultaneously under Air Force Contract F04611-70-C-0052 (Reference 3).

2. Utilize different mechanisms to accomplish the poppet/seat seal. A lip-seal concept incorporating a spherical sapphire poppet mating to a cylindrical copper seat in a Moog, Inc., valve was tested in ClF_5. Modifications to that design were also considered.

3. Metallic coatings onto existing seat and poppet hardware to improve their ability to withstand the dynamic valve impact in ClF_5.

SECTION III
SUMMARY

The materials best suited for ClF_5 ACS valve application are tungsten carbide (with either a nickel or cobalt binder) and boron carbide (binderless). These are the only materials which demonstrated 100,000 actuations (and more) in ClF_5 and maintained acceptable leakage rates. The cycle life of materials undergoing evaluation in this program was increased by two pre-test procedures. Before final assembly, the valve poppet was precisely aligned for more perfectly normal seat contact, and after assembly, the entire valve was baked at 160°F for 4 hours.

The poppet realignment step was incorporated as a result of observations of localized wear on one portion of the poppet sealing area. When this uneven wear increased between tests three and five, the realignment step was made procedural before each new material evaluation. The realignment could be made quickly and effectively to within 0.5 minute on an instrument that was developed as part of a supporting contractual effort (Reference 3).

While the catastrophic effect of moisture contamination with hydrofluoric acid (HF) formation in all fluorinated oxidizer systems has long been recognized, its effect upon the candidate seat and poppet materials was not fully appreciated. At the microscopic level, residual moisture in the pores of materials can react to form HF upon exposure to passivating fluorine or ClF_5, and thus degrade the sealing surfaces. Driving this moisture out of the pores of the seat and poppet materials undoubtedly has played a significant role in improving the cycle life of the valve.

Two identical designs of a lip-seal configuration evaluated for ClF_5 service in a Moog valve failed the leakage requirement within 5000 cycles. A potential re-design was hampered by precision in-house fabrication problems and was never evaluated.

A considerable amount of effort was expended in the metallic coatings investigation for the existing seat and poppet hardware. Although none of the process/material combinations that were tested completely met the program objectives, the problem appears to be one of material compatibility rather than coating technique. The Cathode Sputtering Process was shown to be most effective for this particular application.

SECTION IV
TEST VALVE DESCRIPTIONS

MARQUARDT VALVE (FLAT-ON-FLAT MATERIALS TESTING)

The hardware delivered by Marquardt at the conclusion of their "Advanced ACS Valve Development Program" (Reference 2) constituted the workhorse test beds for all flat-on-flat seat and poppet material evaluation in chlorine pentafluoride. The performance requirements and operating characteristics for the magnetically linked bipropellant valve that resulted from that contractual effort are shown in Table II, page 27. That bipropellant valve is pictured in Figure 2 in its disassembled state.

A single-armature "component" valve (Figure 3) that is essentially half of the bipropellant valve was also used to evaluate candidate materials under the same impact and cycle rate conditions. For test purposes, the valves were cycled at a frequency of 8 to 12 Hz, and impact forces were measured (Reference 4) to be approximately 30 pounds force in water at a 450 psig inlet pressure. The same performance requirements depicted in Table II for the bipropellant valve are applicable to the component valve in the single propellant flow situation (except response mismatch of course). Internal design of the component valve is identical to one side of the bipropellant valve which is depicted schematically in Figure 1.

MOOG VALVE (LIP-SEAL TESTING)

A Moog, bipropellant valve (Model 53 X 120A) was used to evaluate a lip-seal design in chlorine pentafluoride. Unlike the Marquardt valve, this valve utilized a torque motor, which accomplishes poppet motion via the movement of a flexure tube. The internal design of the Moog valve used in this program is shown in Figure 4. The major performance characteristics of this valve are very similar to those of the Marquardt bipropellant valve. It is sized to deliver approximately 75 pounds of thrust at nominal flow rates of 0.135 lb/sec ClF_5 and 0.068 lb/sec MMH.

SECTION V
TEST SYSTEM DESCRIPTION

The closed-loop ClF_5 flow system (Figure 5) located in Cell E of Test Area 1-14 at the Air Force Rocket Propulsion Laboratory was used for all cycle testing conducted under this in-house project. Figure 6 is a schematic representation of that test system.

The capacity of the stainless steel run tank was 5.5 gallons while the ClF_5 catch tank was 6.0 gallons. Liquid ClF_5 level in the run tank was monitored by a radiation source and detector installed near the bottom of the tank. When the level of the ClF_5 liquid in the run tank became sufficiently low (the tank was never emptied completely during test), the propellant was transferred with helium pressurization from the catch tank back through a bypass line to the run tank. Maximum operating pressure of the flow loop was 500 psig.

Propellant run lines were also stainless steel and ClF_5 flow control was accomplished with remotely operated copper-seated Annin valves. In the test section itself, hand-operated control component valves were used for test item isolation during in situ helium leakage measurements. (For a discussion of leak measurement techniques, see Appendix B.) These control component valves were never operated in the propellant environment. Helium gas was used for all test system pressurization and test section purges. Additionally, a gaseous fluorine K bottle was plumbed into the system for test item passivation prior to each test.

Chlorine pentafluoride flow through the system was from the run tank, through a 10-micron (nominal) filter, a turbine flowmeter, the test valve itself and a throttling valve, and into the catch tank. Pressure transducers were located on the tanks and test section to monitor inlet pressures and pressure drops. A flowmeter upstream of the test section measured liquid flowrate through the test valve.

A charcoal reactor processed all vapors vented by the tanks and all line purges prior to release into the atmosphere. Television cameras provided visual monitoring of the effluent vapors from this reactor as well as the test system during operation. Strict safety precautions were followed at all times during operation and maintenance of the system. A more detailed description of the operational steps taken in conducting a seat/poppet material evaluation under dynamic impact in ClF_5 can be found in Appendix A of this report.

SECTION VI
RESULTS OF FLAT-ON-FLAT MATERIALS EVALUATION

CRITERIA/SEAT AND POPPET DESIGN

Marquardt's selection of the all-metal, flat-on-flat sealing concept for this ACS bipropellant valve (Reference 2) was based upon their past experience and a previous AFRPL program with Rocketdyne (Reference 5). Metal seat and poppet materials were originally chosen because they were considered more compatible (at least in static environments) with ClF_5 and MMH and allowed higher operating temperatures. Historically, metals have offered greater shelf and cycle life than plastic or elastomeric materials, though recent advances in soft-seat technology (Reference 6) have been significant. Spherical or conical sealing configurations were not selected because of the difficulties inherent in producing the very smooth surface finishes necessary to achieve low leakage on the nonflat geometry. Current specifications for surface finish on sealing areas (Figure 7) require final lapping to 1 micro-inch arithmetic average (AA) or less roughness and 12 micro-inch (one helium light band on the interference microscope) flatness.

CRITERIA/HELIUM LEAKAGE

Potential mission applications utilizing high-energy propellants such as chlorine pentafluoride, necessitate zero liquid leakage requirements for system components. For this reason, the true success criteria (with regard to internal seat leakage) for the subject Marquardt ACS valve is zero ClF_5 liquid at 450 psig inlet pressure; however, the lack of an acceptable test procedure for the precise measurement of small amounts of liquid ClF_5 leakage that was known to be quick, reliable and above all safe, resulted in the adoption of more conventional techniques utilizing helium gas.

The selection of 30 scc of helium per hour at 450 psig to represent that zero ClF_5 liquid leakage should be recognized as a "ball park" design goal. The decision was based upon a consensus of opinion of the original valve development personnel from Marquardt and AFRPL with reference to the works of Marr (Reference 7) and Weiner (Reference 8). The number 30 is likely to be somewhat conservative for at least two reasons: (1) it is based upon flow equations that assume a single, cylindrical leak path of uniform cross-sectional area, and (2) though it accounts for density, pressure differential and viscosity effects, it neglects the resistance to liquid flow through small openings due to surface tension. For these reasons, the internal helium leakage requirement of 30 scc/hour is somewhat arbitrary and should be regarded as a goal, not an absolute requirement.

MARQUARDT TEST RESULTS

Based upon studies of chemical, mechanical, magnetic and physical properties of materials, Duranickel 301 and Pyromet X-15 were originally selected for the oxidizer (ClF_5) and fuel (MMH) sides of the bipropellant valve, respectively. Of these materials, the Pyromet X-15 successfully underwent 100,000 actuations in MMH at Marquardt with helium leakage remaining well below 30 scc/hour (Reference 2).

As a result of the initial failures of the Duranickel 301 material upon cycling in ClF_5, several other seat and poppet materials were tried by Marquardt, without success, in an attempt to meet the leakage requirement. Table I summarizes the Marquardt tests.

The extreme hardness of the tungsten carbides (Reference 9) as evidenced by their many wear-resistant applications (metal-working dies, tool tips, mining equipment, etc.) made them logical candidates for the seat and poppet. The Marquardt investigation of the K-602 materials was hampered by porosity problems, possibly due to the lack of binder material.

Marquardt's initial tests of the K-96 material (6 percent cobalt binder) were terminated after passivation, when leakage rates began to increase rapidly. Upon disassembly, the sealing surfaces were found to be etched and covered with deposits approximately 12 micro-inches thick. Although moisture contamination was thought to be the cause of the failure, its source could not be identified with certainty. Further tests of the K-96 material at Marquardt were halted after about 1000 cycles in ClF_5 when leakage rates increased to out-of-specification conditions. The relatively low leakages of 50 and 70 scc/hr were the best that had been obtained to that time. Marquardt's inspection of those seats and poppets revealed that their sealing surfaces were not affected to as great an extent as the previous materials had been.

The Marquardt effort was concluded at that time without further testing of the K-96 tungsten carbide material. Residual hardware was delivered to the AFRPL for evaluation tests in ClF_5 under the in-house test program that has recently been concluded. What follows is a discussion of the tests conducted at the Air Force Rocket Propulsion Laboratory to solve the ClF_5 ACS valve leakage problem.

AFRPL TEST RESULTS

Test No. 1/K-96 Tungsten Carbide/Component Valve/ClF_5

The first seat and poppet material to be subjected to the ClF_5 environment under this test program was K-96 tungsten carbide. During this test, a seat and poppet that had previously undergone 215,000 cycles in gaseous fluorine (without excessive leakage) was cycled 40,000 times in ClF_5. Restricted ClF_5 flow due to plugging of a valve inlet filter caused the test to be halted. Leakage measurements taken after removal from the test system were acceptable and inspection showed the sealing surfaces not to be appreciably degraded.

For further evaluation in test No. 2, the valve was cleaned and reassembled with the same set of K-96 seat and poppet, and a new inlet filter was installed.

Test No. 2/K-96 Tungsten Carbide/Component Valve/ClF_5

Test No. 2 was halted after approximately 15,000 cycles when the leakage rate had increased from 20 to 103 scc helium/hour. Upon disassembly and inspection, particles were discovered on the sealing interface. Chemical analysis of these particles showed them to be cobalt. The source of the cobalt particles could not be precisely determined. Possibly, they originated from the seats and poppets (cobalt binder) themselves. This explanation is reasonable for two reasons. First, the system and valve inlet filters would trap any foreign matter originating from lines, fittings and tanks, and would prevent particle recycling. Second, this particular seat and poppet set had now been cycled in excess of 250,000 times. After that many cycles, a "loosening" of the tungsten carbide/cobalt matrix at the impact surface might be expected, with some resultant particle release. This represented the first and only such instance of cobalt particle entrapment on the sealing surface that caused excessive leakage.

Test No. 3/K-96 Tungsten Carbide/Bipropellant Valve/ClF_5

A new K-96 seat and poppet (fabricated to fit the bipropellant valve) was installed for test No. 3. After a total of 50,000 cycles in ClF_5, the test was halted and the valve disassembled for purposes of seat and poppet examination. No particulate matter (cobalt or otherwise) could be found on the sealing surfaces, though exaggerated wear on one side of the poppet was noted. Despite the fact that helium leakage remained at an acceptable 31 scc/hour before disassembly, inability to reassemble the valve with subject seat and poppet in precisely the same wear-pattern position prevented testing beyond 50,000 cycles. The exaggerated wear pattern on the poppet led to the suspicion of a minor poppet/seat misalignment of the bipropellant valve.

Test No. 4/Duranickel 301/Component Valve/ClF_5

This test was conducted in support of a parallel program directed by the Air Force Materials Laboratory to define the failure mechanism of the subject Marquardt valve in ClF_5 service (Reference 10). The principal investigators involved in that effort requested a Duranickel 301 seat/poppet specimen (the original material) which exhibited the failure mode for their analysis. To meet that request, a seat and poppet of the subject material was cycled 6000 times in ClF_5 with resultant helium leakage of about 450 scc/hour. To minimize contamination (water or otherwise) the valve was disassembled and the failed seat and poppet set packaged in polyethylene under a dry nitrogen atmosphere in a glove bag. The parts were then delivered to the investigators for their analysis.

Test No. 5/K-96 Tungsten Carbide/Bipropellant Valve/ClF_5

The bipropellant valve previously used in test No. 3 was assembled with a newly re-lapped K-96 seat and poppet for this test. The test objective was to duplicate the results of test No. 3 for the first 50,000 cycles and continue cycling to the design goal of 100,000 actuations provided that periodic leakage measurements remained acceptable.

After passivation, internal helium leakage measured 400 scc/hour. Dry cycling the valve at this point approximately 100 times (with intermittent leak checks in situ) caused the leakage to decrease steadily to about 62 scc/hour. To conserve test time, the decision was made to begin cycling in ClF_5, even with the leakage rate at this level (approximately twice the requirement). After 6500 cycles in ClF_5, a leakage measurement was taken to observe any change from the initial 62 scc/hour.

No pressure rise was detected in the 10-minute leak check (Appendix B) and cycling was resumed to a total of 25,000 cycles in ClF_5. At this point, leakage measured approximately 200 scc helium/hour, and dry cycling 50 times reduced the leakage to 123 scc helium/hour. Again, the decision was made to continue ClF_5 cycling (despite the excess leakage) to the 50,000 actuation mark. After 52,000 cycles, the leakage had increased to 495 scc helium/hour and further dry cycling failed to decrease that value. For this reason, the test was terminated and the valve disassembled for examination of the K-96 seat and poppet. The same characteristic uneven wear at the sealing interface of the poppet that was reported in test No. 3 with this bipropellant valve was observed. The erratic leakage measurements (decreasing with dry cycling in helium) throughout this test further substantiated the misalignment hypothesis proposed in test No. 3. The decision was made to have the poppet assembly of this particular valve precisely re-aligned for more normal seat contact before using it for further K-96 evaluation (test No. 9).

Test No. 6/Zirconium Diboride/Component Valve/GF_2 Passivation Only

Test No. 6 was to evaluate a seat and poppet set fabricated from zirconium diboride for the component valve in the ClF_5 environment. After installation in the valve, pre-test helium leakage measured nearly 120 scc/hour. Dry cycling in helium did not decrease the leak rate and a porosity problem was suspected. The decision was made to continue the test, disregarding the excess initial leakage, to observe the effect of the GF_2/ClF_5 environment. After passivation in the system with gaseous fluorine (GF_2), helium leakage increased to about 15,000 scc/hour. When dry cycling in helium at that time failed to decrease the leakage, the test was halted. The zirconium diboride parts were returned to the vendor, and when they were found not to meet theoretical density specifications, the initial suspicion of a porosity problem was confirmed. A second set of zirconium diboride seat and poppet hardware was fabricated and delivered for evaluation at a later date (Test No. 15).

Test No. 7/K-96 Tungsten Carbide/Component Valve/ClF_5

Newly relapped K-96 hardware was installed in the component valve for test No. 7. The objective of this test was to demonstrate 100,000 actuations in ClF_5, assuming periodic leakage measurements were acceptable. After GF_2 passivation, helium leakage measured 331 scc/hour. Dry cycling failed to decrease the leakage and the test was halted.

The valve was removed from the test system and disassembled. The seat and poppet exhibited a slight film on the sealing surfaces and moisture contamination was suspected. Failure to maintain a constant gaseous nitrogen (GN_2) purge through the valve after installation in the test system (prior to starting passivation) is a possible source of the moisture contamination.

This same seat and poppet was cleaned and re-installed in the valve for test No. 8.

Test No. 8/K-96 Tungsten Carbide/Component Valve/ClF_5

To minimize future potential moisture contamination (evident in test no. 7), the entire valve was baked in an oven at $160^{\circ}F$ for 4 hours prior to repeat evaluation of the K-96 seat and poppet hardware. Pre-run helium leakage measured 16 scc/hour. After 100,000 cycles in ClF_5, leakage measured between 36 and 54 scc/hour on two separate occasions.

This particular K-96 tungsten carbide seat and poppet hardware was the first to achieve the complete cycle design life with helium leakage remaining near the 30 scc/hour goal.

Test No. 9/K-96 Tungsten Carbide/Bipropellant Valve/ClF_5

The bipropellant valve last used in test No. 5 was used to evaluate newly relapped K-96 seat and poppet hardware in test No. 9. This valve was precisely re-aligned because of the uneven wear noted on the poppets in tests 3 and 5. Additionally, the pre-run bakeout procedure initiated in test No. 8 was followed.

Upon GF_2 passivation, helium leakage increased from 10 to 41 scc/hour. Because past experience had shown that cycling the K-96 material sometimes caused the leakage rate to decrease (test No. 5), cycle testing in ClF_5 was started. After 100, 000 complete cycles in ClF_5, helium leakage measured 10 scc/hour. The K-96 tungsten carbide material had now proved successful in both the component and bipropellant valves.

Test No. 10/K-96 Tungsten Carbide/Bipropellant Valve/ClF_5

The objective of test No. 10 was to duplicate the successful results of the previous test No. 9. A newly re-lapped K-96 seat and poppet set was installed in the bipropellant valve that had been re-aligned for test No. 9. The bakeout procedure was also included and pre-test helium leakage measured 5 scc/hour. After passivation with GF_2 the leakage rate measured in situ (Appendix B) remained at 20 scc/hour. Periodically throughout the test (after 21, 000/43, 000/102, 000 cycles in ClF_5), leakage measurements were taken to evaluate the material's performance. Zero pressure rise was detected in each of these 10-minute leak checks and the valve was removed from the test system after apparently completing the cycle requirement with acceptable helium leak rate. Following past practice, the leakage rate was double checked by the water-displacement technique. The helium leakage measured by this method was 240 scc/hour.

This wide inconsistency of these particular leakage measurements in this instance cannot be explained. In all previous tests (as well as all subsequent tests), the helium leakages measured by the water-displacement manometer technique after removal from the test system were found to correspond precisely with the in situ pressure rise measurements.

In an effort to check the validity of the pressure-rise measurement technique, the test system was inspected for faulty pressure transducers, instrumentation, control volume isolation valves, etc. No cause for errors from the system standpoint could be found. A reasonable explanation for the anomaly in the data is the possibility of post-test moisture contamination of the valve during the time period between removal from the system and prior to the water-displacement leak check. The tremendous affinity of metallic fluorides for water (Reference 11) and the resultant oxides or oxyfluorides that would form on, and thus degrade, the fine finish on the sealing surfaces could explain the phenomenon. The relative humidity on this particular test day was 78 percent, considerably higher than normal for the area.

Test No. 11/K-801 Tungsten Carbide/Component Valve/ClF_5

The objective of test No. 11 was to evaluate the Kennametal K-801 material in ClF_5 service. This material is identical to the Kennametal K-96 in all respects except for the binder. No dramatic differences in performance of the nickel binder (K-801) and cobalt binder (K-96) were expected.

The poppet assembly was again precisely re-aligned for normal seat contact for this test and the valve was baked at 160°F for 4 hours. Pre-run helium leakage measured 10 scc/hour, and after 100,000 cycles in ClF_5 the leakage increased to only 28 scc helium/hour. No appreciable difference in seat and poppet wear was noted between the K-96 and K-801 materials, and both varieties of tungsten carbide had now met the helium leakage goal after 100,000 cycles in ClF_5.

Test No. 12/K-801 Tungsten Carbide/Component Valve/GF_2 Passivation Only

In an attempt to duplicate the initial highly successful results of the K-801 material (test No. 11), a second set of seat and poppet hardware was installed for evaluation in test No. 12. Prior to cycling in ClF_5, a small fluorine fire in the test section of the system ruined the second set of K-801 seat and poppet specimens and halted the test at the passivation point. The fire appeared to originate in a contaminated inlet filter and quickly passed through the test item itself and out the stainless steel run line downstream of the valve. The fire completely destroyed the component valve's armature assembly and fused the seat and poppet specimens together. A photo of the test item and test section is shown in Figure 8.

Test No. 13/Duranickel 301/Component Valve/GF_2

The fire which occurred during the previous test fortunately left the valve's body and coil housing in workable condition. although the armature/poppet assembly and seat and poppet set were destroyed. The valve was returned to the manufacturer for refurbishment with spare component parts. A Duranickel 301 seat and poppet set was installed for this test to demonstrate the failure mode in GF_2 cycling instead of in ClF_5.

After 11,000 cycles in intermittent gaseous fluorine flow at 10 to 250 psi ΔP, helium leakage increased from 10 to 120 scc/hour and the test was halted. As expected, the appearance of the failed seat and poppet sealing surfaces was similar to that of past Ni 301 hardware after ClF_5 cycling. Figures 9 and 10 are photographs of the subject seat and poppet after test No. 13. Based upon the leakage data of previous Ni 301 hardware which had been cycled in ClF_5 (test No. 4), it appears that the GF_2 environment may be less severe. In that test, leakage measured 450 scc helium per hour after 6000 actuations.

Test No. 14/K-96 Tungsten Carbide/ClF_5

This test was the final evaluation of the Kennametal K-96 tungsten carbide (cobalt matrix) material. Previous testing had demonstrated its ability to meet the 100,000-cycle requirement with low leakage rates (tests 8, 9 and 10). The objective of this test was to accumulate up to 500,000 cycles in ClF_5 over an extended time period. During non-test days, the valve would remain in the test system being intermittently exposed to ClF_5 at its vapor pressure.

This test encompassed a 23-day period during which 10 days of ClF_5 exposure and 470,000 cycles were accumulated. Figure 11 depicts the leakage characteristics of the valve throughout the test.

Upon initial assembly, the valve leaked 30 scc helium per hour (Point A), and the leakage increased to 41 scc/hour (Point B) upon GF_2 passivation. During the first day's testing in ClF_5, approximately 100,000 cycles were accumulated, resulting in a leak rate of 40 scc/hour (Point C). The valve was then left pressurized with helium for a 10-day period. For some reason, the leak rate measured at the end of this 10-day period increased to over 150 scc/hour (Point D). Cycling in ClF_5 was begun and an additional 63,000 actuations were accumulated. Leakage returned to a level of about 40 scc/hour after this day's test (Point E).

The valve was then exposed to ambient temperature ClF_5 (Reference 1) at the normal vapor pressure for a period of 4 days. Again, without further cycling of the valve in ClF_5, the leakage increased, this time to about 100 scc/hour (Point E). The lines were drained and the valve purged, dried and pressurized with helium. After a 3-day exposure to the helium, leakage measured slightly over 200 scc/hour (Point G), and cycling in ClF_5 was begun again. As had happened before, after an additional

153,000 cycles in ClF_5, the internal leakage returned to an acceptable 51 scc helium/hour (Point H). A total of 316,000 cycles in ClF_5 had now been accumulated on this hardware during this test.

After a 3-day exposure to static ClF_5, cycling was resumed to a cumulative total of 471,000 actuations in propellant. The final leakage rate on the valve was an acceptable 46 scc/hour (Point I).

