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Key Words:
Wear
Hydrogen
Coating
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Retention:
Permanent

**WEAR TESTING OF STAINLESS STEELS IN HYDROGEN –
EFFECTS OF COATING AND PRE-CHARGE (U)**

**Poh-Sang Lam
Thad M. Adams**

MARCH 2010

Savannah River National Laboratory
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Aiken, SC 29808

**Prepared for the U.S. Department of Energy Under
Contract Number DE-AC09-08SR22470**



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1.0 EXECUTIVE SUMMARY

Phase I wear testing was previously conducted to evaluate the effects of hydrogen atmospheres on wear behavior of stainless steel materials. A Phase II wear testing campaign was run to investigate 1) effects of hydrogen pre-charge and increased load and 2) impact of surface coatings on wear behavior.

Testing was conducted with guidance from the ASTM G 99, "Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus," test standard. Testing was conducted in air or in gaseous hydrogen at a pressure of 13.8 MPa (2000 psig) at room temperature. The hydrogen pre-charge samples were exposed to gaseous hydrogen at an elevated temperature to produce a minimum of 30wppm atomic hydrogen at the sample surface. For the coatings tests, samples were coated with either a plasma nitride or a diamond like carbon (DLC) surface coating applied by a commercial coating vendor.

Material loss due to wear was determined by (1) weighing the specimens (disks and pins) before and after the test; and (2) by tracing the specimen surface roughness across the wear region with a profilometer for both Phase I and Phase II campaigns. Testing was performed for all Phase II samples with a nominal travel distance of 2000 m and a unidirectional disk linear speed at nominally 800 mm/sec. The phase II test data indicate the following:

- Materials transfer and excrescence was observed for metal-to-metal test conditions. Examination of the surface finish of the post-test specimens was made with optical microscopy, scanning electron microscopy (SEM), and profilometer tracing. The results are consistent with previous Phase I results and are indicative of adhesive wear and galling characteristics of stainless steels.
- Similar to Phase I results, AUM10 had a higher wear rate than 316L. Pre-charging with hydrogen showed minimal effect. Increasing load from 45.4 g (0.1lb) to 91 g (0.2 lb) resulted in a small increase in wear.
- Surface coatings reduced the overall wear of the pin and disk materials. For tests with nitride coated disks and uncoated pins increased pin wear was displayed.
- DLC coating appears to offer the most potential as a wear reducing coating—it can be used for either case of both disk and pin coated or one coated and one non-coated, with improved wear resistance.

2.0 INTRODUCTION

Joint research between the Toyota Research Institute North America and the Savannah River National Laboratory (SRNL) was performed to characterize the effects of hydrogen for materials used in fuel cell vehicles. Mechanical equipment for high pressure gaseous hydrogen service includes moving parts where two surfaces may be in contact and slide against each other, resulting in wear that may damage the component. Consideration must be given to the wear characteristics of the alloys of construction; this is especially critical for valves, pumps, bearings, seals and fasteners.

The sliding wear mechanisms of metallic and ceramic components in hydrogen may be expected to be different from the wear experienced in air or inert atmospheres. The interactions of materials with similar hardness and surface reaction behavior could be more deleterious in a hydrogen environment than in air or other atmospheres where protective surface scales may form. Excessive adhesive transfer or fragmentation of these materials could also occur in hydrogen that would not occur in air applications.

Phase I wear testing of stainless steel materials in gaseous hydrogen was completed in FY09. A Phase II program was conceived, to evaluate the impact of pre-charging samples with hydrogen to ensure that the test surface was saturated in hydrogen. A second aspect to the Phase II effort was to evaluate selected commercial coatings to assess their impact on wear behavior in gaseous hydrogen.

Results from this Phase II study together with the initial Phase I results provide key information to designers for material selection for hydrogen service applications involving metallic components with mating dynamic surfaces.

3.0 TEST EQUIPMENT AND SAMPLES

A wear test apparatus has been designed by following the guidelines described in ASTM G 99-05, “Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus,” as schematically shown in Figure 1. The system was designed to allow testing samples in high pressure hydrogen environment by providing a pressure boundary made of Super Alloy HASTELLOY® C276 with outside diameter of 12.7 cm (5 in.), thickness of 3.81 cm (1.5 in.), and the internal cavity height of 17.78 cm (7 in.).

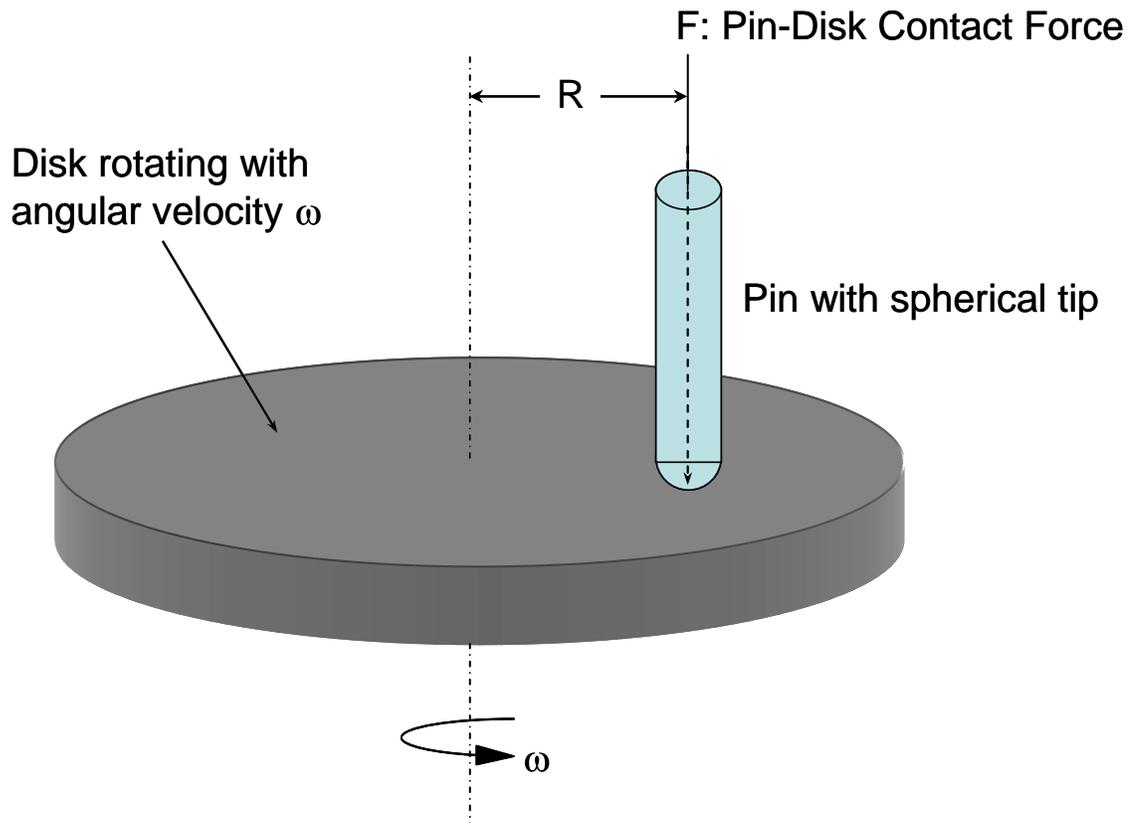


Figure 1. Schematic of disk-pin wear test system (R is the wear track radius).

Figure 1 shows the schematic diagram of the wear test mechanism for a stationary pin and a rotating disk with angular velocity ω (rad/sec). The linear velocity of the disk speed (v) is calculated in term of revolutions per minute ($\text{RPM} = \frac{\omega}{2\pi} \times 60$) as $v = \omega R = \frac{\pi R \times \text{RPM}}{30}$

Disk samples for wear testing were fabricated from 316L and AUM10 stainless steels. As shown in Figure 2, they are 4.76 cm (1.875 in.) in diameter and 0.635 cm (1/4-in.) thick. The disks were machined from 2-inch diameter bar stock (see vendor certificate in Appendix A and Aichi Steel

information sheets in Appendix B). Two parallel flat surfaces were machined on opposite edges of each disk to keep it from rotating in the sample holder. Each disk was engraved with a unique serial number. The test side of the sample is the non-engraved side and was machined to a minimum $0.81 \mu\text{m}$ ($32 \mu\text{-in.}$) arithmetic average (Ra), per ASTM G 99.