The seat and poppet are shown in Figures 12 and 13, respectively. Figure 14 is a proficorder trace of the poppet sealing surface which clearly shows the 300μ inch groove that was worn into it.

The erratic increases in leakage with static helium and ClF_5 exposure after valve cycling is unexplainable. Film formation, contaminant buildup and external contamination can all be speculated upon. This test clearly demonstrates the K-96 material's ability to withstand 100,000 actuations in the propellant environment without development of excessive internal leakage. Explaining the apparent increase in leakage when cycling is stopped and the valve is statically exposed to helium and ClF_5 for brief periods is beyond the scope of investigation. (See the Conclusions Section of this report for a discussion of this phenomenon.)

Test No. 15/Zirconium Diboride Component Valve/ClF_5

Previous test results of zirconium diboride at the AFRPL (test No. 6) had been inconclusive. In that first evaluation, the valve failed the leakage requirement upon GF_2 passivation. The hardware was then found to be below the manufacturer's theoretical density specifications, resulting in porosity problems. Another set of seat and poppet hardware was machined from a new batch of material known to be of acceptable quality. Figures 15 and 16 show the final lapped seat and poppet which was assembled in the Marquardt component valve for this evaluation.

The large chip noticeable at about the 9 o'clock position on the poppet was outside the critical sealing area and pre-run leakage measured an acceptable 19 scc helium per hour.

Passivation of the test item for this particular test was attempted by allowing a small amount of liquid ClF_5 to slowly fill the test section. The decision to do so was made when the fluorine passivation system normally used was deemed unsafe because of an inoperative outlet valve. This represented the first such use of liquid ClF_5 passivation under this program, although it was used successfully on numerous occasions in a parallel AFRPL in-house program which was evaluating various injector and chamber designs for the ClF_5/MMH propellant combination. Consequently, the effect of this test procedure on seat and poppet performance was considered to be minor or nonexistent.

After approximately a 1-minute exposure to the passivating ClF_5 liquid at 100 psig with the valve held open, the test was halted when a severe heat buildup on the valve body was noted by the test engineer. The system was isolated from the ClF_5 supply, purged, and the valve was removed for disassembly and inspection. The insides of the valve were found in the condition described in test No. 12. The seat and poppet were fused together (as well as to the armature assembly itself), indicating the presence of intense heat. Once again, it is not possible to establish the precise cause of the fire. Contamination might be suspected as was theorized in the previous burn-through. The ClF_5 passivation (rather than GF_2) might be suspected, but this procedure was later shown to be safe in test No. 19. The last possibility that arises (probably the one that should have been considered first) is that this material simply is not compatible with fluorine or fluorinated oxidizers such as ClF_5, even in the nonimpact situation. In both tests of the zirconium diboride, the hardware failed to get past the passivation point.

Test No. 16/Boron Carbide/Component Valve

Refurbishment of the component valve was accomplished in-house, and the first of two boron carbide seat and poppet sets was installed. This boron carbide hardware was residual hardware from the recently completed Marquardt/Air Force contract F04611-70-C (Reference 3). An excellent pre-run leakage rate of 9 scc/hour was measured after assembly.

Random cycling of the valve during gaseous fluorine passivation revealed erratic flow and shutoff characteristics that caused the test to be halted prior to ClF_5 evaluation. Upon disassembly, the boron carbide poppet itsel was found to be fractured in several places within the retaining nut. Boron carbide parts molded without a binder are lightweight (specific gravity = 2.51) and have low tensile strengths of about 22,500 psi (Reference 12). By comparison, the tensile strength of Duranickel 301 is over 100,000 psi, while some tungsten carbides may go as high as 200,000 psi. The cause for poppet fracture in this case can reasonably be attributed to overtightening of the retaining nut which put undue tensile stresses on the poppet material. Cycling the valve then caused the poppet to fracture, probably along the lines of stress that were created upon poppet installation. A second boron carbide seat and poppet set was installed (with great care not to overstress the poppet) for evaluation in test No. 17.

Test No. 17/Boron Carbide/Component Valve/ClF_5

After the initial leakage measured 18 scc helium/hour, the valve was dry cycled 100 times prior to installation in the test system. The poppet did not break, and the valve was passivated with GF_2. Leakage increased to 49 scc/hour and the cycling in the ClF_5 propellant was begun.

No leakage could be detected by the pressure use method in a 10-minute check after 27,000 cycles. Cycling was continued that same day to the 50,000-cycle point where a leak of about 60 scc/hour was measured. Testing was halted on this test day after 101,000 actuations in the ClF_5 and a leak rate of 41 scc/hour. The following day, cycling was resumed (valve seat pressurized with helium overnight) to a total of 263,000 actuations in ClF_5. The final leak rate measured less than 40 scc helium/hour.

Based upon the results of this test, boron carbide may be considered to be an acceptable seat and poppet material for this application. Figures 17 and 18 are photos of the boron carbide hardware after test 17. Many tiny holes are readily visible on the sealing surfaces themselves, but apparently too few of the holes are interconnected sufficiently to create leakage paths which extend entirely across the seat. This test substantiated the selection of boron carbide as an acceptable material by Marquardt in Contract F04611-70-C (Reference 3).

Test No. 18/Binderless Tungsten Carbide/Component Valve/ClF_5

This material was found by Marquardt to be unacceptable for ClF_5 seat and poppet service (Reference 3). Except for the lack of a binder material, it is exactly the same as Kennametal K-96 and Kennametal K-801. The purpose of test No. 18 was to substantiate these Marquardt test findings.

Initial pre-run leakage measured only 5.6 scc helium/hour, and passivation in GF_2 failed to have any noticeable effect. However, after approximately 20,000 actuations in ClF_5, leakage increased to over 3000 scc/hour and the test was halted. The sealing surfaces appeared grainy and coarse, with literally thousands of tiny holes in addition to several larger holes (Figures 19, 20 and 21). It is now apparent that the binder material somehow plays a key role in the success of the tungsten carbide-based hardware.

Test No. 19/K-801 Tungsten Carbide Component Valve/ClF_5

The objective of this test was to duplicate the good results of test No. 11. The K-801 material was last evaluated, however, in test No. 12, when a fire of unknown origin destroyed the seat and poppet specimens and most of the internal workings of the valve. This was to be the concluding test of this program.

Initial pre-run leakage after assembly was an acceptable 25 scc/hour, and the valve was installed in the flow system for passivation. Passivation in this instance was with liquid ClF_5, not GF_2. No deleterious effect of the ClF_5 passivation was observed. (Compare with test No. 15 - zirconium diboride.) The valve was not leak checked after passivation, and cycling was begun immediately.

After 22,000 cycles, a 20 scc helium/hour leak was measured in situ. This increased to about 40 scc/hour after 44,000 cycles. At the end of 103,000 actuations in ClF_5, no pressure rise could be detected in a 10-minute leak check in situ. The valve was removed from the test system and found to be leaking only 6 scc helium/hour. This extremely low leakage after 100,000+ cycles in ClF_5 represents the best results that were obtained under the program. The initial successful results of the K-801 tungsten carbide (28 scc/hour leakage after 100,000 cycles in test No. 11) were indeed duplicated, and the material is considered compatible for this application. Figures 22 and 23 show the seat and poppet as they looked after the test. Figure 24, with slightly different lighting, shows the poppet groove more clearly and how the nonsealing areas were relatively unaffected.

TABLE I. MARQUARDT VALVE CYCLE TESTS IN ClF_5/MMH
(Contract F04611-68-C-0074)

Test Series No.	Poppet and Seat Material	Primary Constituents (weight percent)	Cycle Test Fluid	Total No. of Cycles	Final Helium Leak Rate (scc/hour)
1	Pyromet X-15	62 iron, 20 Cobalt, 15 chromium	MMH	100,000	6
2	Pyromet X-15		MMH	47,800	110
3	Duranickel 301	93 ni, 4.5 aluminum, 2 beryllium	ClF_5	200	100
4	Duranickel 301		ClF_5	5,000	300
5	Duranickel 301		ClF_5	200	1,500
6	Pyromet X-15		ClF_5	1,000	900
7	Pyromet X-15		ClF_5	10,000	9,000
8	Duranickel 301		ClF_5	10,000	4,000
9	Duranickel 301		ClF_5	10,000	900
10	Duranickel 301		ClF_5	5,000	600
11	Pyromet X-15		ClF_5	5,000	800
12	Berylco Ni 440	97.5 nickel, 2 beryllium	ClF_5	1,000	1,000
13	Berylco Ni 440		ClF_5	1,000	800
14	Kennametal K-602	90 tungsten carbide, 10 tantalum carbide, 0.5 cobalt (essentially binderless)	ClF_5	9,000	90,000
15	Kennametal K-602		ClF_5	29,000	7,000
16	Kennametal K-602		ClF_5	1,000	900
17	Kennametal K-602		ClF_5	1,000	4,000
18	Kennametal K-96	94 tungsten carbide, 6 cobalt binder	ClF_5	1,000	50
19	Kennametal K-96		ClF_5	1,000	70

TABLE II. MARQUARDT BIPROPELLANT VALVE PERFORMANCE REQUIREMENTS

Pressure Drop, Maximum	40 psi at 0.167 lb/sec ClF_5 40 psi at 0.0757 lb/sec MMH
Dribble Volume, Maximum	Less than 0.02 $in.^3$ per valve
Operating Pressure (psia)	450
Proof Pressure (psia)	675
Burst Pressure (psia)	900
Opening Response, Maximum	8 ms at 28 VDC, 450 psia and 70°F
Closing Response, Maximum	8 ms at 28 VDC, 70°F and nominal Flowrate
Operating Voltage Range	18 to 32 VDC
Operating Temperature Range (°F)	-100 to 350
Operating Current, Maximum	2 amperes at 28 VDC and 70°F
Response Mismatch, Maximum (milliseconds)	0.5
Internal Leakage, Maximum	5 scc/hr helium at 0 to 435 psig initially 30 scc/hr helium at 0 to 435 psig after 100,000 cycles in propellant
External Leakage, Maximum	10^{-7} scc/sec helium at 675 psia
Operating Life, Minimum (cycles)	100,000
Acceleration (g's)	0 to 10 in any direction
Vibration	6 g's sinusoidal and 31.6 g's rms random
Inlet Filter Rating	18 microns absolute, 5 psi ΔP at nominal flowrates

TABLE III. AFRPL VALVE CYCLE TESTS IN ClF_5/GF_2

Test No.	Poppet and Seat Material	Total No. Cycles	Final Helium Leakage
1	Kennametal K-96	40,000	18
2	Kennametal K-96	15,000	103
3	Kennametal K-96	50,000	31
4	Duranickel 301	6000	450
5	Kennametal K-96	52,000	496
6	Zinconium Diboride	GF_2 passivation only	15,000
7	Kennametal K-96	GF_2 passivation only	331
8	Kennametal K-96	100,000	36 to 54
9	Kennametal K-96	100,000	10
10	Kennametal K-96	102,000	0, 240
11	Kennametal K-801	100,000	28
12	Kennametal K-801	Fire upon passivation	
13	Duranickel 301	11,000 (GF_2)	120
14	Kennametal K-96	470,000	52, 75
15	Zirconium Diboride	Fire upon passivation	
16	Boron Carbide	Broken poppet	
17	Boron Carbide	263,000	41, 60
18	Binderless Tungsten Carbide	20,000	3100
19	Kennametal K-801	103,000	6

TABLE III. AFRPL VALVE CYCLE TESTS IN ClF_5/GF_2

Test No.	Poppet and Seat Material	Total No. Cycles	Final Helium Leakage
1	Kennametal K-96	40,000	18
2	Kennametal K-96	15,000	103
3	Kennametal K-96	50,000	31
4	Duranickel 301	6000	450
5	Kennametal K-96	52,000	496
6	Zinconium Diboride	GF_2 passivation only	15,000
7	Kennametal K-96	GF_2 passivation only	331
8	Kennametal K-96	100,000	36 to 54
9	Kennametal K-96	100,000	10
10	Kennametal K-96	102,000	0, 240
11	Kennametal K-801	100,000	28
12	Kennametal K-801	Fire upon passivation	
13	Duranickel 301	11,000 (GF_2)	120
14	Kennametal K-96	470,000	52, 75
15	Zirconium Diboride	Fire upon passivation	
16	Boron Carbide	Broken poppet	
17	Boron Carbide	263,000	41, 60
18	Binderless Tungsten Carbide	20,000	3100
19	Kennametal K-801	103,000	6

SECTION VII

LIP-SEAL CONFIGURATIONS

Continuing the three-way approach taken to solve the ClF_5 ACS valve leakage problem, the second sealing concept investigated was the lip seal. This technique depends upon the scrubbing and flexing action of poppet/seat combinations and not the extremely smooth surfaces necessary in the flat-on-flat specimens. Figure 25 depicts schematically the configuration that was tested in ClF_5 under this program.

This design consists of a removable copper seat mated with an integral spherical sapphire poppet in a Moog bipropellant valve assembly (Model 53X120A). The stroke of this particular valve was measured to be approximately 0.020 to 0.021 inch total poppet travel. Ninety percent of this stroke was unobstructed motion with the remainder being movement after contact with the inner edge of the lip. In the closure mechanism, the lip expands outwardly (flexes) with the ever-increasing poppet diameter at the points of contact. A certain amount of "scrubbing" of the inner edge of the copper lip by the extremely hard sapphire poppet plays an important role in the sealing process.

For the initial test, the Moog bipropellant valve (Figure 4) was assembled with the copper lip seats installed in both the oxidizer and fuel sides. It should be noted here that during the time of lip-seal evaluations, the pre-run bake precaution had not yet been made procedural. Pre-run helium leakages were so low that none could be detected within a reasonable length of time, even by the water displacement technique.

With passivation in GF_2, helium leakage on the oxidizer side jumped to 200 scc/hour. Fifteen dry cycles in helium at this point failed to decrease the leakage, but the decision was made to continue the evaluation to note additional effects of cycling in ClF_5. After 5000 cycles, helium leakage measured approximately 7200 scc/hour. The test was halted at this point and the valve was removed from the test system. Figure 26 is a photograph that shows a portion of the lip of the seat that was cycled in ClF_5 during this test. A simplified schematic of the pre- and post-test lip is shown in Figure 27. The inner edge was actually permanently deformed, resulting in the buildup of a ridge of copper.

The results of the initial lip-seal test were duplicated in a second test evaluating an identical poppet/seat configuration. Again pre-run leakage was undetectable, this time even after CF_2 passivation. The valve was cycled 5000 times at a frequency of 8 Hz in ClF_5. The test was halted at this point when in situ helium leakage measured over 8000 scc/hour. Disassembly of the valve and examination of the oxidizer side showed the same wear pattern on the lip as was depicted previously in Figure 26. Additionally, a white semi-transparent film covered the entire seat, except in the sealing area where it appeared to be broken away (Figure 28).

The film was very brittle and chemical analysis showed it to be primarily chlorides and fluorides of copper. Because of its bulk, it is not likely that this material is the thin film formed upon passivation. A more logical explanation is that it represents reaction products formed upon exposure of the original passivation layer to atmospheric contaminants such as moisture (Reference 11). The absence of this film in the first test is unexplainable.

Because this particular lip-seal design failed in both tests to meet the leakage requirement after ClF_5 cycling, possible modifications were considered. Discussions with the configuration designer failed to reveal any sophisticated approaches leading up to the creation of the final seat and poppet hardware in the valve delivered to the AFRPL. The selected design resulted from leak testing several copper seats of varying lip thicknesses. Essentially, the lowest leak design was chosen.

The characteristic wear on the inner edge of the lip as evidenced by Figure 26 suggests that the poppet/lip angle (θ) may not have been quite correct to ensure maximum flexing with minimum surface abrasion. When that edge becomes degraded sufficiently, a spherical-on-conical seal configuration results, and this represents a different, less-effective sealing mechanism for this application. The extreme hardness of the sapphire poppet may also have played an adverse role in degrading that inner edge excessively. Theoretically, if the seal could be designed to flex outward before permanent deformation of the inner edge occurs, leakage characteristics should improve.

The initial lip seal that was designed and fabricated in-house under this program featured a stainless steel poppet mating to a copper seat. The hardware was sized for testing in the Marquardt component valve. The original intent was to evaluate several poppet/seat designs, each basically a lip-seal concept. The Marquardt valve was the only test valve that was available for such an investigation, because the Moog valve had the spherical sapphire poppet integral to its design and would have required extensive rework.

Figure 29 depicts schematically the new design selected. This design differed from the original Moog design of Figure 25 by two key parameters. The l/t ratio (lip "length" to lip "thickness") was increased by approximately 8 percent in hope of increasing the flexing tendencies of the sealing area. Similarly, to reduce the scrubbing of the inner lip edge, the initial impact angle (Figure 30) between the poppet and seat (θ) was reduced approximately 15 percent.

The seat and poppet set depicted in Figure 29 was inserted into the Marquardt single-armature component valve and the pre-run helium leakage was measured. Minimum leakage rates achieved, even after approximately 100 cycles in helium. were around 240 scc/hour. The valve was disassembled and the seat examined. There was no evidence of severe scratches, nicks or particles on the sealing edge and all dimensions of the hardware seemed to be within tolerances specified. Further visual inspection revealed that the thin-walled (cylindrical) lip appeared to be slightly out of round with respect to the center line. This lack of precise concentricity, the effect of which would be magnified by the slightest poppet misalignment, is the probable source of leakage. Because of the excessive pre-run leakage (that did not come down with cycling in helium), the design did not warrant cycle evaluation in chlorine pentafluoride. No further design or testing of the lip-seal concept was done and efforts were re-directed toward the metallic coatings investigation.

SECTION VIII

METALLIC COATINGS

GENERAL REQUIREMENTS

The application of protective coatings to metals has long been a common method of improving their corrosion resistance. Extensive coating research has resulted in suitable material identification and the development of application techniques for a wide variety of situations. The seat and poppet deterioration evident in the Marquardt valve after cycling in ClF_5 was one such situation in which the metallic coating technology could prove feasible. For this particular application, the material had the additional requirement of withstanding the dynamic impact conditions of the valve, as well as static exposure to the ClF_5 oxidizer in the non-flow situation.

The objective of this coating investigation was primarily the improvement of the corrosion-resistant properties of Duranickel 301, the original seat and poppet material selected for ClF_5 service. In-house testing at the AFRPL as well as extensive qualification testing by Marquardt (Reference 2) proved this material to be dynamically incompatible with ClF_5 in valve service. One test using Pyromet X-15 (the fuel side material) as the substrate was also conducted.

Coating thickness, uniformity, composition (purity), density, continuity and adherence all depend upon the particular process employed. The selection of hot dipping, vapor deposition, pack-cementation, chemical vapor deposition (thermal decomposition), cathode sputtering, cladding or electroplating as the process depends primarily upon: (1) the base metal, and (2) the protective material to be deposited.

The necessity for a very dense/continuous coating with a tenacious adherence to the base metal was of paramount importance for this application, because in most cases, the parts required final precision lapping to meet the roughness and flatness requirements imposed by the flat-on-flat sealing concept. These requirements included flatness within one helium light band (12 micro-inches) and surface roughness of the sealing surfaces (Figure 7) not to exceed 1 micro-inch arithmetic average (AA). An additional physical restriction was that coating thickness could not exceed 0.003 inch on the poppet without causing a significant stroke change within the valve. Specific materials under investigation as candidate coatings for Duranickel 301 were tungsten and aluminum. One test of chromium-coated hardware was also conducted.

CHEMICAL VAPOR DEPOSITION

It was proposed in Phase I of Air Force Contract F04611-70-C-0052, "Advanced ACS Valve Sealing Surface Compatibility Investigation," that seat and poppet materials could form solid, liquid or gaseous fluorides under the temperature and pressure conditions of the subject valve in ClF_5 service (Reference 11). Solid fluoride film formation results ultimately in fracture of that film and particle buildup that may result in a degradation of the fine surface finishes. This degradation in surface finish will cause excessive leakage. Theoretically, by choosing materials which form only liquid or gaseous metallic fluorides, this particle buildup and surface degradation becomes instead an extremely slow and uniform removal of seat and poppet material in sealing areas.

The fact that tungsten is one such material thought to form a gaseous fluoride, along with recent (at that time) successful results of K-96 tungsten carbide seat and poppet testing, led to the selection of tungsten as the initial candidate coating for the Duranickel 301 hardware.

Chemical vapor deposition (CVD) is a common process for coating small objects uniformly with tungsten. In the CVD process, tungsten hexafluoride gas is thermally decomposed with hydrogen at 500°C according to the following reaction:

$$WF_6 + 3H_2 \xrightarrow{500^\circ C} W + 6HF \quad \text{(Reference 13)}$$

In this manner, the tungsten (W) is vapor deposited onto the object being coated. The coated surface thus formed (Figures 31 and 32) is obviously rough and coarse, necessitating final lapping to attain low leakage prior to evaluation in the valve. Figures 33 and 34 show typical Duranickel 301 hardware that has been final lapped.

During the subsequent lapping operation, it soon became apparent that although the tungsten coat deposited by the CVD process was indeed dense and continuous, it lacked a strong bond to the base nickel metal. Attempts to lap the first coated seat resulted in a nearly total loss of the tungsten material in the central sealing area (Figure 35). The accompanying poppet (Figure 36) was lapped successfully with only slight edge loss.

A second tungsten-coated seat was delivered to the vendor (Microsurface Engineering) for final lapping. Measurements taken by Microsurface personnel indicated the part to be warped out-of-flat by approximately 0.0007 inch (58 helium light bands) across the diameter (Figure 37).

Before the lapping of the critical sealing area could proceed, it was first necessary to establish a flat reference plane. This was done by removal of the 0.0007 inch of material from the outside edge of the bottom of the seat. Figure 37 depicts the seat, schematically, in its precoated condition (dashed lines) and post-coated condition (cross-hatched area) with the warping greatly exaggerated for clarity. The material removed (0.0007) is represented by the shaded portion, though greatly out of proportion with respect to the rest of the seat.

Instruction given to lapping personnel was to remove only enough coated tungsten from the sealing land to ensure a smooth flat surface in that area. Because of the convexity of the top face of the seat, this critical sealing area was the first area affected in the lapping process. After lapping to a depth of 0.0005 inch, the part was removed from the apparatus and the sealing surface was examined. Despite the fact that the land width was still somewhat irregular, ranging from 0.010 to 0.016 inch across, fear of separating the remaining tungsten coat from the nickel-base material prevented further lapping. This second tungsten-coated seat in its post-lapped condition is shown in Figure 38. This lapped tungsten-coated seat and poppet set was installed in the Marquardt component valve, and pre-run leakage initially measured low enough to warrant its evaluation in ClF_5. To test the integrity of the CVD tungsten coating, the valve was dry-cycled in helium approximately ten times at 450 psig inlet pressure. A repeat leakage measurement was grossly out-of-specification and the valve was disassembled and inspected. The tungsten coating on the seat sealing area (Figure 39) was obviously fractured on cycle impact and severely damaged. The hardware was considered unfit for testing in ClF_5.

In an attempt to create a more effective tungsten/nickel diffusion layer at the interface to improve adhesion, a third nickel seat was coated by CVD and subjected to an additional vacuum diffusion bonding process at elevated temperatures. In this final instance, the tungsten coating literally could be "peeled away" from the nickel base. Further attempts to re-adhere the coating were also unsuccessful.