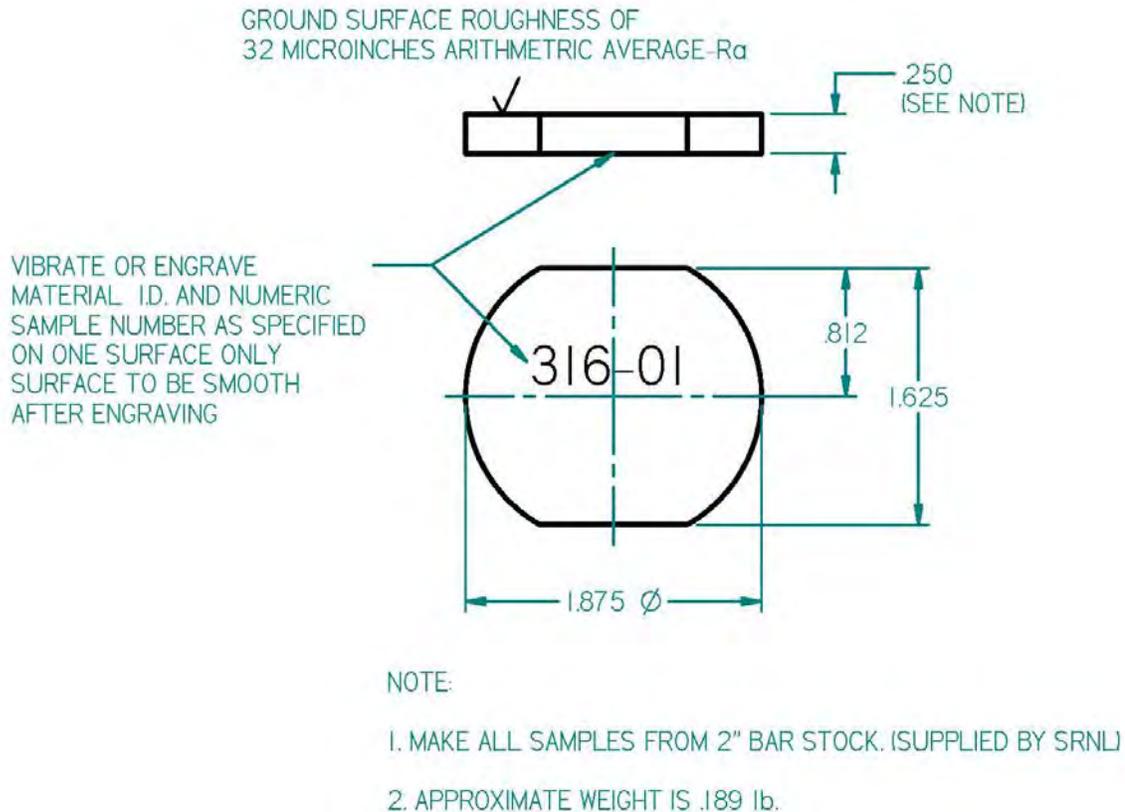


Figure 2. Wear sample disk design and dimensions.

Additionally, pin samples for wear were fabricated from 316L and A286 stainless steel materials. They were machined from 9.525 mm ($3/8\text{-in.}$) rod (see vendor certificates in Appendices B and C, respectively). Each pin is 7.94 cm ($3\frac{1}{4}\text{ in.}$, which is slightly longer than that used previously $2\frac{3}{4}\text{ in}$ [9].) long with a spherical tip on one end and is threaded on the other (Figure 3). Each sample pin is engraved with a unique serial number. The spherical shape was machined on a lathe, so the tip at the end of the pin was not perfectly spherical. For this reason, the pin was mounted in the test machine at a slight angle to the vertical position so the point of contact with the disk would be made on a spherical pin surface.

To maintain the same surface finishing of the specimens as previously tested in the Phase I study [1], the same commercial fabrication vendor was used and the same fabrication instructions were provided. Typical as-finished sample disk and pin can be seen in Figure 4.

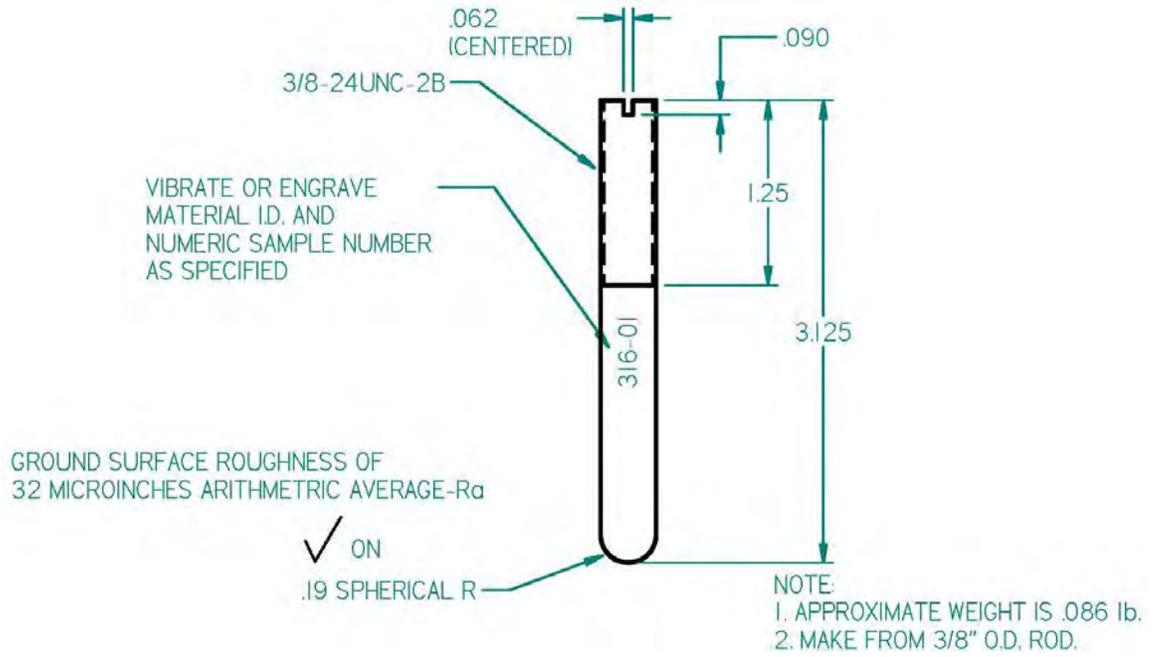


Figure 3. Wear sample pin design and dimensions.



Figure 4. The disk and pin wear test samples.

Tensile properties and material hardness values for the as-received disk and pin materials can be found in the vendor certificates or the material information sheets (Appendices A to D) and are summarized in Table 1.

Table 1. Mechanical properties of disk and pin sample materials

| | 316L Disk (Appendix A) | AUM10 Disk (Appendix B) | 316L Pin (Appendix C) | A286 Pin (Appendix D) |
|--------------------------------|---------------------------|---|--------------------------|--------------------------|
| Bar Stock Diameter cm (in.) | 5.08 (2) | 5.486 ¹ (2.16) | 9.525 (3/8) | 9.525 (3/8) |
| 0.2% Yield Stress MPa (ksi) | 269 (39) | 390 ² (57) | 561 (81.43) | 924 (134) |
| UTS MPa (ksi) | 593 (86) | 530 ² (77) | 688 (99.84) | 1158 (168) |
| Elongation (%) | 66 | 37 ² | 46.01 | 23 |
| RA (%) | 73 | 74 ² | 75.2 | 39 |
| Hardness | HB 185 | HV 183 ¹ (3mm below surface) HV 186 ¹ (D/4) HV 183 ¹ (D/2) HRB 88 ² | HB 212 | HRC 34 HB 321 |

Note 1: Information provided by Toyota Motor Company.
The heat treatment is 920 °C x 0.5 Hr, furnace cool to 650 °C (2.5 Hr),
650 °C x 0.5 Hr, air cool.
HV 183 is equivalent to HB 174.

Note 2: See Appendix B for the information sheets from Aichi Steel Co. (Japan) [2].
The heat treatment is 900 °C x 2 Hr which is different from that in Note 1.
HRB 88 is equivalent to HB 175.

4.0 COATING

Two types of coating were selected to be evaluated for their wear performance in hydrogen service:

(1) Diamond-like-carbon (DLC)

This is a surface coating of diamond like carbon that exhibits a very low sliding friction and high surface hardness. It is possible to mask with areas and selectively coat a substrate. The coating is deposited using a combination of a physical vapor deposition process (PVD) and plasma enhanced chemical vapor deposition process (PECVD). The PVD process will deposit an initial adhesion layer and the PECVD deposits the DLC coating. In the process a carbon carrying gas is introduced into the chamber and a power supply accelerates electrons and cracks the gas breaking the carbon and hydrogen bonds and ionizing the gas as well. The ions are then drawn to the fixture holding the substrates with a negative bias potential forming a coating with a diamond like structure.