The obviously weak tungsten-nickel bond that was apparent in each of the three attempts can possibly be explained by the differences in thermal expansion between the two metals. Nickel has a thermal expansion of 7.39×10^{-6} inches per inch per degree F, roughly three times tungsten's 2.55×10^{-6}. Uneven shrinkage upon cooldown probably led to stress development at the interface, and hence failure on lapping. Multidirectional stresses at the tungsten/nickel interface on the seat land area (with its small edge and corner radii) could magnify the problem at that location. This would explain the more successful lapping results on the poppet face proper, with the only apparent loss of material being near the edges (Figure 36).

PACK CEMENTATION

Excellent bonds between metals (coating/substrate) are attainable utilizing the pack cementation process (Reference 14). In this process, the part to be coated is heated while "packed" in a powdered form of the coating desired. An alloy of the coating/substrate is formed at the interface, with this diffusion layer serving as a link to hold the two dissimilar metals together.

The second material selected for evaluation as a protective coating for Duranickel 301 was aluminum. This material is known for its success as a coating to prevent oxidation of iron at elevated temperatures (Reference 13). The protective mechanism of the aluminum coating in this instance is attributed to an inert oxide layer (Al_2O_3) that forms on its surface immediately upon exposure to the atmosphere. This solid aluminum oxide layer is extremely thin and clings tightly to the aluminum coating. Exposure of aluminum-coated seat and poppet hardware to a fluorinated environment (during GF_2 passivation) will result in displacement of the oxygen atoms of the Al_2O_3 layer with fluorine atoms. Thus, the outermost protective coating becomes the AlF_3 species (Reference 11) in this situation.

Within the operating conditions of the Marquardt valve (temperature and pressure) aluminum trifluoride exists as a solid. Whereas it is theorized (Reference 11) that "any" solid fluoride film formation of this type on the surfaces of the seat and poppet should be avoided, aluminum is possibly the best candidate of this class of materials. If a material forms a solid fluoride, it should:

a. Adhere tenaciously to the base metal
b. Exhibit structural strength
c. Have a density similar to that of the base metal

The lack of available data regarding properties a and b for Al/AlF_3 prevents their use as truly meaningful predictions as to the success of aluminum coatings. With fluoride film formation on the finely lapped seat and poppet surfaces, a volume change (usually an expansion) occurs (Reference 11). If this volume change can be minimized, i.e., satisfaction of property c above, less degradation of that surface finish will result. It is known that the densities of aluminum fluoride (AlF_3) and aluminum are 2.88 gm/cc and 2.70 gm/cc, respectively (Reference 15). From this standpoint, aluminum certainly appears more desirable than some other materials. For example, the densities of NiF_2 and nickel, respectively, are 4.8 gm/cc and 8.90 gm/cc.

Two sets of Duranickel 301 seats and poppets were coated with approximately 0.0015 inch of aluminum by pack cementation. Figures 40 and 41 show one of these sets. Visual comparison with the tungsten coated (via CVD) parts in Figures 31 and 32 illustrates dramatically the difference in surface roughness attainable between the two processes. Lapping of these aluminum-coated parts was accomplished with no difficulty, and they are shown in Figures 42 and 43 prior to installation in the component valve. Initial leakage measurements indicated an extremely acceptable 6 scc/hour helium by the water-displacement technique. The valve was baked and installed in the test system for cycling in ClF_5. After passivation with GF_2, leakage increased somewhat to approximately 40 scc helium/hour as measured by the pressure-rise technique, and cycling in ClF_5 was begun; the test was halted after 25,000 cycles in propellant when a rapid pressure rise was detected during the first in situ leakage measurement (coincident with the first propellant transfer operation to conserve test time). A leak rate of over 6000 scc helium/hour was measured by the water-displacement technique. The aluminum-coated Duranickel 301 seat/poppet hardware had obviously failed dramatically at some time during the first 25,000 actuations in ClF_5.

The valve was disassembled and photographs of the failed seat and poppet were taken. As Figures 44 and 45 explicitly show, the sealing surfaces in the area of impact were severely degraded. It appears that the coating was literally "eaten through" by the repeated impacts in the ClF_5 environment. A proficoder trace of the poppet is approximately 200μ inches deep (Figure 46).

CATHODE SPUTTERING

This was the third and final process investigated for applying protective metallic coatings to the original seat and poppet materials. In this process, a 500- to 2000-volt potential difference is applied between two electrodes in a partial vacuum to cause a glow discharge. This results in a disintegration or sacrifice of the cathode material which subsequently deposits on nearby objects within the chamber itself (Reference 13). The films thus produced by the cathode sputtering technique deposit atom-by-atom on the substrate material and adhere more tenaciously than the heavier, more bulk-deposited coatings produced by either chemical vapor deposition or pack cementation. The successful coating of a film thin enough to approach the peak-to-valley roughness heights (items 1 and 4 of Table IV) of the final-lapped substrate yet thick enough to meet density and continuity requirements, was the first prime objective of the investigation into the cathode sputtering technique.

As baseline reference points to put the roughness heights and coating thickness terminology on a similar basis, the following dimensions are presented:

TABLE IV. BASELINE REFERENCE DIMENSIONS

1. Surface finish of final-lapped substrate material	≤ 1	μ inch AA
2. Coating thickness of aluminum by pack cementation (1.5 mil)	~1500	μ inch
3. Coating thickness of tungsten by CVD (3.0 mil)	~3000	μ inch
4. Coating thickness of cathode sputtering (1000-6000 Angstroms)	~4-20	μ inch

5. Four sets of seats and poppets were coated by this process. They were:
   - (i) Pure Tungsten - 1000 Angstroms (Å) on Duranickel 301
   - (ii) Pure Tungsten - 6000 Angstroms (Å) on Duranickel 301
   - (iii) Chromium - 6000 Angstroms (Å) on Duranickel 301
   - (iv) Tungsten - 6000 Angstroms (Å) on Pyromet X-15

NOTE: What follows is a discussion of those tests.

1000 Å Tungsten on Duranickel 301/ClF_5

The sputtering chamber of the Micro-electronics Section of the China Lake Naval Weapons Center was used for all coating work by this technique. Approximately a 3-minute exposure within the chamber was sufficient to deposit 1000 Å of pure tungsten on the nickel-based substrate. The substrate, which had previously been lapped to ≤ one μ inch AA surface roughness is shown before and after coating in Figures 47 and 48. These photographs (taken with the aid of a scanning electron microscope) clearly show that the resultant surface finish after coating is of comparable quality or better than the lapped substrate. No further lapping of the coated parts was required to meet the pre-run leakage requirement.

Valve leakage after assembly with these parts was an acceptable 25 scc/hour. To test the integrity of the coating, the valve was dry cycled approximately 75 times in helium. Unlike the case of the vapor-deposited tungsten coating, no step jump in leakage due to surface damage could be detected. Further leakage measurements taken after fluorine passivation in the test system indicated zero pressure rise in a 10-minute check. Cycling in ClF_5 was begun.

The first leakage measurement was made during a normal propellant transfer operation at the 20,000-cycle point. The valve was found to be leaking at a rate of 1200 scc/hour. The test was halted and the valve was removed from the test system. The failed seat and poppet are shown in Figures 49 and 50. Clearly, there was a severe degradation of the sealing surfaces at the poppet/seat interface which caused the excess leakage. Close examination of those areas revealed that the exposed metal resembled Duranickel 301 after similar cycle tests in ClF_5. It was concluded that the repeated impact in ClF_5 (20,000 cycles) resulted in a steady removal of the thin tungsten coating ($\approx 4\ \mu$ inches) in those contact areas and eventual exposure of the incompatible substrate. The next test was to evaluate a heavier coating weight.

6000 Å/Tungsten on Duranickel 301

Increasing exposure time inside the sputtering chamber to 20 minutes provided approximately a 6000 Å ($20\ \mu$ inches) tungsten coat on this Duranickel 301 hardware. It was speculated that this added thickness would be sufficient to increase the seat/poppet life beyond that of the last test.

Initial pre-run leakage measured a very low 7.5 scc helium/hour. This verified a very fine and uniform tungsten deposition. The valve was baked out and in the system when a final leak check was made prior to fluorine passivation. A step jump of leakage to nearly 80 scc/hour was measured. The valve was cycled 90 times in 450 psig nitrogen and leakage increased to approximately 300 scc/hour. The test was halted at this point, prior to any controlled exposure to fluorine of ClF_5. A water-displacement leak test after removal from the test system showed a 360 scc/hour leakage.

Inspection of the failed seat and poppet revealed a dramatic degradation and discoloration of the sealing surfaces. Figure 51 is a photograph of the poppet face that shows what appear to be three distinct "burn spots." Also readily apparent is severe channeling where seat contact is made. This channeling, or creation of a groove on the poppet face, is a common occurrence after extensive cycling, but not usually so apparent prior to repeated impact or propellant exposure.

Figure 52 is a close-up of one of the three burn areas. It indicates fairly severe discoloration and degradation near the center of the poppet (within the groove) and extreme surface roughening at the impact area itself. Except in the three burn areas, the outermost area of the poppet face seemed relatively unaffected. Though the precise explanation for this phenomenon will probably never be known, the author suggests the following.

The valve was closed (normally-closed) when installed in the test system. Contamination of an unknown type and origin was introduced somehow downstream of the seat/poppet interface. This contamination reacted (burned) with small amounts of residual ClF_5 in the outlet line and a minute, yet extremely intense fire of short duration existed. This intense heat made its way upstream to the poppet face (central area) and radially outwa. across the contact surface before diminishing. This explanation accounts for the appearance of the poppet face depicted in Figure 53 with the key areas numbered for reference. Area No. 2 was directly exposed to the fire, while most of Area No. 4 was not, being "upstream" of the seat/poppet interface. A simplified schematic of the seat and poppet in the closed position is shown in Figure 54. Damage to the seat and poppet prevented cycle testing of the hardware in ClF_5.

6000 Å Chromium on Duranickel 301/ClF_5

Previous Marquardt investigations into electroplated chrome coatings were hampered by porosity problems. About 6000 Å of chromium were deposited onto the Duranickel 301 substrate for testing in ClF_5. An exceptionally fine quality deposition was reflected with the initial pre-run leak rate of 1.5 scc helium/hour. Cycle testing in ClF_5 was begun when zero pressure rise was detected in the 10-minute check after passivation with gaseous fluorine.

Leakage measurements were made after 25,000, 40,000, and finally 60,000 cycles. Leak rates were 175, 269, and 1,014 scc/hour, respectively. The test was halted at the 60,000-cycle point when leakage had increased dramatically. It is speculated that the chromium coat withstood the dynamic impact conditions in ClF_5 until sometime between 40,000 and 60,000 cycles. At this time, the Duranickel substrate was exposed and the sealing characteristics fell off accordingly. Figures 55 and 56 depict the failed chromium-coated seat and poppet hardware.

6000 Å Tungsten on Pyromet X-15/ClF_5

This final attempt to meet the 100,000-cycle requirement with coated seat and poppet hardware again involved sputter-deposited tungsten. In this case, the substrate material was chosen to be the original fuel-side, iron-based alloy, Pyromet X-15. The objective of the test was to note any significant increase in cycle life due to greater substrate hardness. After 23,000 cycles in ClF_5, helium leakage went from 20 scc/hour to over 3000 scc/hour, and the test was halted. Figures 57 and 58 show the failed seat and poppet.

SECTION IX

CONCLUSIONS

1. Tungsten carbide (with either a cobalt or nickel binder) is the material best suited for ClF_5 ACS valve application to date.

2. Boron carbide is also an excellent candidate material, though further investigation is required because of its brittle nature.

3. Residual moisture contamination in the microscopic pores of seat and poppet materials can cause considerable degradation of their effective ClF_5 cycle life.

4. Localized wear on the seat and poppet sealing interface is minimized with precise re-alignment.

5. Future development programs for liquid rocket components (especially valves) should outline performance testing approaches that more realistically simulate the environment and duty cycles of the hardware during the expected mission. In this program, the performance requirement was simply acceptable leakage after 100,000 actuations in ClF_5; however, cycling the valve periodically over extended time periods in ClF_5 to represent more realistically the mission requirement revealed additional problems for consideration.