The resulting diamond-like coating is structurally amorphous and chemically inert, and may be self-lubricating with very good wear resistance. This coating will replicate the surface finish of the substrate. The typical thickness of the coating is 2 to 5 microns (8×10^{-5} to 2×10^{-4} inches) with Vickers hardness between HV 1000 and 3500 [3].

(2) Plasma nitriding:

Plasma Nitriding is a thermo-chemical diffusion process that will create a hard case several thousandths deep. Unlike a surface coating which will provide properties independent of the substrate being coated, the results from the plasma nitriding process are dependent on the composition of the steel being nitrided. Case depth and hardness will vary depending on the nitriding forming elements present in the alloy. The processing temperature is fixed to between 900 and 950 degrees F. The hardness achieved will generally increase depending on the chromium content in the steel. The surface roughness will increase after nitriding. During the initial heating step of the plasma nitriding process, a hydrogen gas is introduced into the chamber and a pulsed DC bias is supplied to the product to be nitride. The product's surface is bombarded with hydrogen ions during the initial heating cycle. This helps elevate the temperature and cleans the surface as well. When the product reaches the required processing temperature, a gas mixture of nitrogen and hydrogen is introduced and the nitriding cycle begins. The nitrogen will diffuse into the substrate reacting with nitride forming elements and create a hard layer that will continue for up to several thousandths into the steel. On the surface of the product, a compound layer will form and again will vary in thickness depending on the alloy being nitride. This is sitting on the surface and is the primary cause for the uniform growth you receive from the nitriding process. This layer is comprised of epsilon or gamma prime depending on the gas mixture used. If a small amount of carbon is included in the nitrogen and hydrogen gas mixture during the nitriding cycle, the compound layer will be an epsilon layer.

The surface hardness for the plasma nitrided 304 or 316 stainless steels is HRC 62-66 with a total hard case depth 50 to 200 microns (0.002 to 0.008 inches) [3].

Based on the test matrix, nitride coating was applied on twenty AUM10 disks and four 316L pins, and DLC was coated on twenty AUM10 disks and four 316L pins. The DLC-coated disks and pins have a bright black finish (Figure 5) that appears to have the same surface configuration (machine marks) as the original, non-coated specimens except for the black color. The nitride-coated disks (Figure 5) have a dull satin finish that masks the nitrided surface. This soft and rough layer could easily be removed with a Scotch-Brite™ pad, or would quickly wear away at the beginning of the wear test. However, the surface condition of the nitride-coated pins looks similar to that of the DLC-coated pins (Figure 5). This indicated that the nitride surface finish is sensitive to the original substrate surface roughness, and to the curvature of the surface being coated.