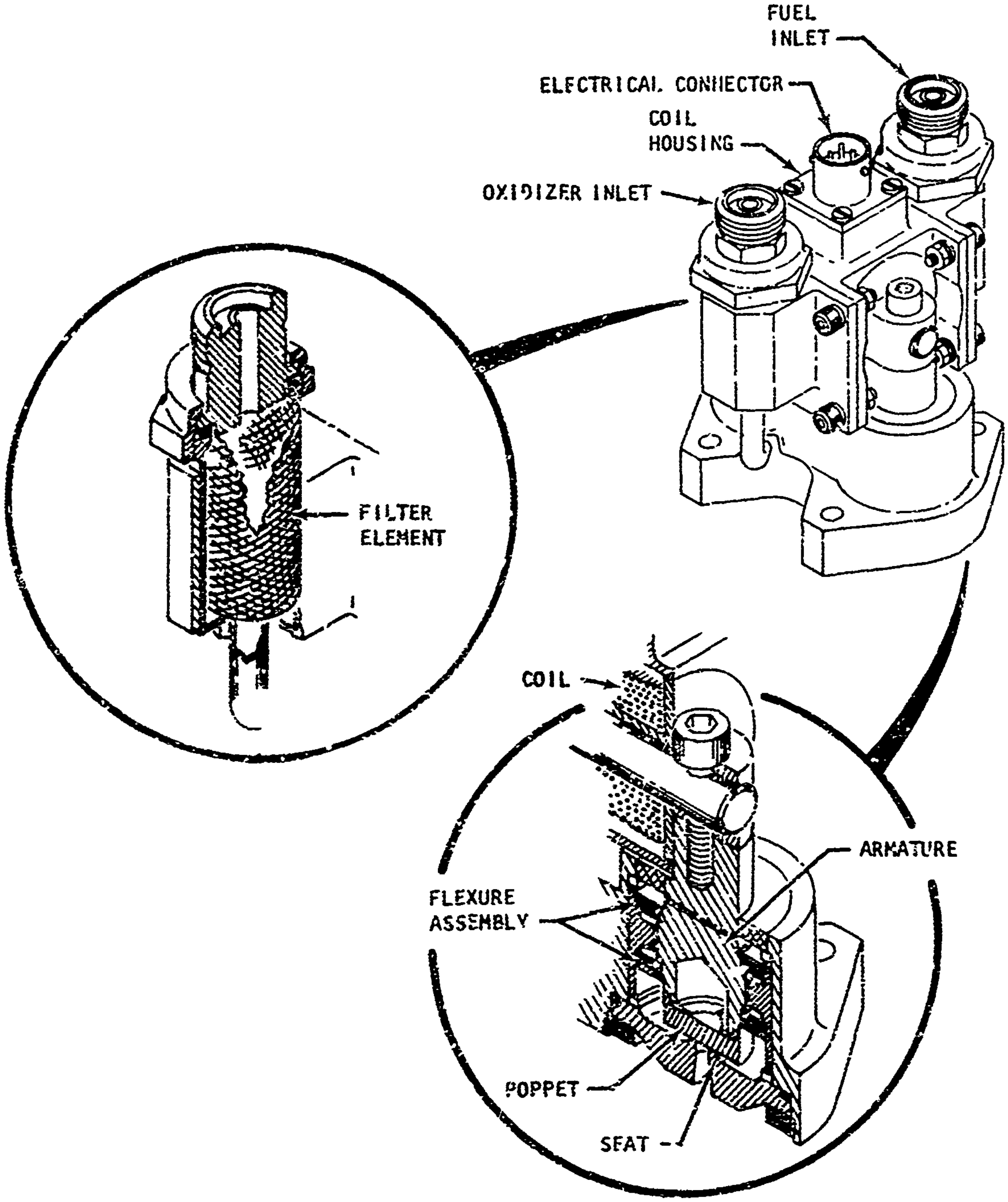


Figure 1. Sectioned Marquardt Bipropellant Valve

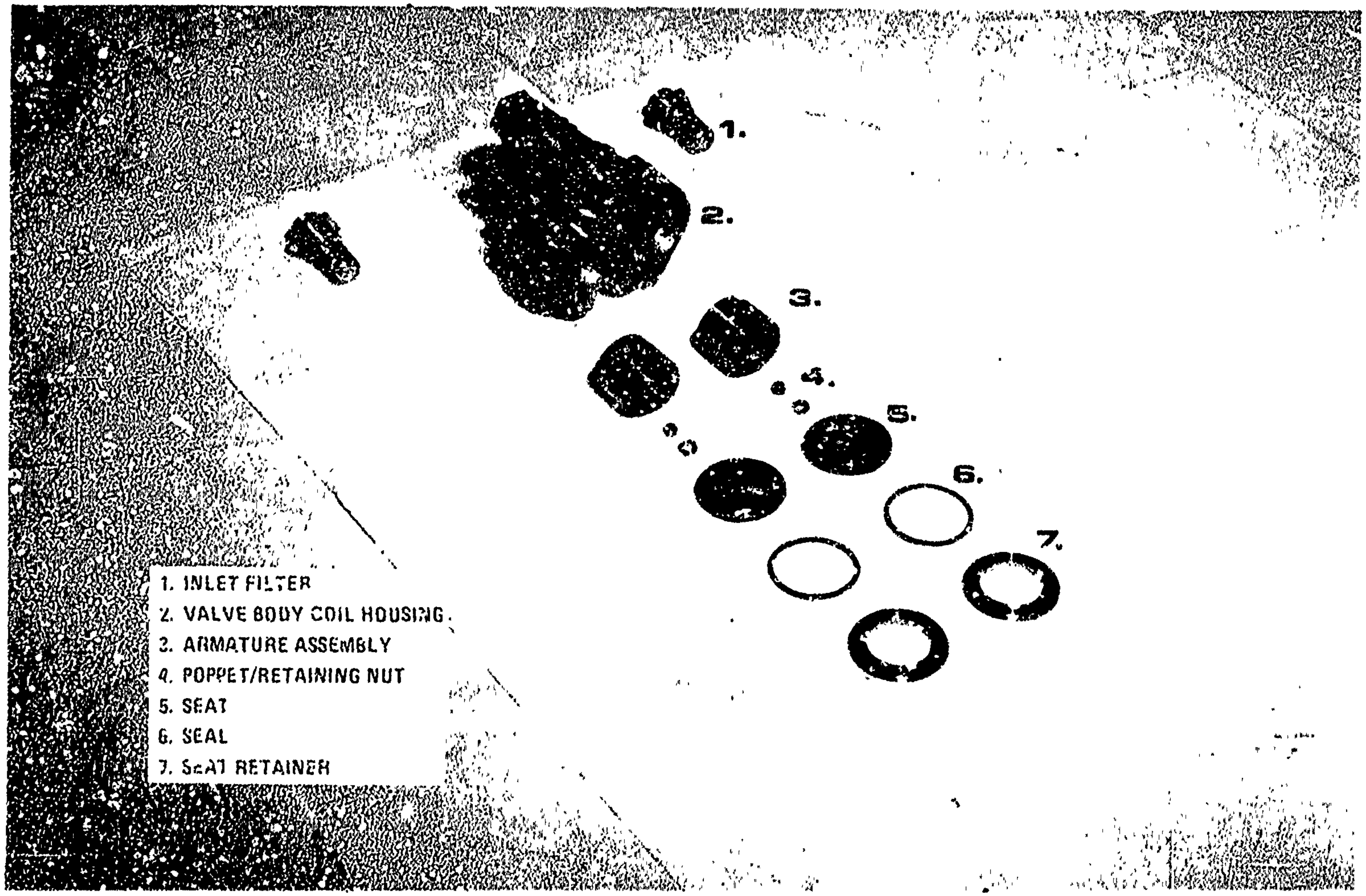


Figure 2. Marquardt Bipropellant Valve

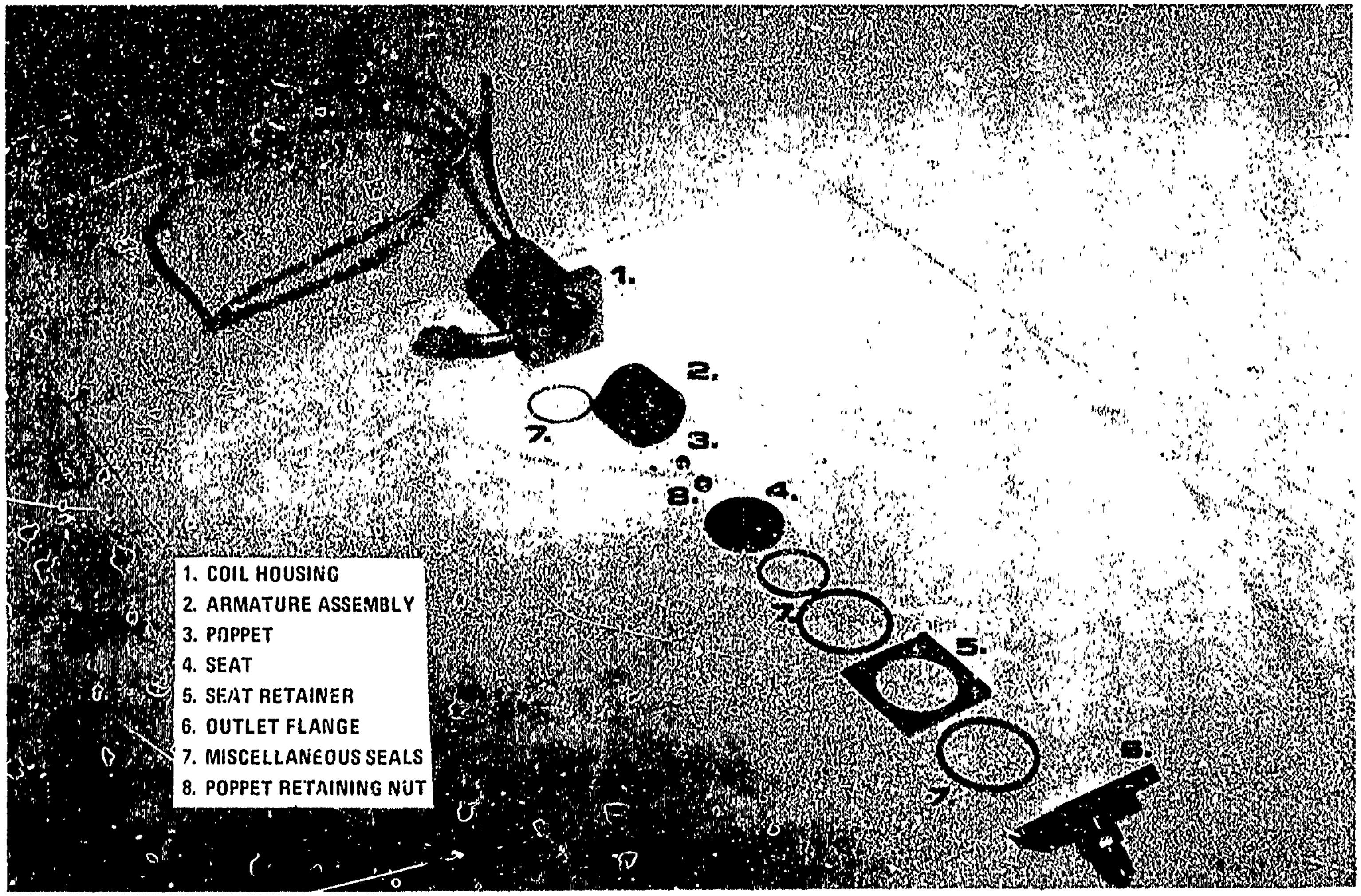


Figure 3. Marquardt Single-Armature Component Valve

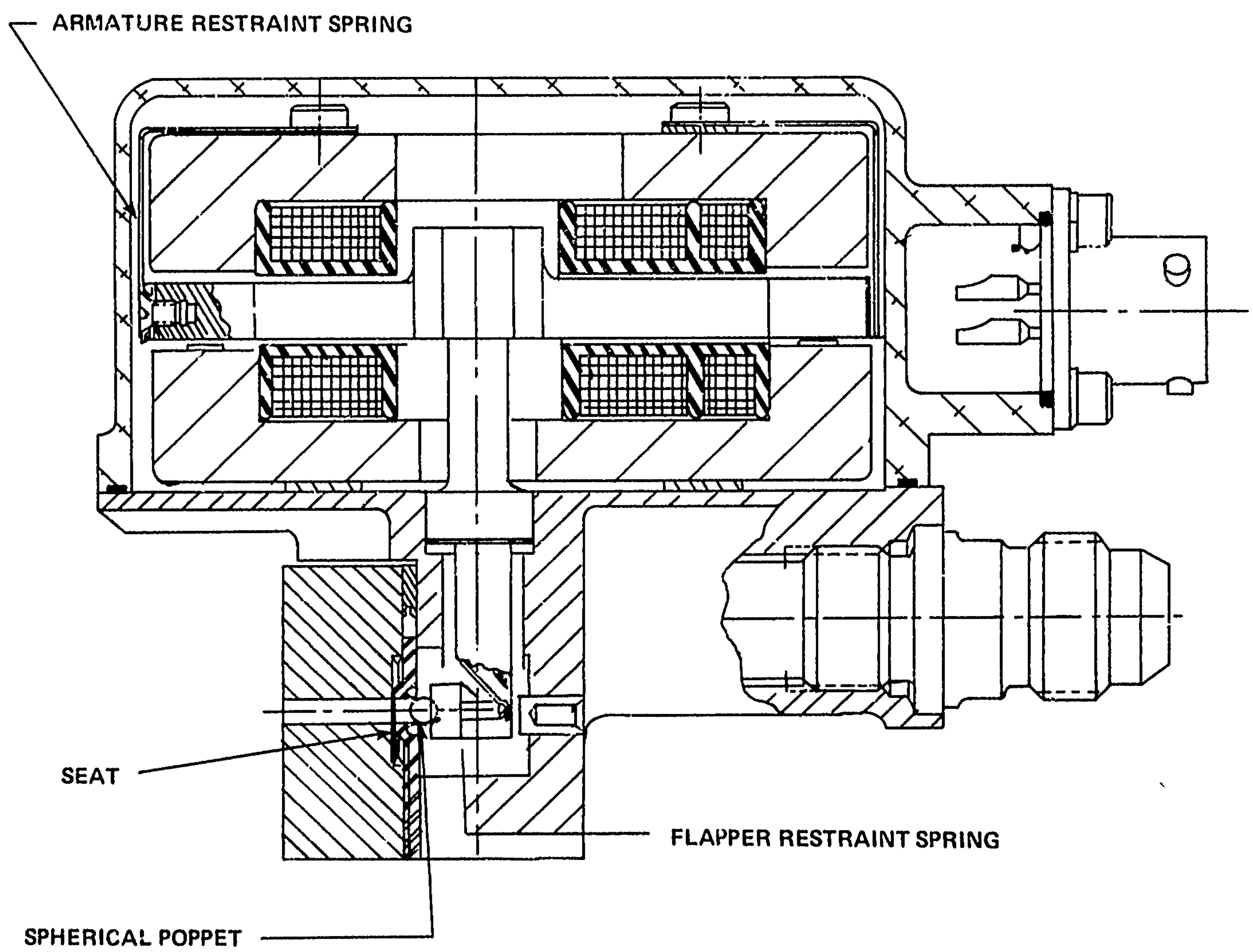
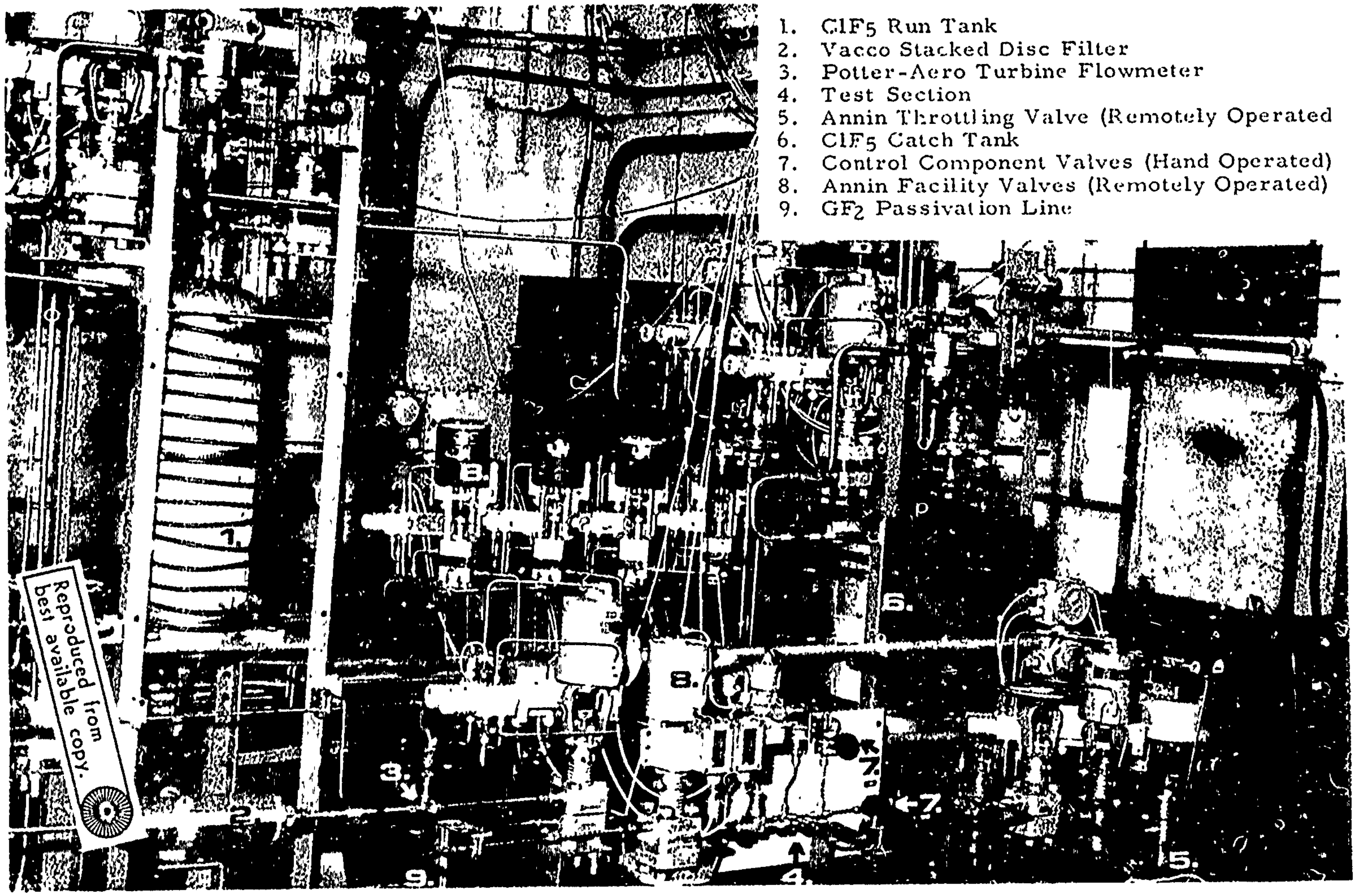


Figure 4. Sectioned Moog Bipropellant Valve



1. ClF5 Run Tank
2. Vacco Stacked Disc Filter
3. Potter-Aero Turbine Flowmeter
4. Test Section
5. Annin Throttling Valve (Remotely Operated
6. ClF5 Catch Tank
7. Control Component Valves (Hand Operated)
8. Annin Facility Valves (Remotely Operated)
9. GF2 Passivation Line

Figure 5. AFRPL ClF_5 Flow Loop

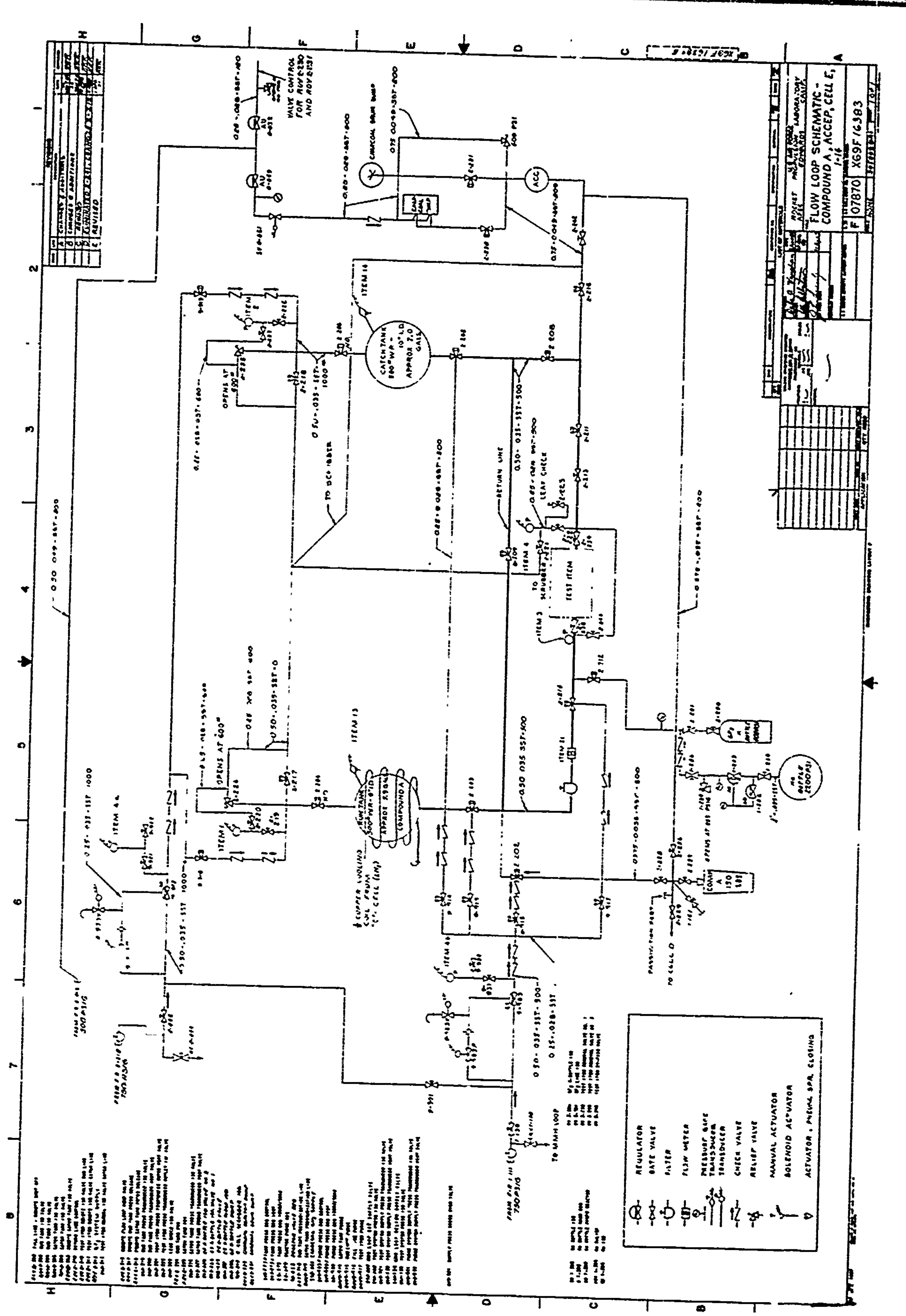


Figure 6. Schematic, AFRPL ClF_5 Flow Loop

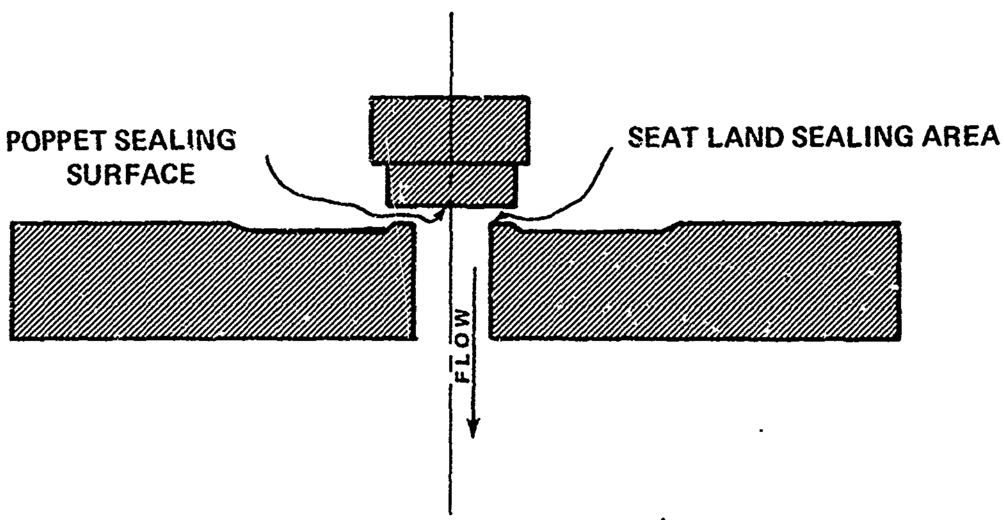


Figure 7. Schematic, Marquardt Flat-on-Flat Seat and Poppet (Component Valve)

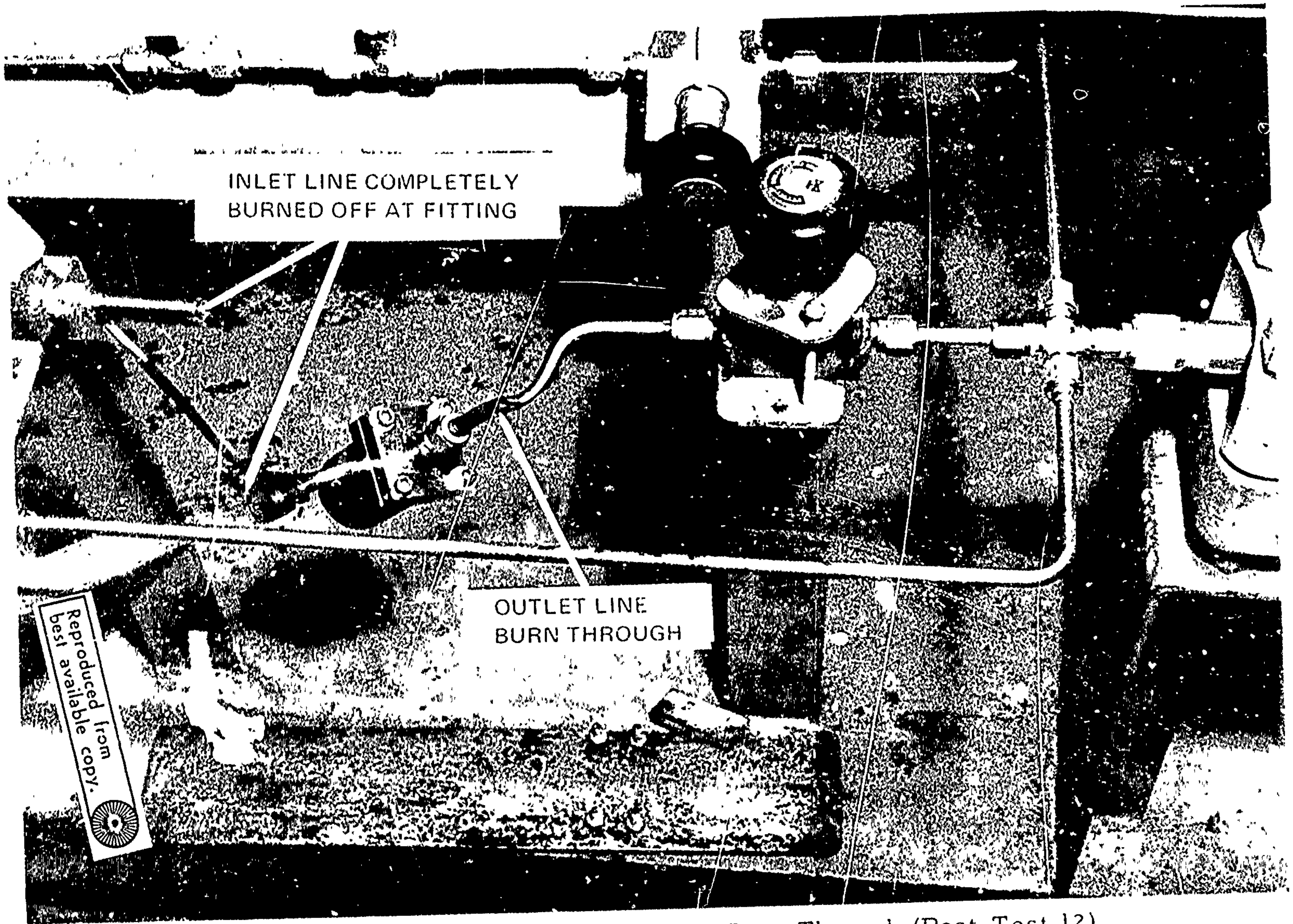


Figure 8. Marquardt Component Valve Burn Through (Post-Test 12)

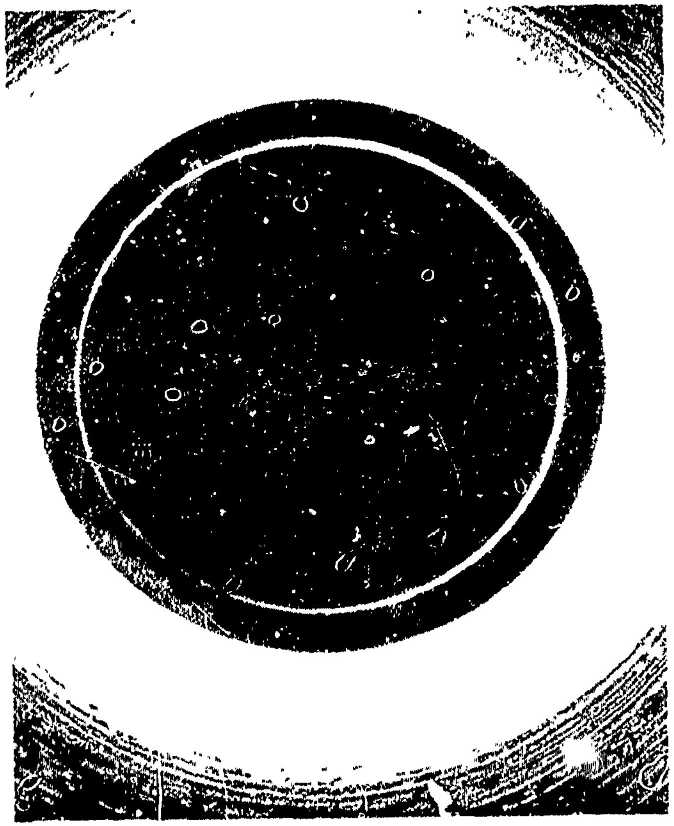


Figure 9. Seat, Ni 301 Post Test 13 (25X)

Figure 10. Poppet, Ni 301 Post Test 13 (20X)

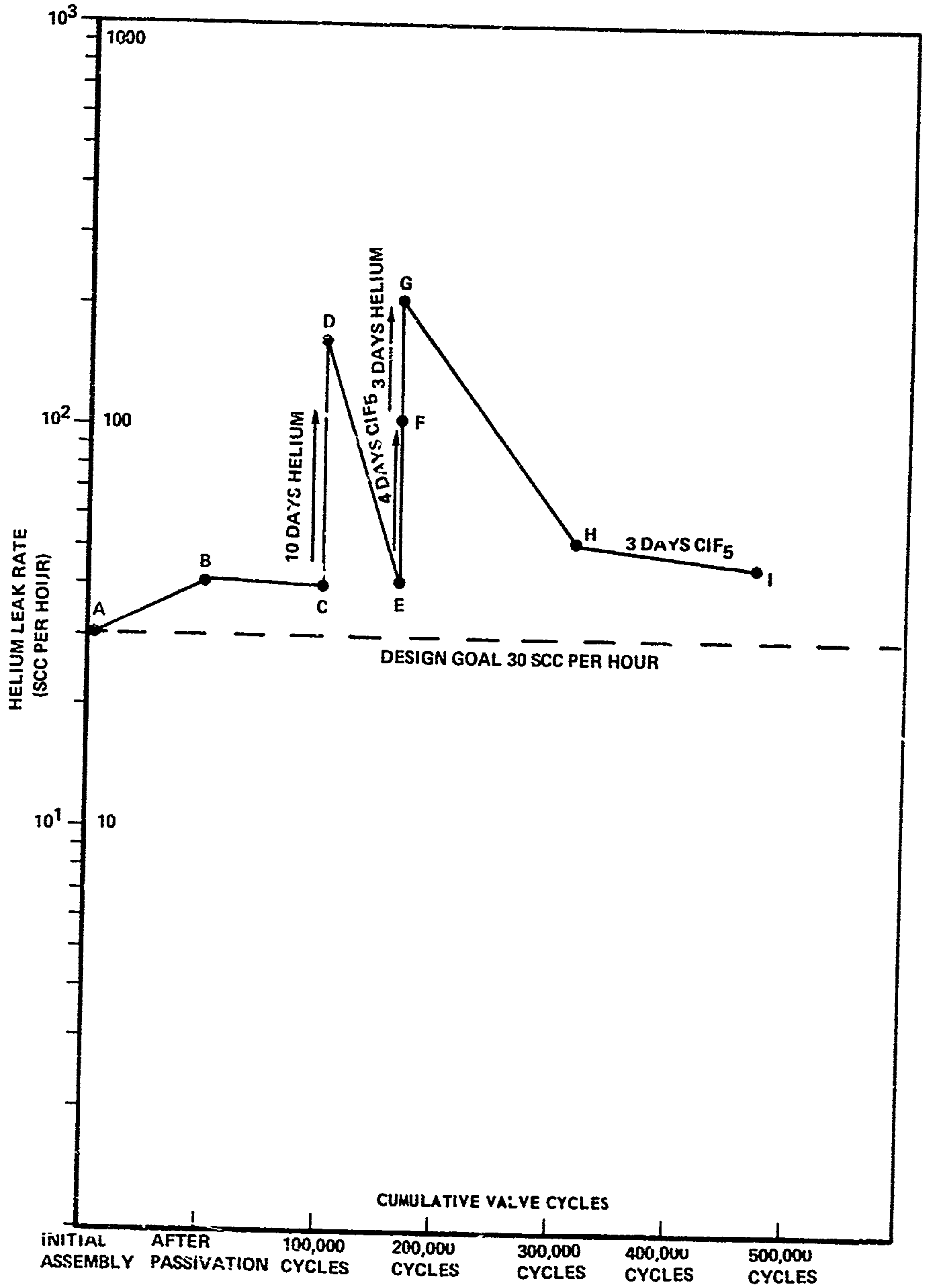


Figure 11. Leakage versus Valve Cycles, Test No. 14.

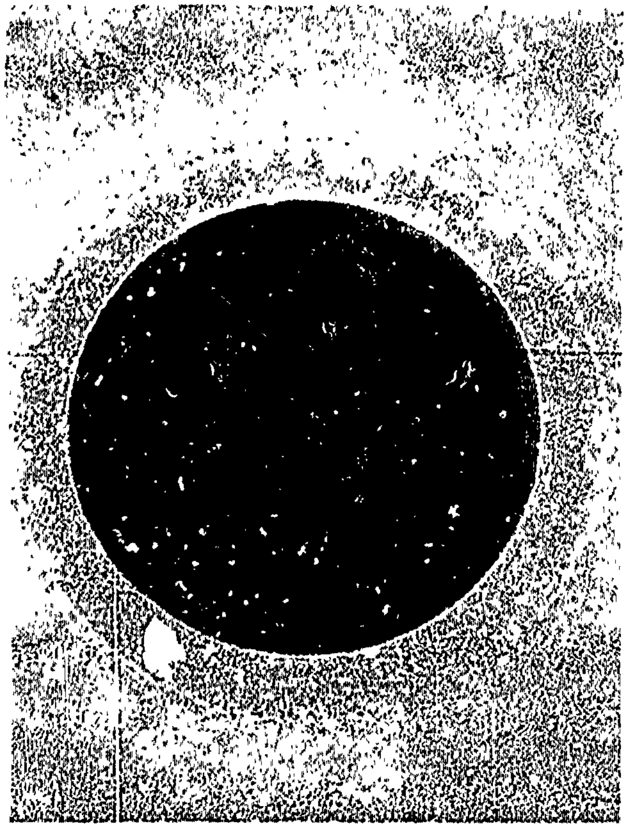


Figure 12. Seat, K-96 Post-Test 14. (25X)

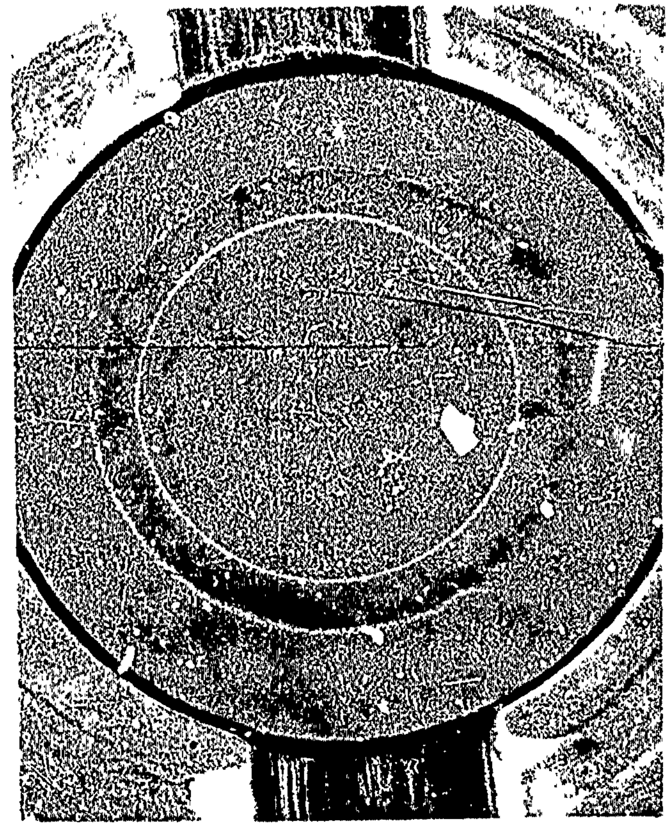


Figure 13. Poppet, K-96 Post-Test 14 (20X)

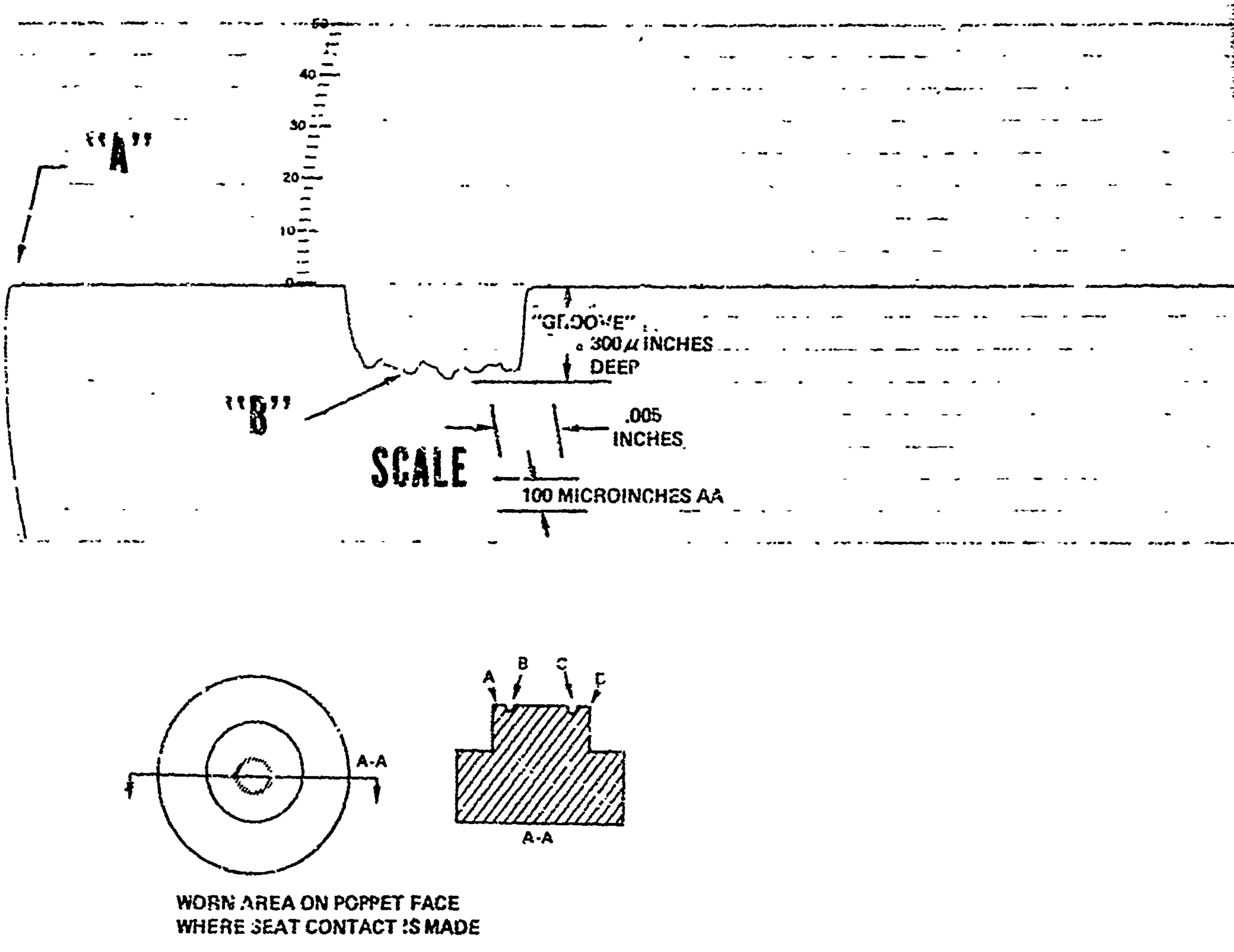
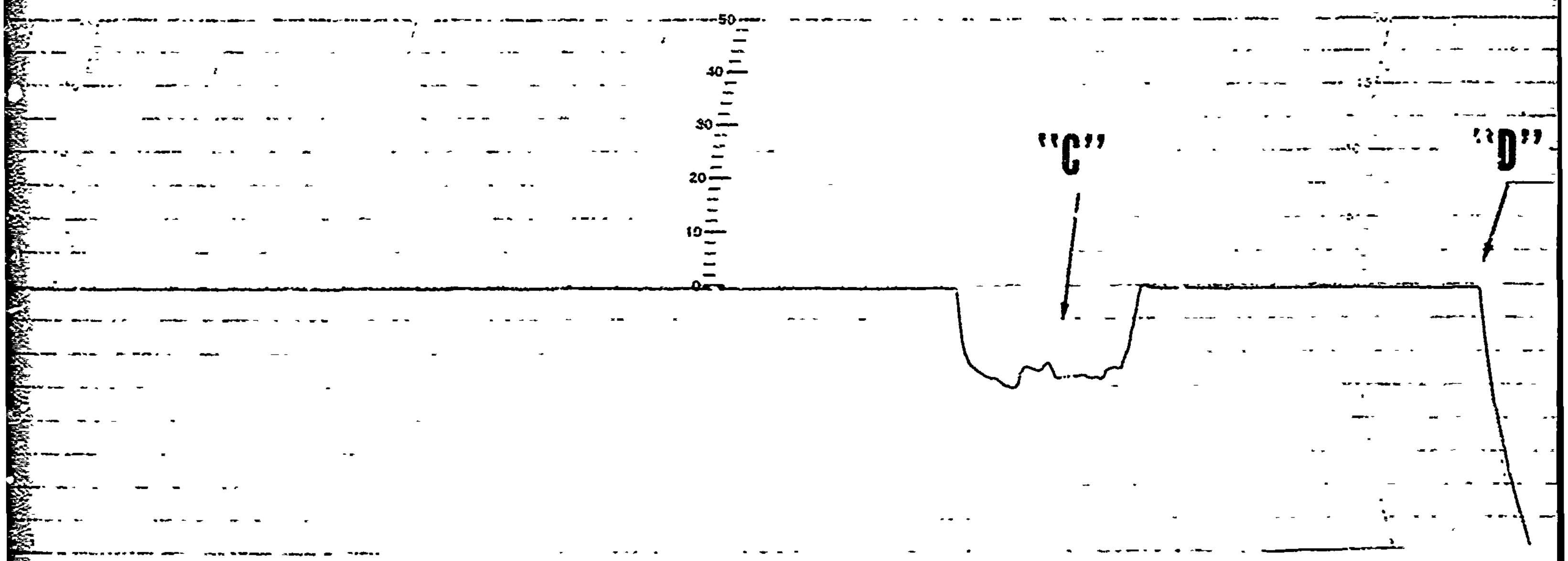


Figure 14. Proficorder Trace, K-96 Poppet, Post-Test 14.


50
40
30
20
10
0
"C"
"D"

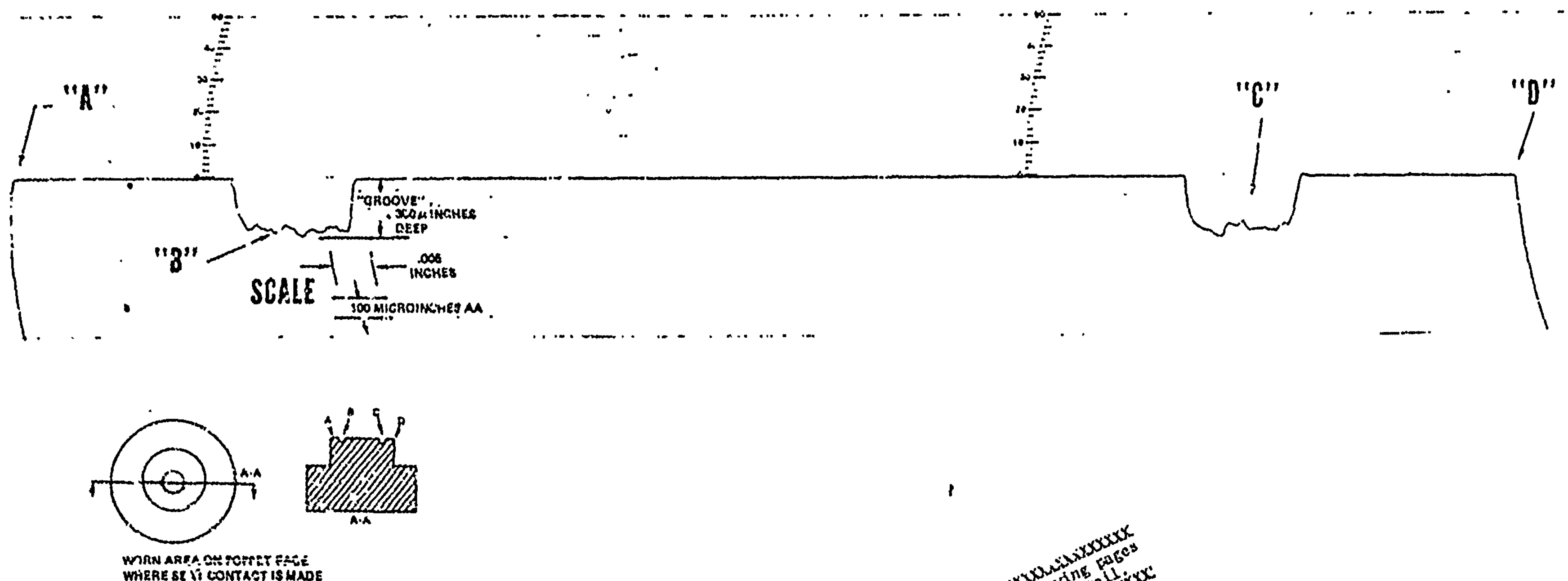


Figure 14. Proficorder Trace, K-36 Poppet, Post-Test 14.

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See the following pages
for greater detail.
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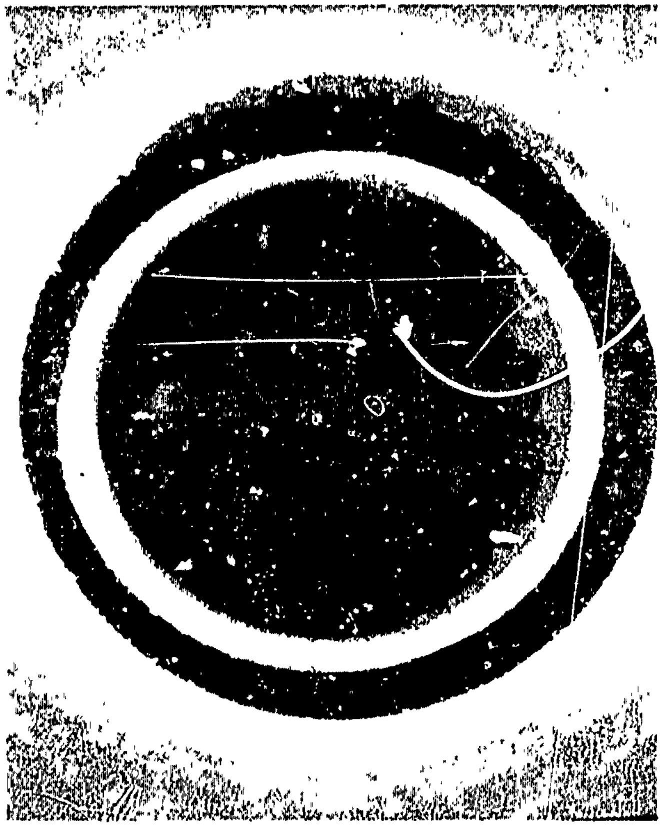

Figure 15. Seat, ZrB_2, Pre-Test 15 (25X)

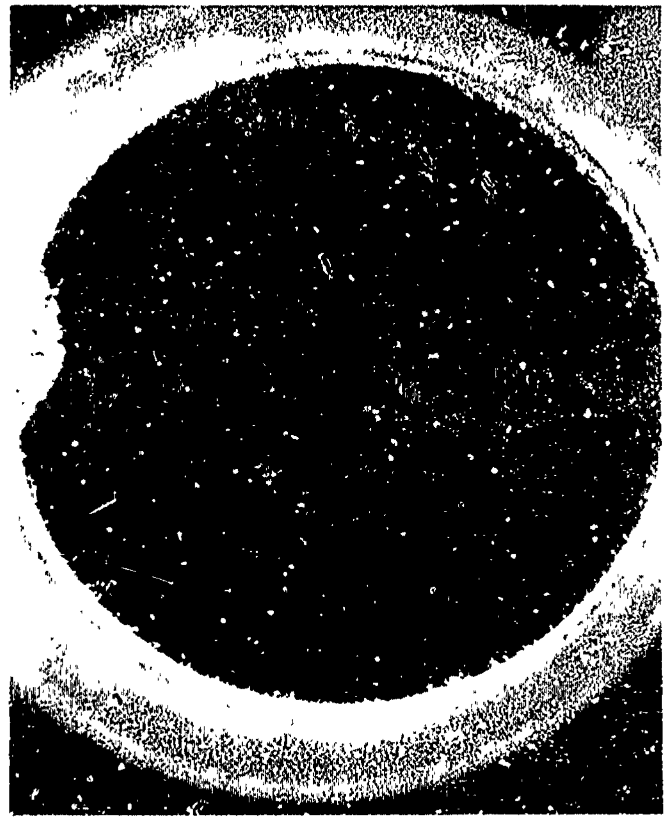

Figure 16. Poppet, ZrB_2, Pre-Test 15 (20X)

Figure 17. Seat, B_4C, Post-Test 17 (25X)

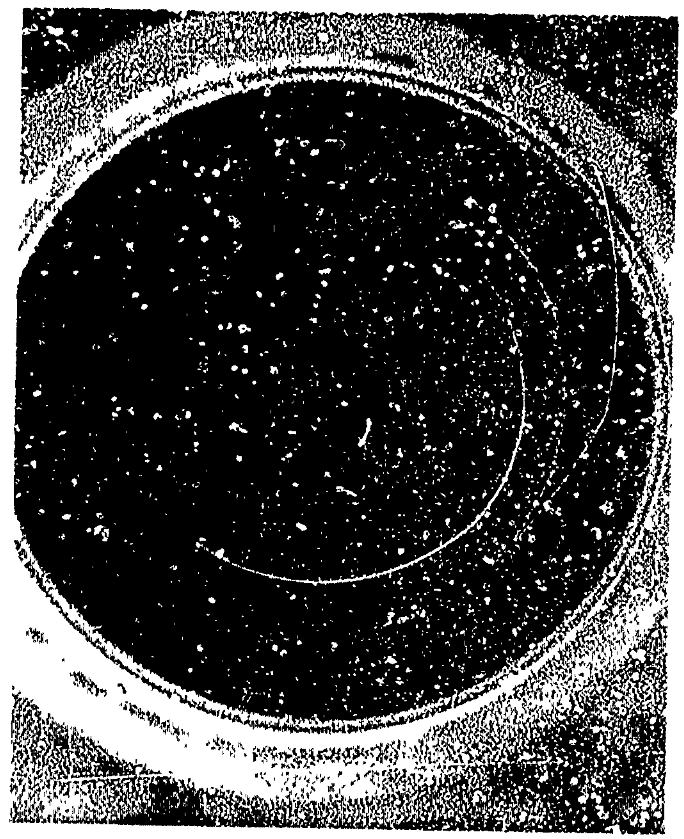

Figure 18. Poppet, B_4C, Post-Test 17 (20X)

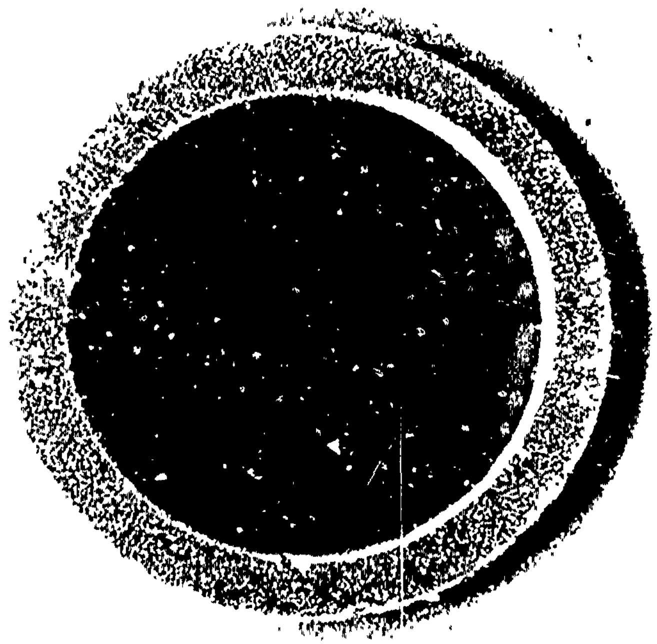

Figure 19. Seat, Binderless Tungsten Carbide, Post-Test 18

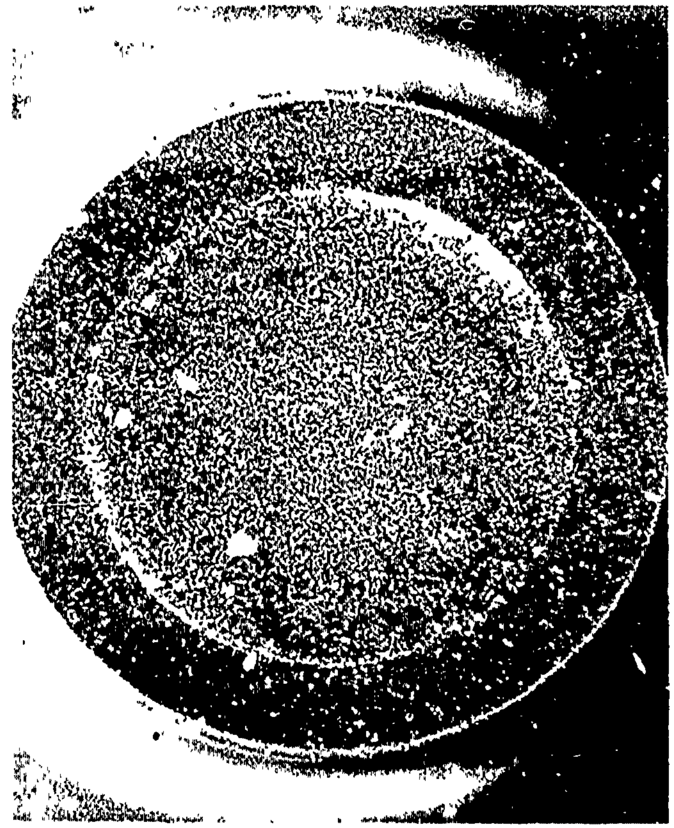

Figure 20. Poppet, Binderless Tungsten Carbide, Post-Test 18

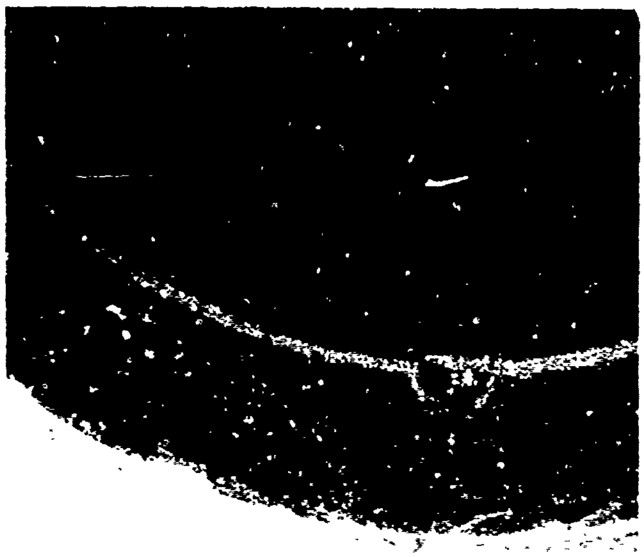

Figure 21. Seat, Binderless Tungsten Carbide, Post-Test 18 (Close-up) (80X)