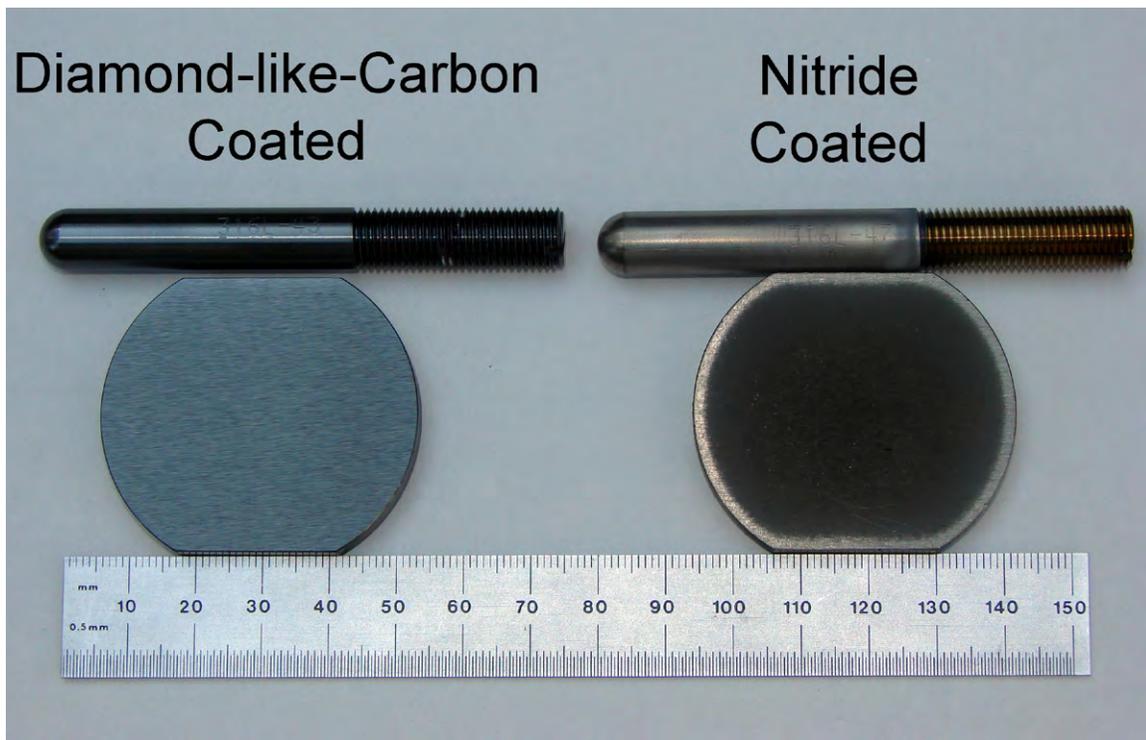


Figure 5. As received DLC and Nitride coated samples.

5.0 HYDROGEN PRE-CHARGE

The test matrix required that five 316L disks and 39 pins (26 316L and 12 A286) be pre-charged with hydrogen for the wear test. None of the coated samples were pre-charged. Note that AUM10 is a soft magnetic ferritic stainless steel based on Type 410L martensitic stainless steel. The hydrogen diffusivity is high in this alloy so pre-charge was also not conducted for AUM10 samples.

The SRNL hydrogen charging facility has a capability of 68.9 MPa (10,000 psi) charge pressure. For these wear samples, an optimum charge condition was determined by SRNL in-house software such that the criterion of 30 wppm (1680 appm for typical stainless steels) at the sample surface could be met. All the samples were nominally pre-charged with 17.2 MPa (2500 psig) research grade hydrogen gas (Appendix E) at 350 °C for one week. The atomic hydrogen concentrations in a disk and at the tip of a pin are shown in Figure 6. It can be seen that the condition of 30 wppm is exceeded everywhere at the tip of a pin, and within 1.5 mm (0.06 inch.) from the surface of a disk. Note that the boundary condition of the numerical calculation maintained at 2531 appm based on the hydrogen charge parameters. A detailed discussion on diffusion properties can be found in Reference [4].

The charge temperature (350 °C) has been shown not to alter the microstructure of the steels. After each charge, the specimens were immediately cooled with liquid nitrogen (-196 °C at 1 atm) and removed from the charge vessel, then transported in dry ice (-78 °C) to a freezer for temporary storage at -44 °C until wear testing was conducted. The length of the freezer storage and a short term exposure to room temperature prior to initiating wear testing will not affect the hydrogen concentration in the samples.

The SRNL charge container has an effective length of 20-23 cm (8-9 inch.) and 3.18 cm (1¼ inch.) in diameter. As a result, the standard disk sample with a diameter of 4.76 cm (1.875 in.) (see Figure 2) must be re-designed for hydrogen pre-charge. An adaptor was fabricated for the smaller pre-charged disks so the wear testing can be conducted normally. Due to the size of the charge tube, only 10 pins (or 5 disks and 5 pins) could be charged at each time.