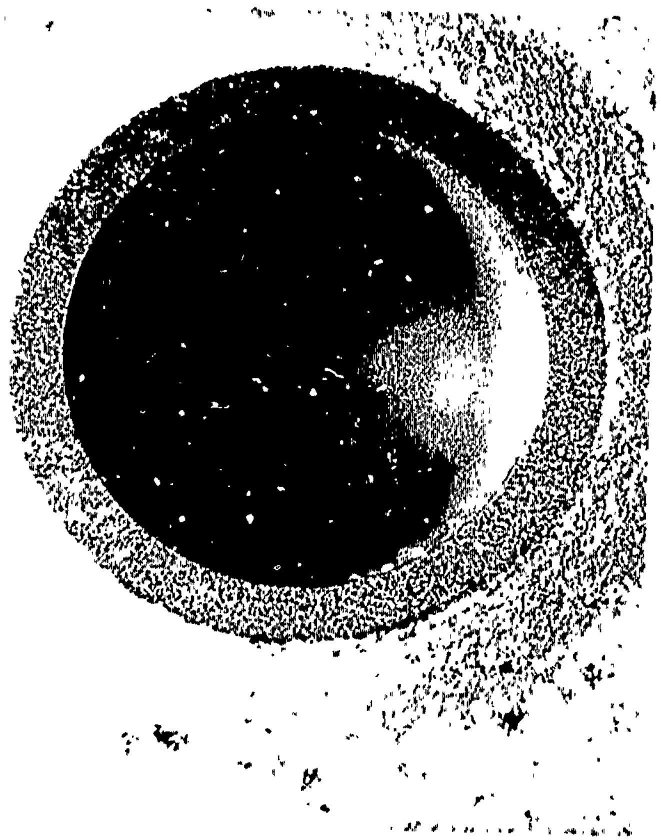

Figure 22. Seat, K-801 Post-Test 19 (25X)

Figure 23. Poppet, K-801, Post-Test 19 (20X)

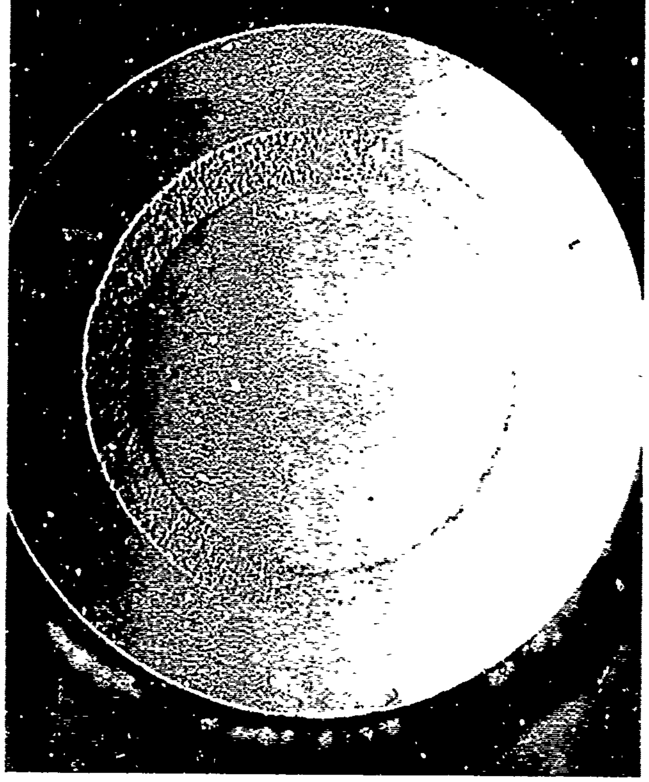

Figure 24. Poppet, K-801, Post-Test '9 (Dark-Field Lighting-20X)

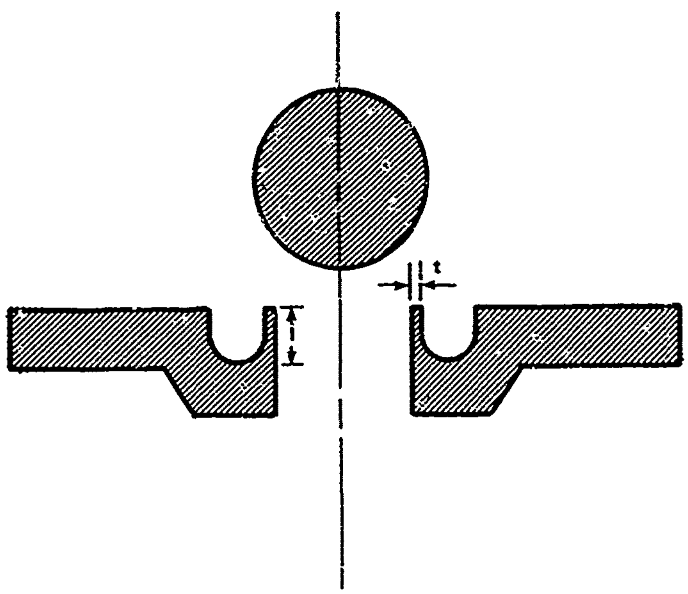

Figure 25. Schematic, Moog Lip-Seal Seat and Poppet (Approximately 10X)

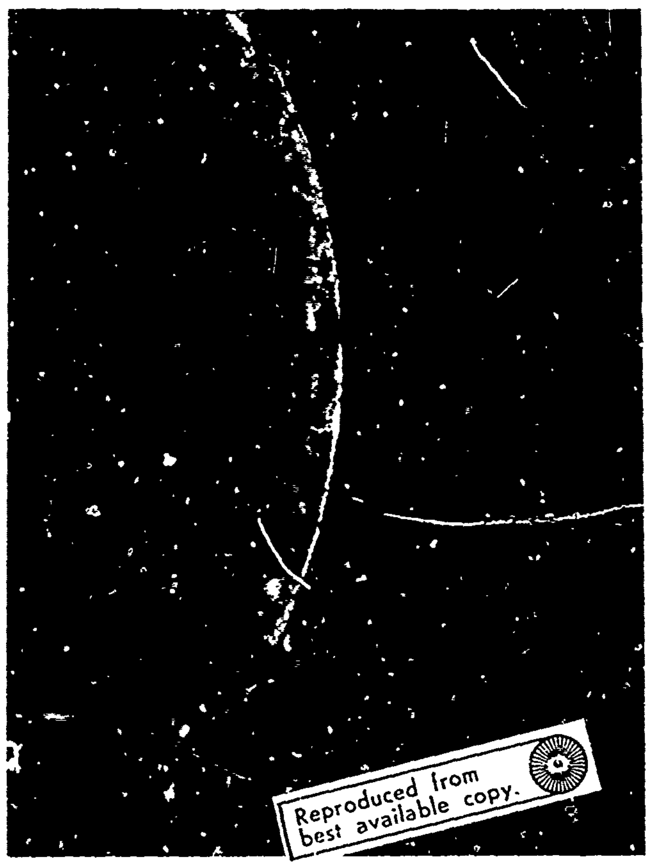

Figure 26. Moog Copper Lip Seat, Post-Test 1 (80X)

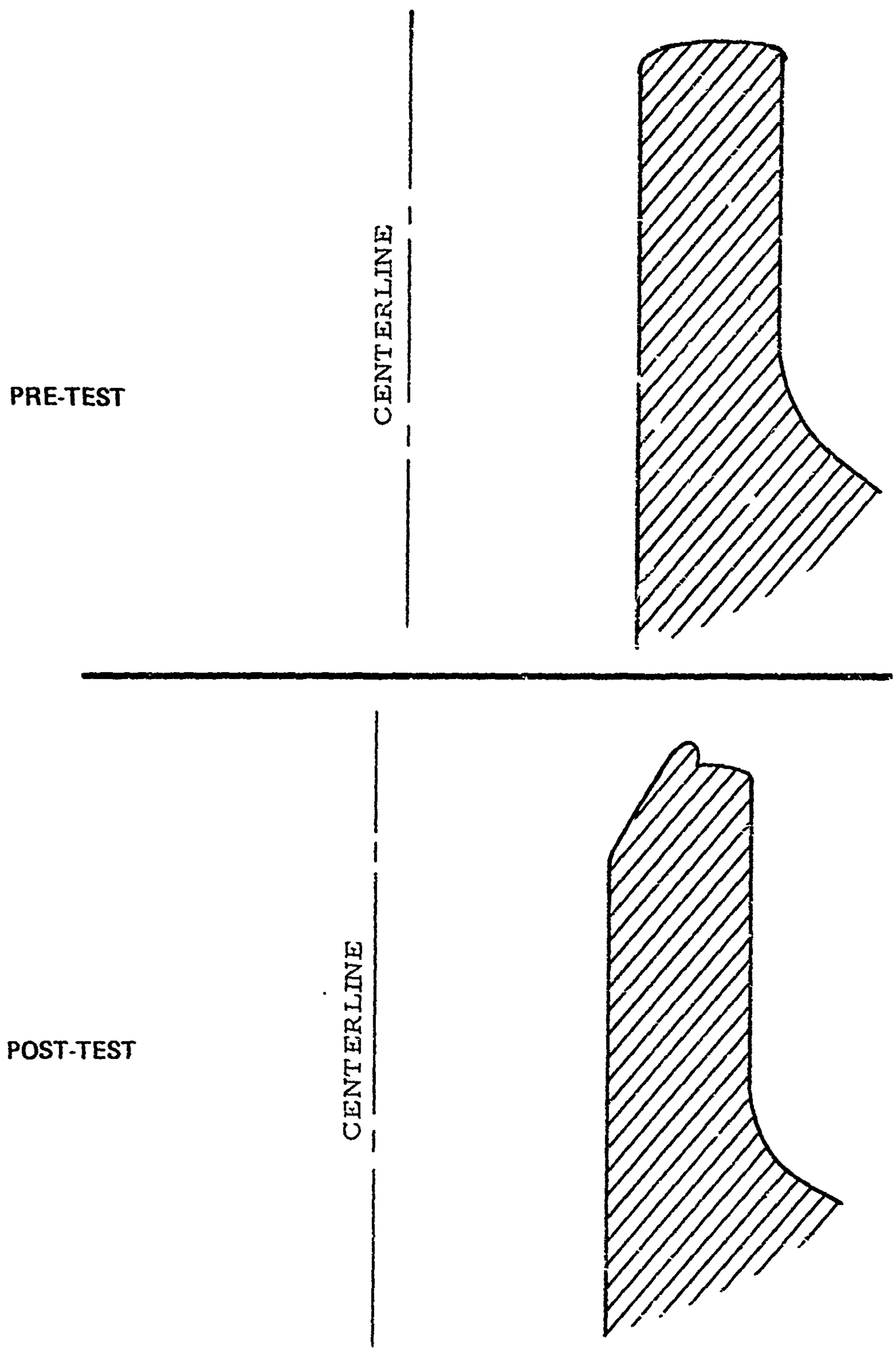

Figure 27. Schematic, Moog Lip Seat Wear

Figure 28. Moog Copper Lip Seat, Post-Test 2 (80X)

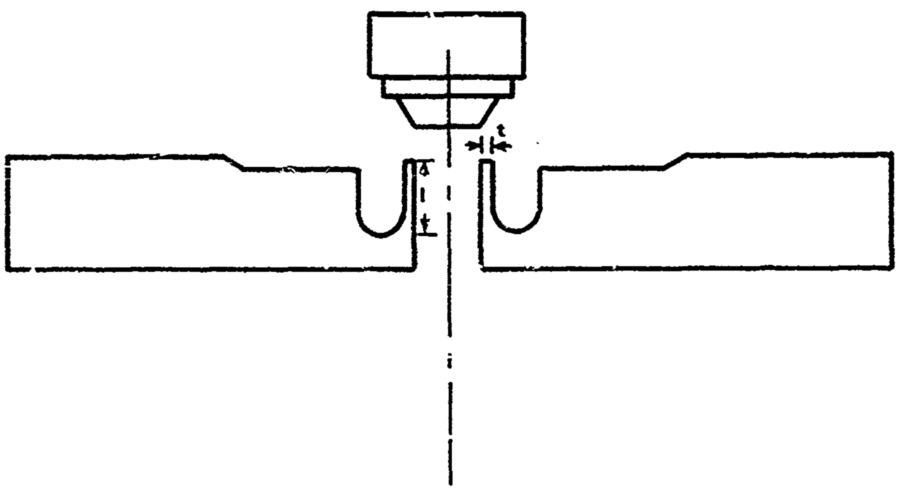

Figure 29. Schematic, AFRPL Conical Poppet/Lip-Seal Design

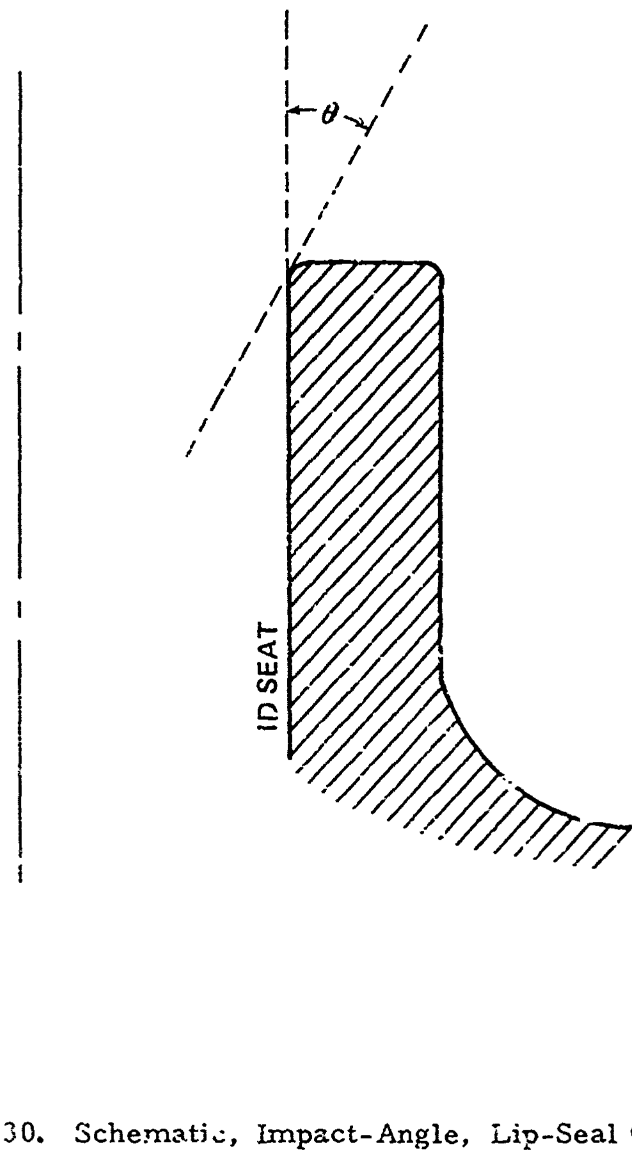

Figure 30. Schematic, Impact-Angle, Lip-Seal Concept

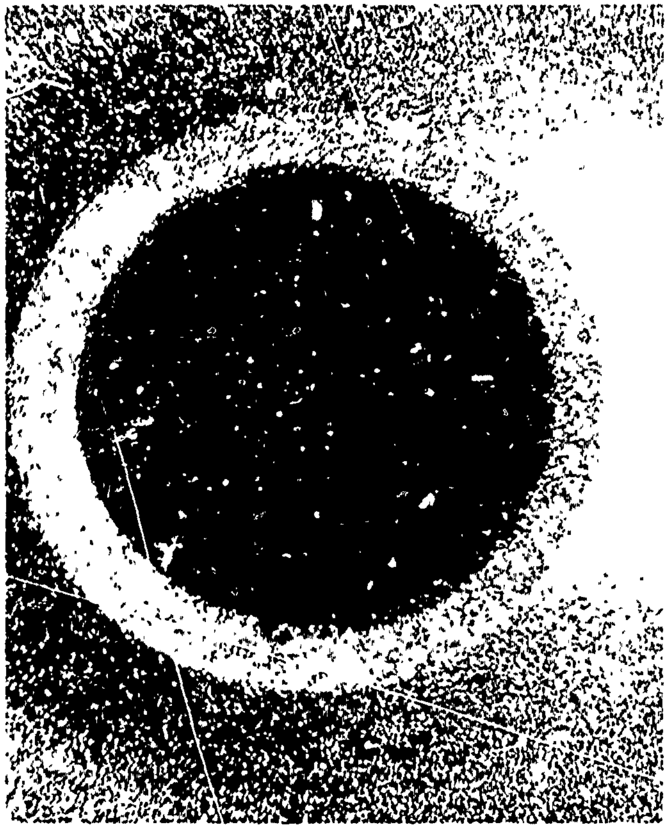

Figure 31. Seat, CVD Tungsten on Ni 301, Prelapped (25X)

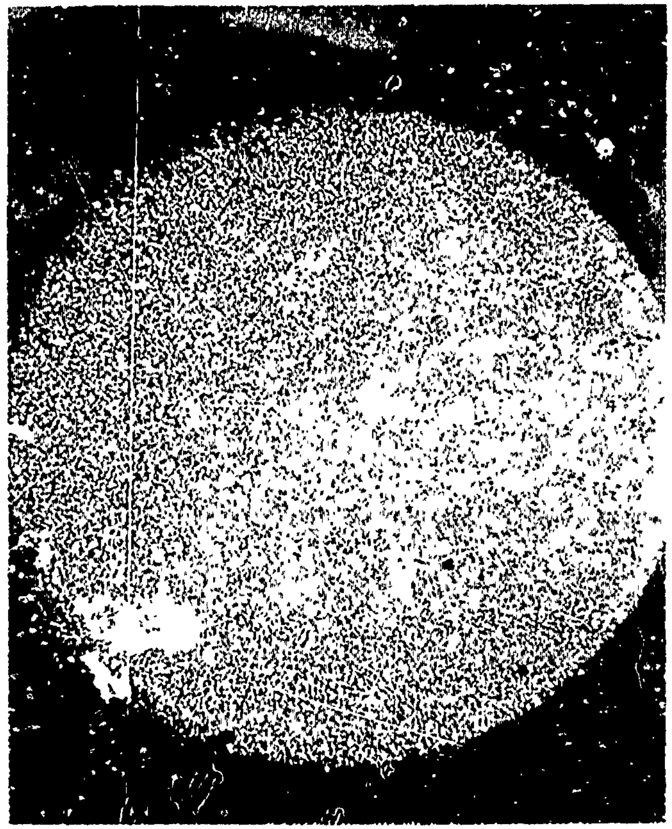

Figure 32. Poppet, CVD Tungsten on Ni 301, Prelapped (20X)

Figure 33. Seat, Ni 301, Final Lapped (25X)

Figure 34. Poppet, Ni 301, Final Lapped (20X)

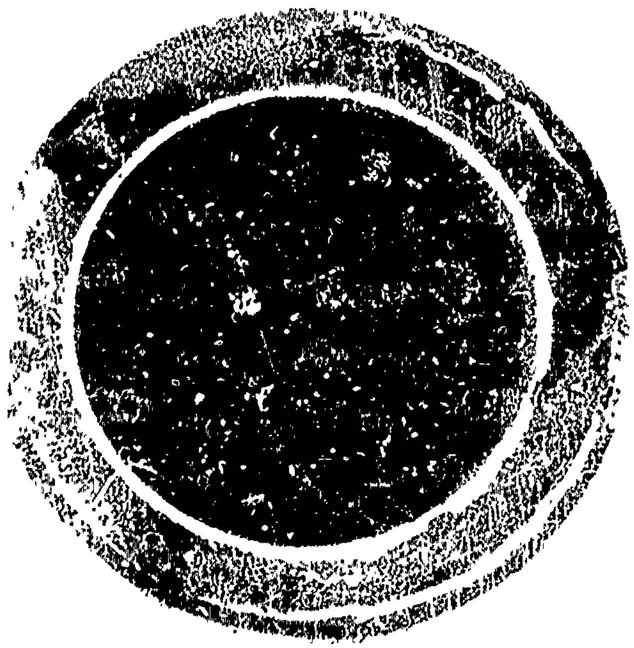

Figure 35. Seat, CVD Tungsten (No. 1) on Ni 301, Postlapped (25X)

Figure 36. Poppet, CVD Tungsten on Ni 301, Postlapped (20X)

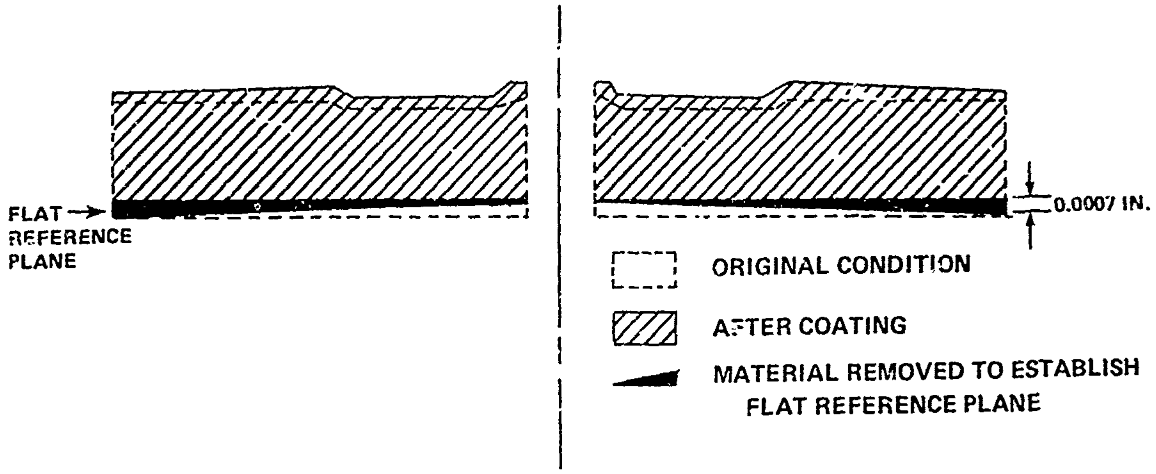

Figure 37. Schematic, Seat-Warping Phenomenon from CVD Process

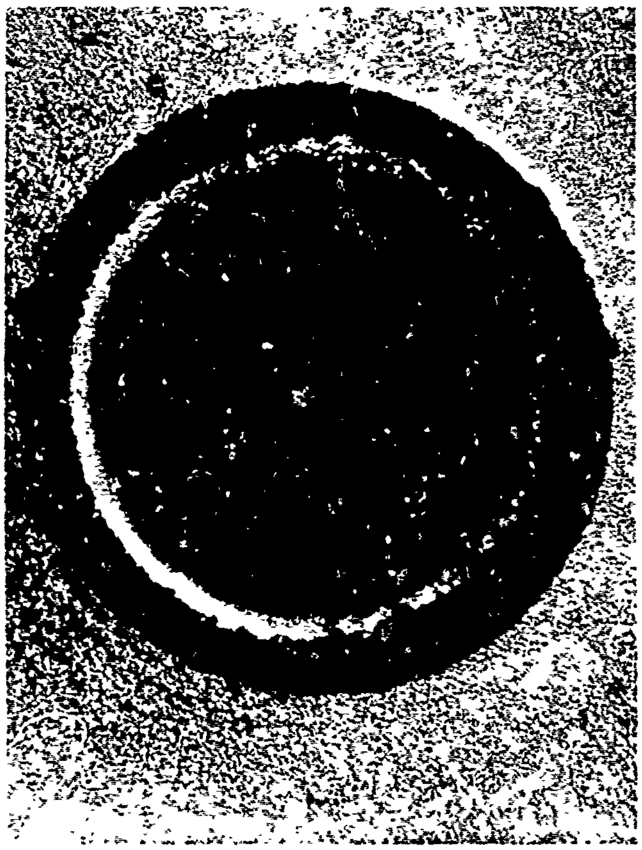

Figure 38. Seat, CVD Tungsten (No. 2)
on Ni 301, Post-lapped (25X)

Figure 39. Se-., CVD Tungsten (No. 2) on Ni 301 Post Dry Cycle (25X)

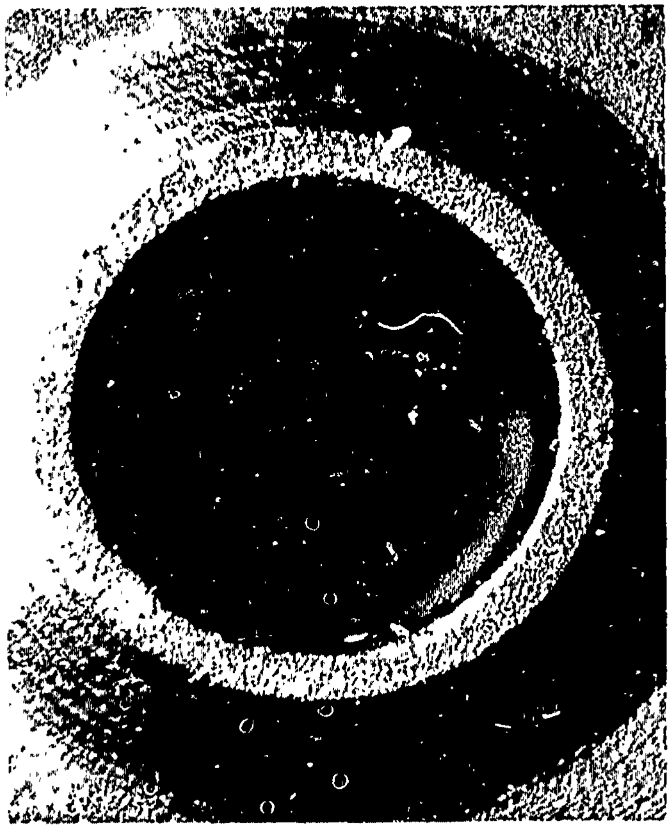

Figure 40. Seat, Aluminum on Ni 301, Prelapped (25X)

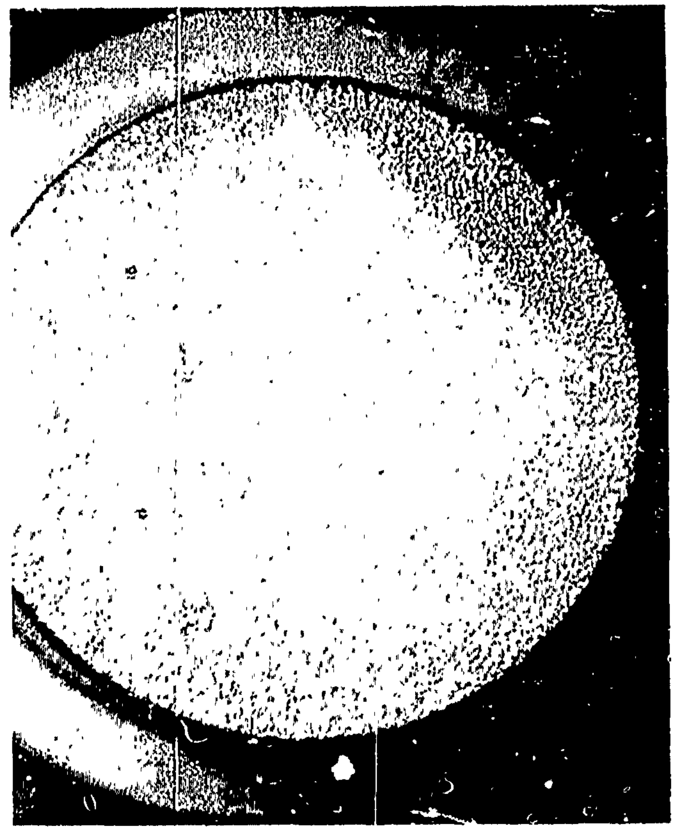

Figure 41. Poppet, Aluminum on Ni 301, Prelapped (20X)

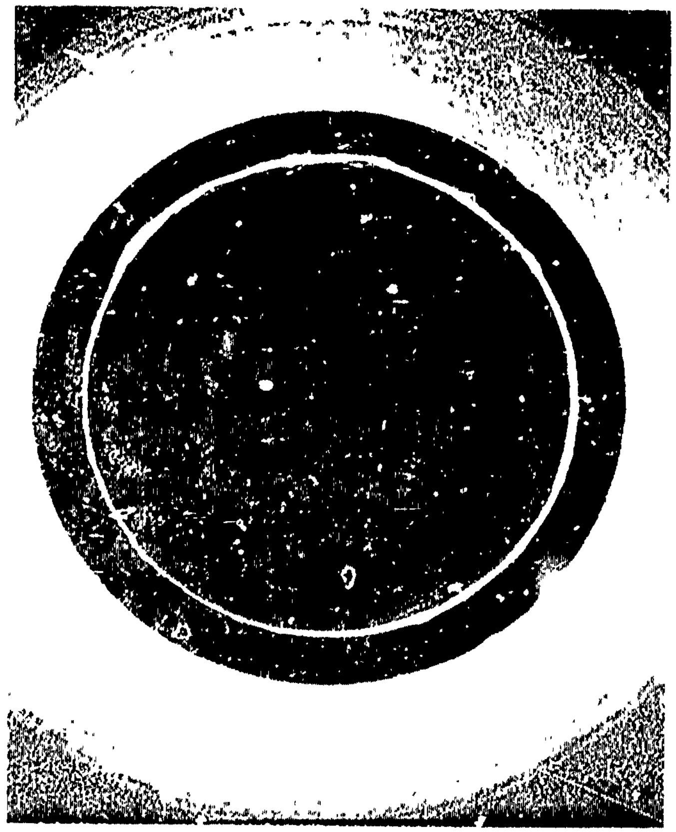

Figure 42. Seat, Aluminum on Ni 301, Post-Lapped (25X)

Figure 43. Poppet, Aluminum on Ni 301 Post-Lapped (20X)

Figure 44. Seat, Aluminum on Ni 301, Post-ClF_5 Testing (25X)

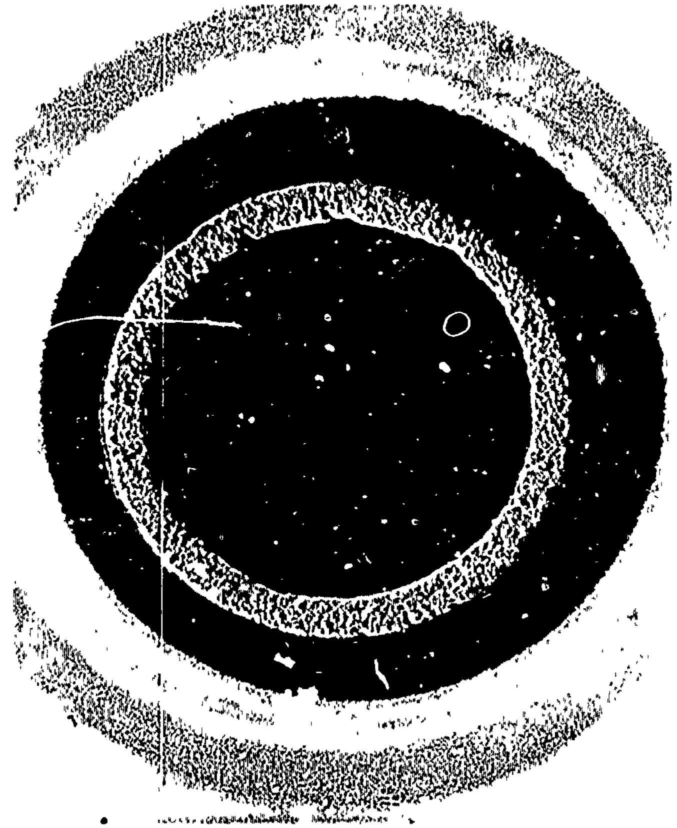

Figure 45. Poppet, Aluminum on Ni 301, Post-ClF_5 Testing (20X)

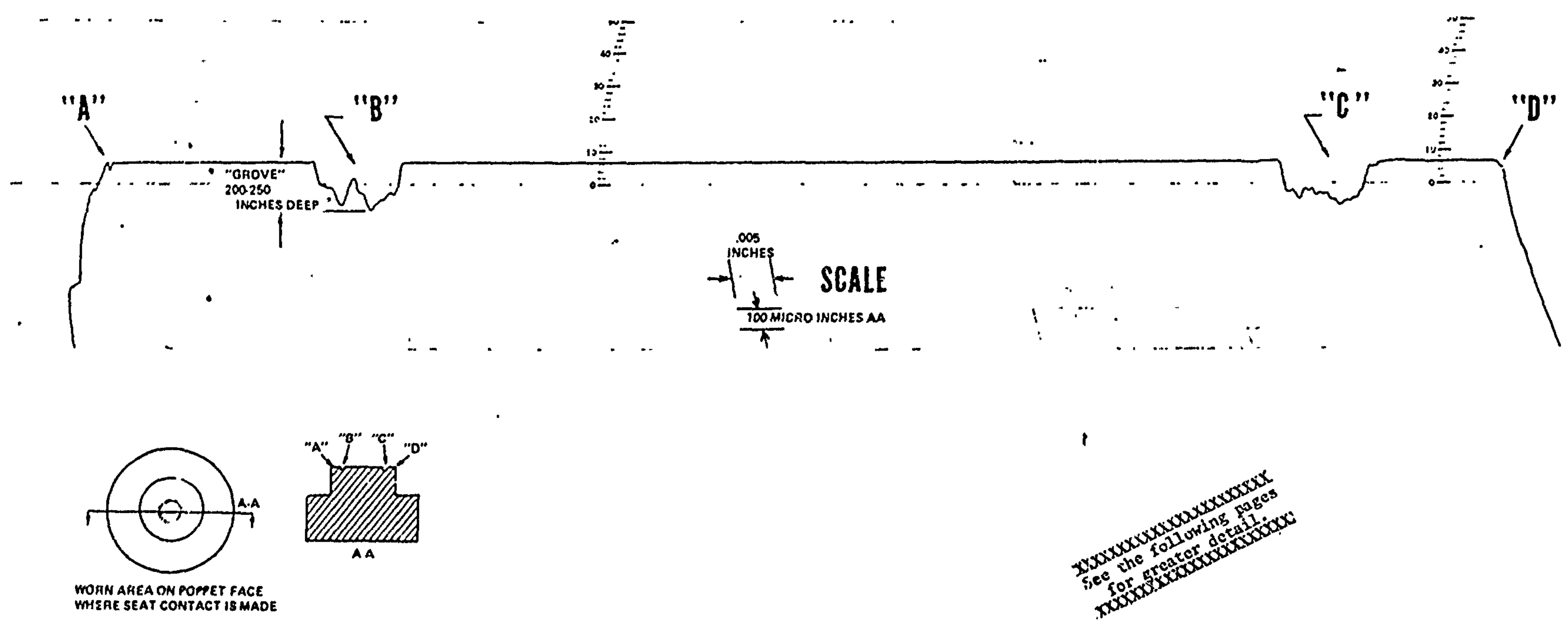

Figure 46. Proficorder Trace, Aluminum on Ni 301 Poppet, Post ClF_5 Testing

See the following pages for greater detail.

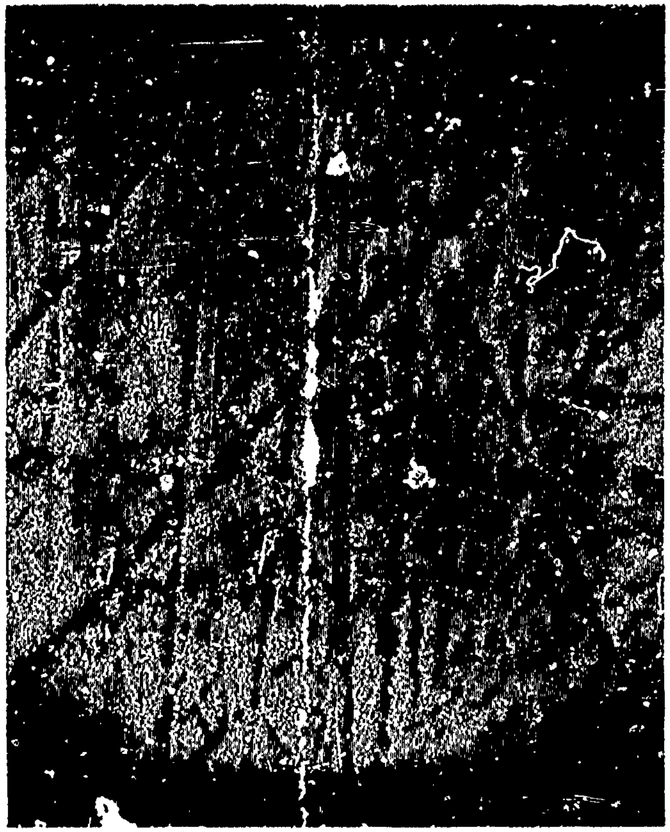

Figure 47. Poppet, Ni 301 (5500 X)

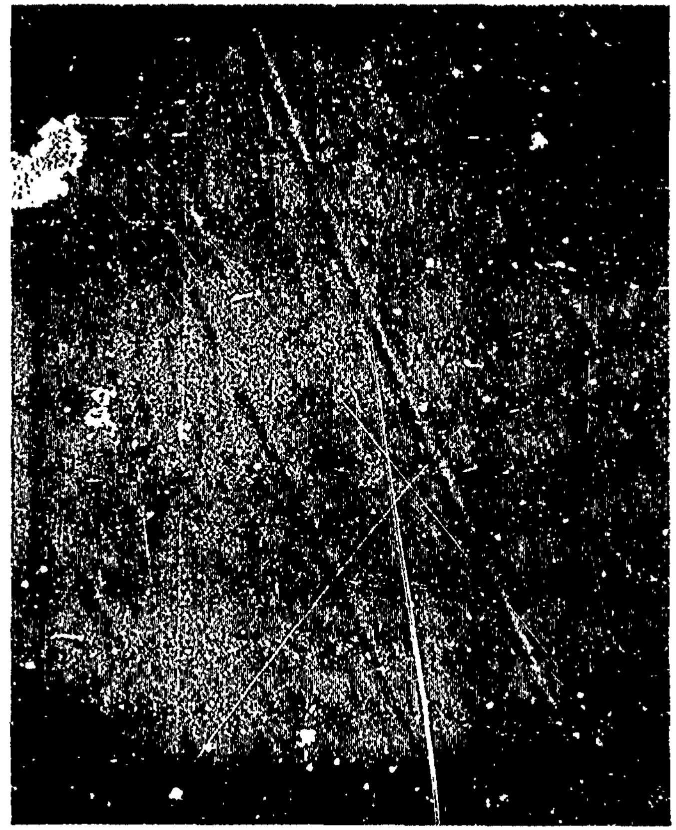

Figure 48. Poppet, 1000Å Tungsten on Ni 301 (5500 X)

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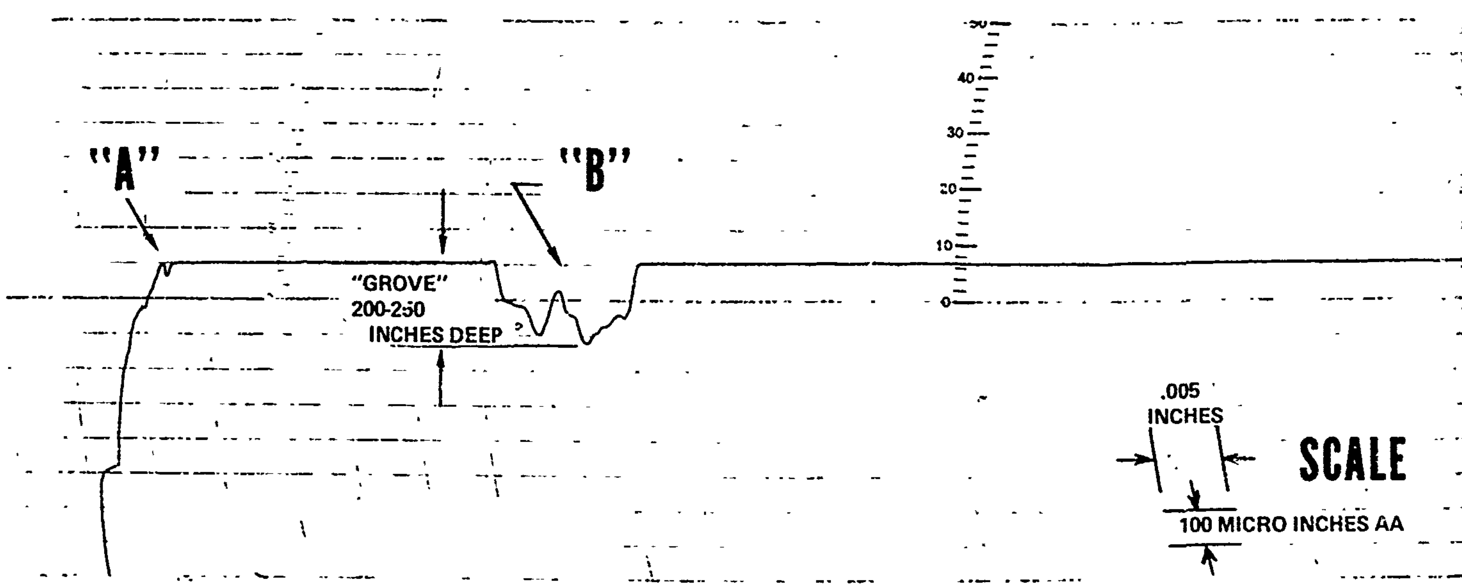


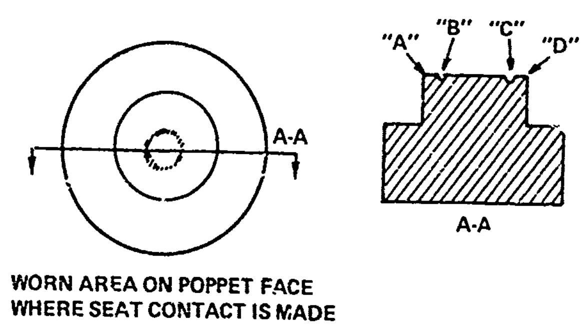
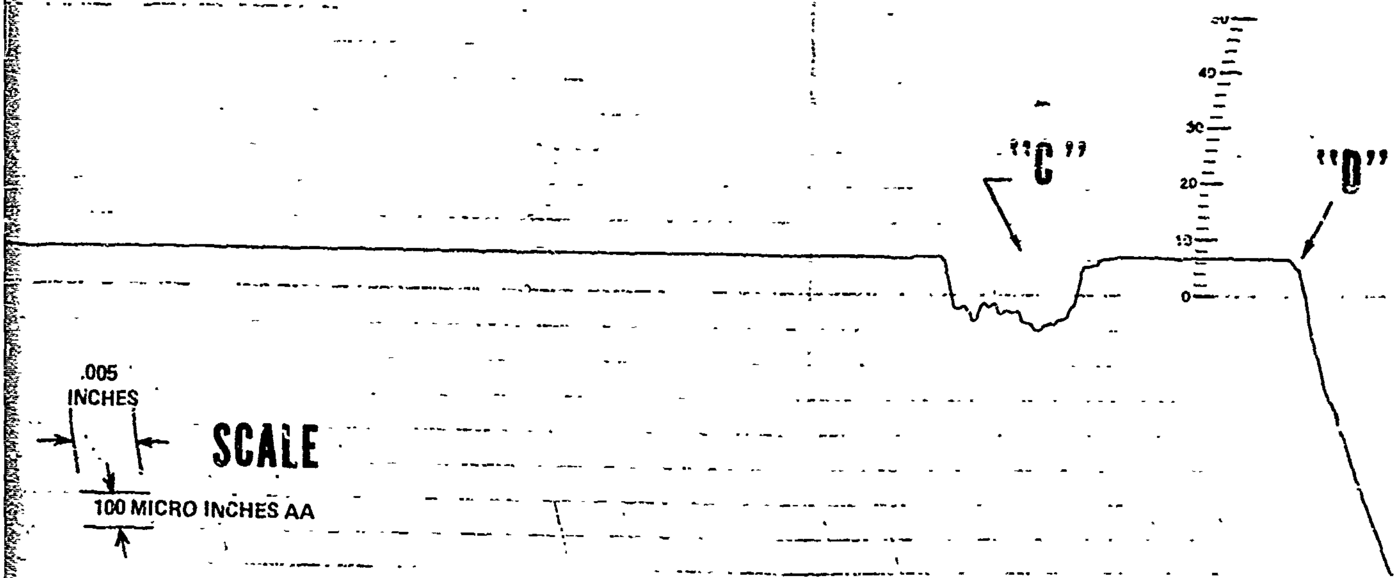


Figure 46. Proficorder Trace, Aluminum on Ni 301 Poppet, Post ClF_5 Testing



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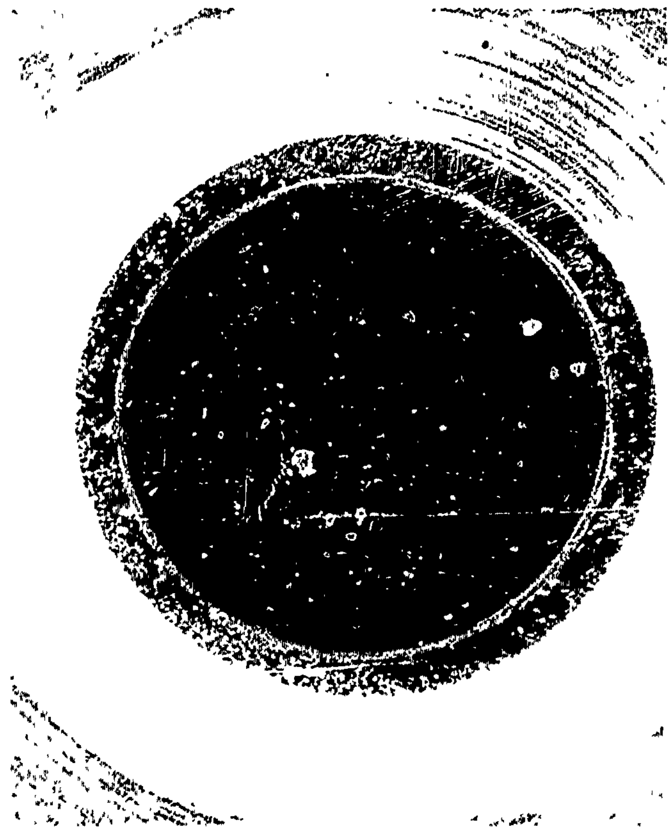

Figure 49. Seat, Sputtered Tungsten (1000Å) on Ni 301, Post-ClF_5 Testing (25X)

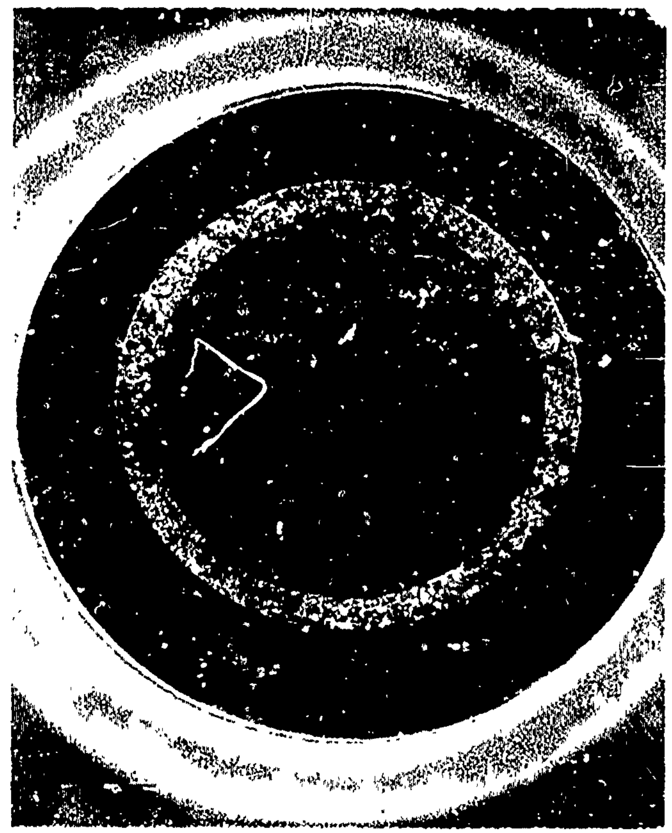

Figure 50. Poppet, Sputtered Tungsten (1000Å) on Ni 301 Post-ClF_5 Testing (20X)

Figure 51. Poppet, 6000Å Tungsten on Ni 301, Pre-Passivation (20X)

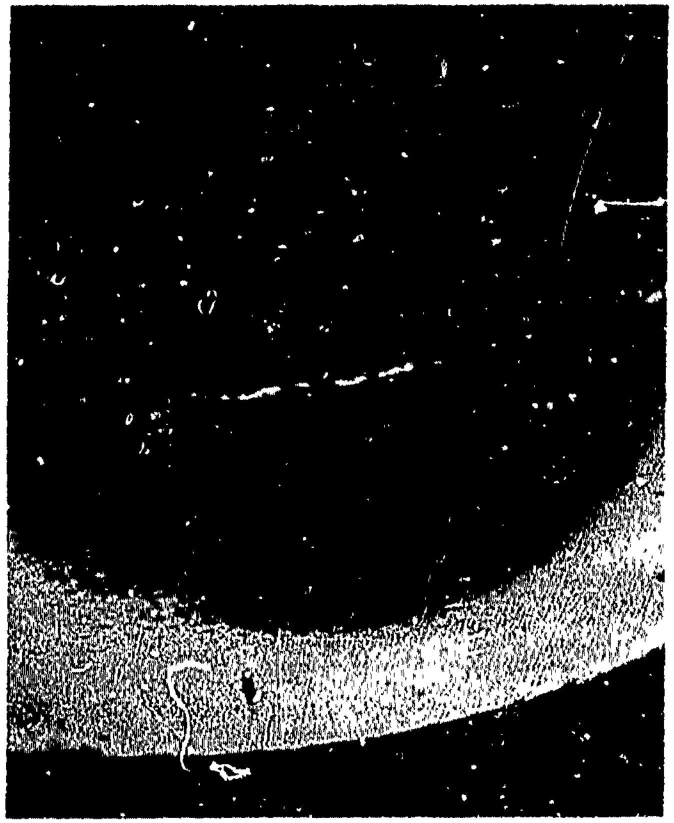

Figure 52. Poppet, 6000Å Tungsten on Ni 301, (80X), Pre-Passivation (Close-Up Seal Area)

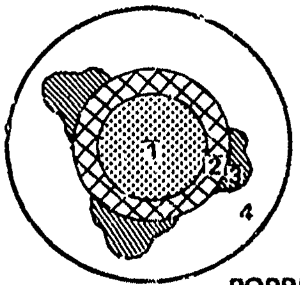

Figure 53. Schematic, Poppet Face "Burn Areas"

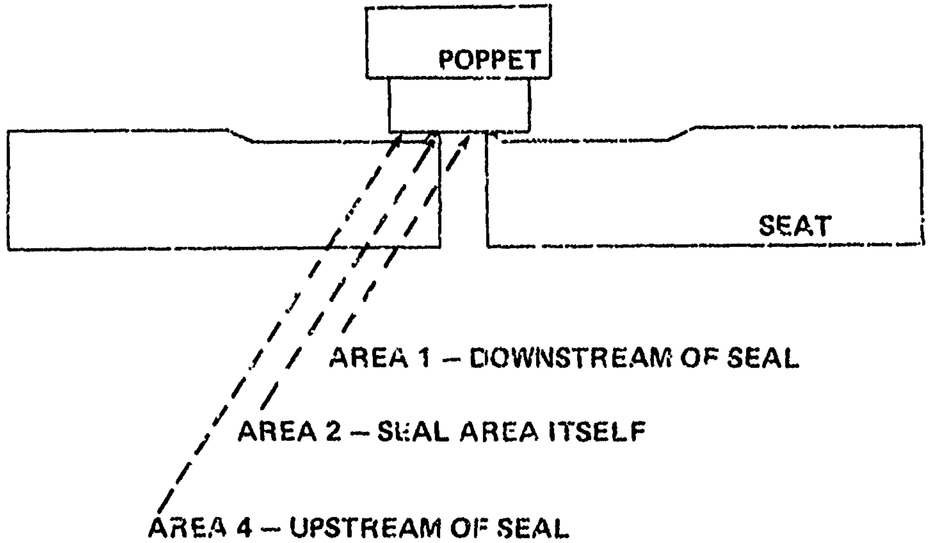

Figure 54. Schematic, Seat/Poppet In Closed Position

Figure 55. Seat, 6000Å Chromiun on Ni 301, Post-ClF_5 Testing (25X)

Figure 56. Poppet, 6000Å Chromiun on Ni 301, Post-ClF_5 Testing (20X)

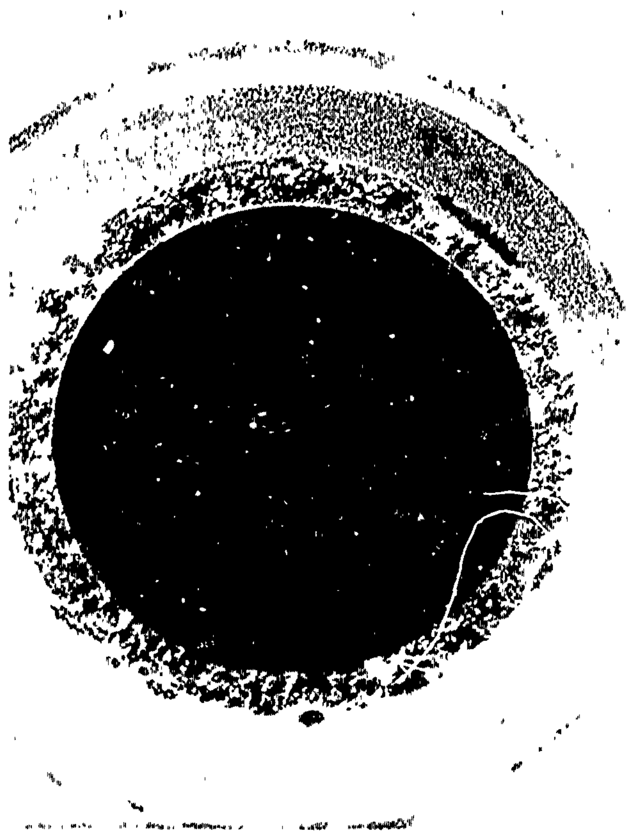

Figure 57. Seat, 6000Å Tungsten on Pyromet X-15, Post-ClF_5 Testing (25X)

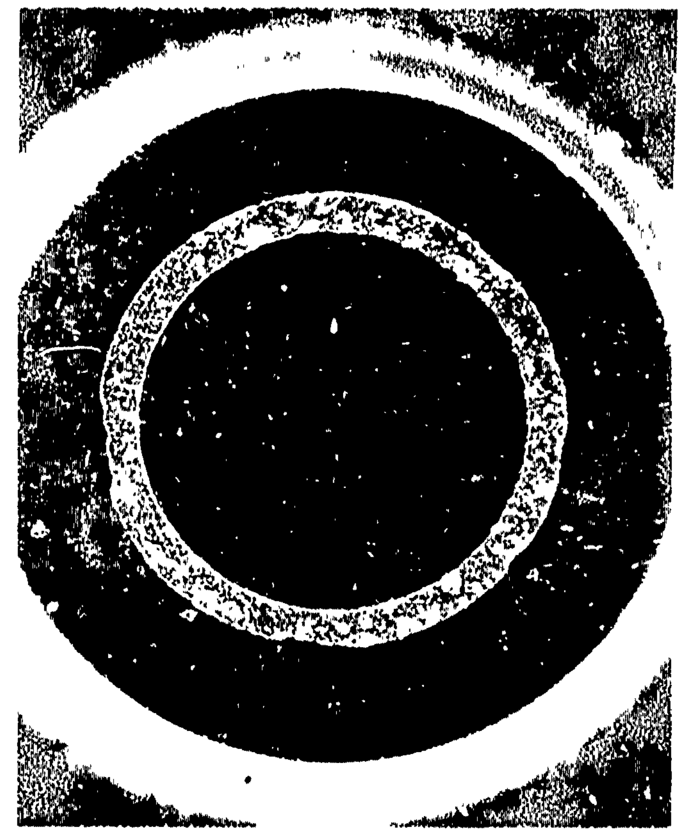

Figure 58. Poppet, 6000Å Tungsten on Pyromet X-15, Post-ClF_5 Testing (20X)

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In his present position as Project Engineer in the Engine Components Branch of the AFRFL's Liquid Rocket Division, he manages and conducts in-house attitude control system (ACS) valve test programs. In addition to the subject ClF_5 ACS valve program just completed, he handles the AFRPL'S Transtage ACS Valve Storage Test Program and is presently investigating fluid flow phenomena in catalytic monopropellant ACS systems.

Sgt Gunderson is a member of the American Institute of Chemical Engineers and Sigma Tau.

APPENDIX A

VALVE CLEANING AND PREPARATION PROCEDURE

Component evaluation in chlorine pentafluoride, as in all fluorinated oxidizers, requires that the hardware be thoroughly cleaned prior to testing. Contamination, such as fingerprints, dirt, oil, metal chips, etc., can all react and create hot spots that may burn through the containing body. All test valves (with their candidate materials and sealing concepts) undergoing evaluation in this program were subjected to a rigid decontamination/preparation procedure prior to testing. What follows is a general description of the step-by-step procedure that was followed during this program to prepare test item for ClF_5 service.

After previous exposure to fluorine or fluorine-containing compounds, the item (valve) is first flushed with tap water and disassembled. Trichloroethylene-vapor degreasing, followed by a second flush with tap water, precedes the pickling stage in which all parts are dipped in an alkaline solution, with subsequent exposure to either a phosphoric (nickel parts) or nitric (stainless steel parts) acid bath. Flushing with demineralized water, rinsing with alcohol or freon and drying in an oven (or with GN_2) completes the cleaning process.

After a visual inspection of all parts for cleanliness, the valve was reassembled with new candidate seats, poppets and seals. The sealing surfaces of the seats and poppets themselves were usually rinsed with freon immediately prior to final assembly.

Pre-test helium leakage measurements at 450 psig by the water displacement method are usually made at this time. If helium leakage is at or below the 30 scc/hour requirement, the item is considered ready for GF_2 passivation and evaluation in ClF_5. If helium leakage measures much more than 30 scc/hour (greater than 50 or 60 scc/hour in most instances) and cycling the valve a few times does not improve the situation, the valve must be disassembled and the sealing surfaces examined. Microscopic examination of the seats and poppets often reveals scratches, cracks, foreign particles, porosity problems or severe misalignment. The test item is not considered ready for testing in ClF_5 until the pre-run leakage is deemed acceptable by the project engineer. When the pre-run leakage measurements are acceptable, the valve is baked at approximately 160°F for 4 hours to remove any residual moisture, specifically from the pores of the seat and poppet material. This last step prior to passivation with GF_2 was initiated with test No. 8 and made procedural from that time on.

Upon installation in the ClF_5 flow loop, the test valve is opened and purged with helium for 15 minutes to remove any new moisture which may have entered. The valve is then isolated and pressurized with gaseous helium to 75 to 80 percent of available K bottle GF_2 pressure. The helium supply is then cut off and the GF_2 K bottle is plumbed into the isolated section containing the test valve. GF_2 is admitted from the K bottle until the pressures equalize and this condition is maintained for 10 minutes. Then the section is "bled-down" through the reactor to a pressure again about 75 percent of available K bottle GF_2 pressure. Then the procedure described above is repeated two more times, after which the item is considered passivated. A second leakage measurement made with helium by the pressure-rise method is recorded, and if acceptable, the item is ready for cycle testing in ClF_5.

The valve being tested is then cycled at a frequency of 8 to 12 Hz (unless specified otherwise) and leak checked periodically to evaluate its performance. In situ leakage measurements are usually made in conjunction with the propellant transfer operation (approximately every 20,000 to 25,000 cycles) but can be taken more or less often depending upon the hardware being evaluated.

After the testing is concluded, and the final leakage measurement is made in situ, the valve is removed from the test system and its leakage is double-checked in a clean room by the helium water-displacement method. For a discussion of the two techniques used to measure helium leakage before, during and after testing, see Appendix B.

APPENDIX B

VALVE LEAKAGE MEASUREMENT TECHNIQUES

WATER-DISPLACEMENT METHOD

When leakage measurements are desired after valve assembly (prior to testing) or after testing is completed, the water-displacement technique is used. Figure B-1 depicts the system, basically a graduated pipette partially filled with liquid (water) and attached to the valve under consideration. The upstream side of the valve is plumbed to a gaseous helium supply regulated to a 450 psig inlet pressure. Helium leaking through the valve displaces the fluid through the pipette. Leak rate in standard cubic centimeters of helium per hour (scc/hour) is determined directly by measuring the pipette length (d) traversed by the upstream meniscus during a specified time increment.

PRESSURE-RISE METHOD

When leakage measurements are needed after passivation and at other appropriate points during cycle testing of the valve, the known-volume, pressure-rise technique is employed. This system allows gross leakage measurements to be made without physical removal of the valve from the test system. Shown schematically in Figure B-2, the in situ system consists simply of an isolated section of the flow loop immediately down-stream of the test item (of known volume) that contains a pressure-sensing device. Figure B-3 shows the pressure-rise/helium leakage relationship derived from the ideal gas law.

Leakage measurements performed in situ are regarded as "go/no-go" indicators to determine whether testing shall continue once the valve is installed in the test system. Both leakage measurement techniques have performed satisfactorily, and on only one occasion (test No. 10) was any significant difference noted. Usually, the pipette method is used to determine the precise post-test helium leakage, unless there may be reason to suspect contamination of the valve as in test No. 10.

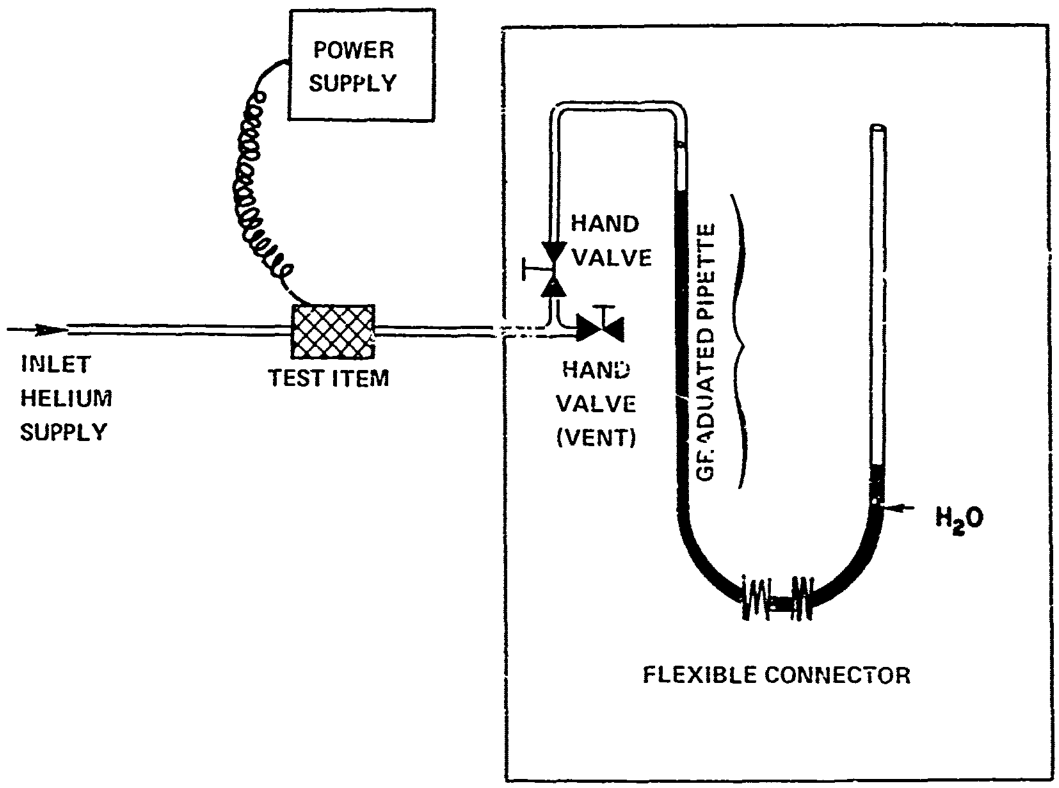

Figure B-1. Schematic Water-Displacement Leakage Measurement Device

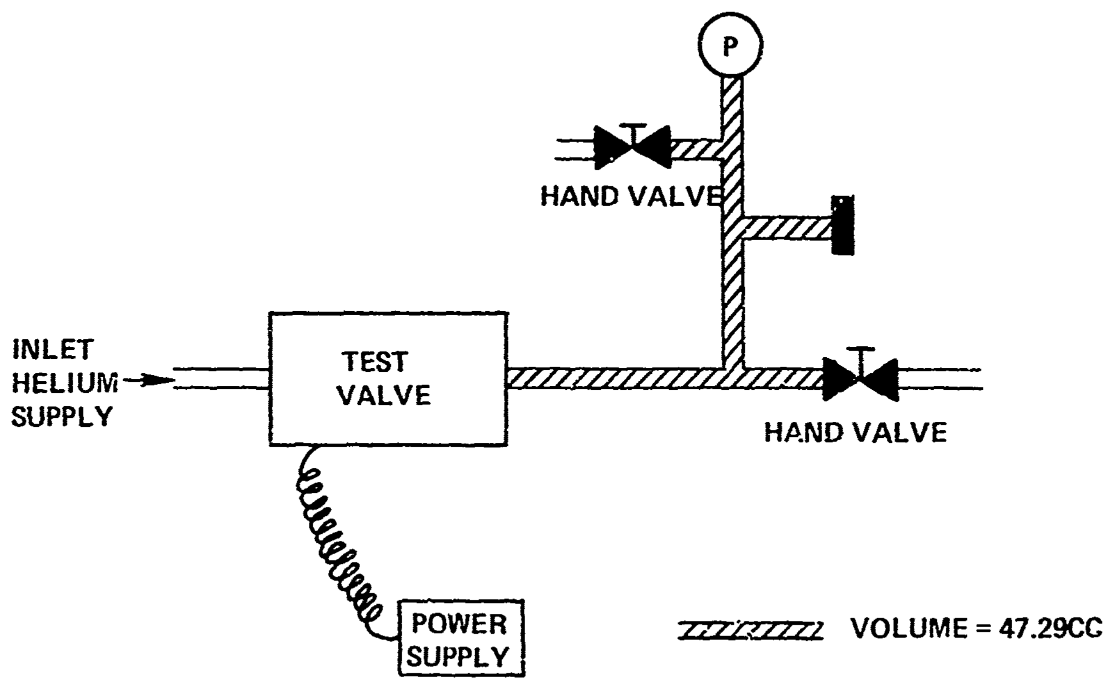

Figure B-2. Schematic Pressure-Rise
Leakage Measurement Device

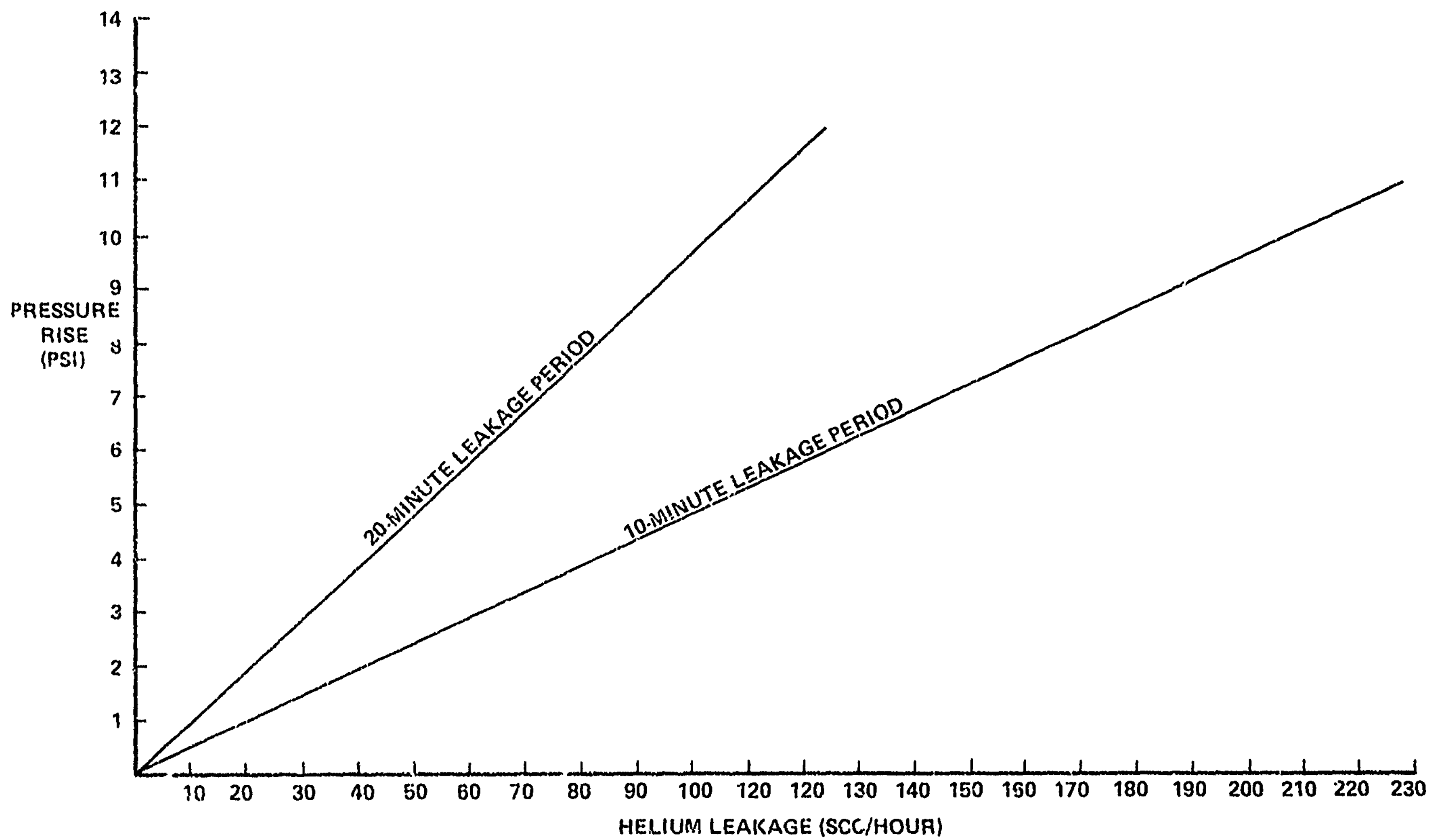

Figure B-3. Control Volume Pressure Rise Versus Helium Leakage