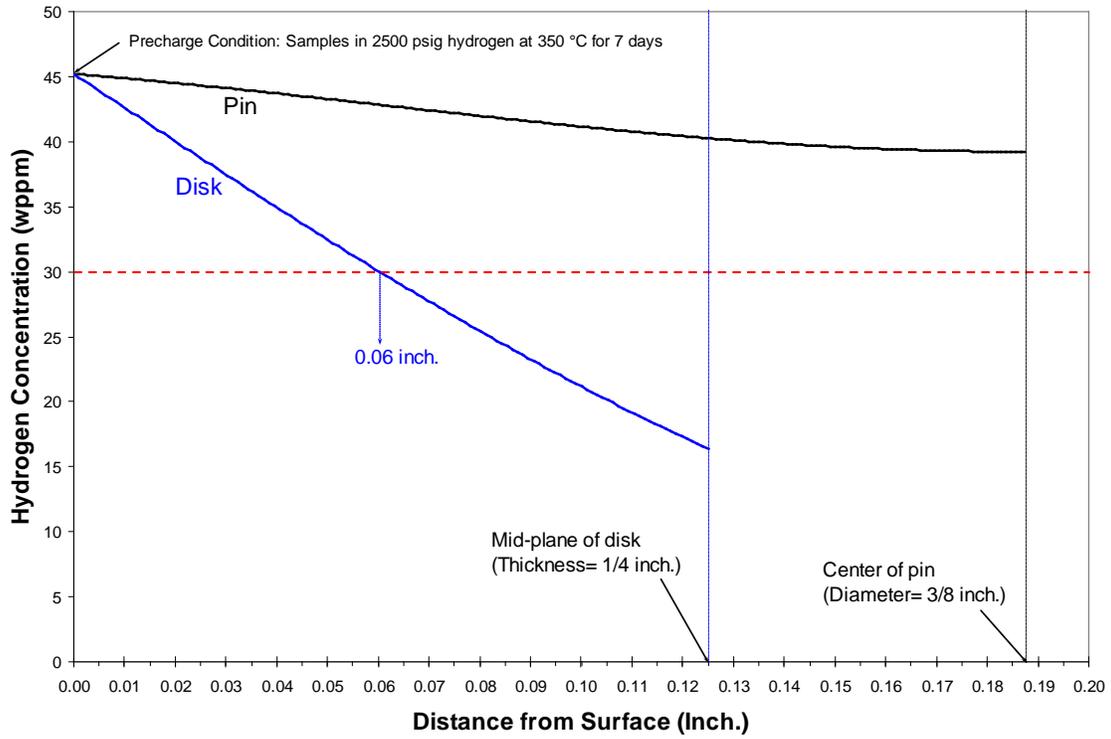


Figure 6. Hydrogen concentration in pre-charged disk and pin samples

6.0 TEST PROCEDURE AND EQUIPMENT

The non-coated sample disks and pins were cleaned in an ultrasonic cleaner, handled without any type of surface contamination, and stored in plastic bags prior to testing. The coated samples were tested with the as-received condition, except the superficial layer on the nitride-coated disks was removed with the Scotch-Brite™ pad. A four- place balance is used to obtain the pre/post-test weights of the samples.

The overall layout of the wear test laboratory can be seen in Figure 7, with the wear mechanism and its components shown in Figure 8. The threaded end of the pin (Figure 3) is screwed into a threaded hole in one end of a first class lever (Figure 9). The pin is prevented from moving by the addition of a lock nut. The fulcrum of the lever is supported by two ball bearings and the other end of the lever is threaded to hold a movable weight. The entire pin-holding assembly is designed to be positioned over the pan of a balance for setting the pin loads. The surface of the balance pan is in the same relative position as the surface of the disk in the test machine. A weight is adjusted until the force of the pin on the balance is exactly 45.4 g (0.1 lb) or 91 g (0.2 lb).

The pin-holding assembly is then mounted over the sample disk (Figure 9-Figure 10) with the load preset to either 45.4 g (0.1 lb) or 91 g (0.2 lb), the radial distance from the center of the disk to the point of contact with the pin is approximately 2.45 cm (1 in.). As discussed earlier, the pin is slightly tilted when the disk rotates because the apex of the pin head is not exactly on a spherical surface due to machining constraints. However, the contact point between the disk and the pin remains on a spherical surface as recommended by ASTM G 99-05.

Figure 11 displays the wear system with the pressure vessel lowered to enclose the disk-pin test mechanism for tests run in a hydrogen atmosphere. The pressure chamber (enclosure) is first evacuated, filled with helium, evacuated again and then filled to the desired pressure with gaseous hydrogen. The hydrogen gas used in this experiment was research grade with a purity of 99.9995% (Appendix E). Pressure is measured simultaneously by two independent pressure gages.

The disk-pin sample holder is rotated by a Magnedrive®. No high pressure rotating seals are required in the system so the leak path is eliminated. The Magnedrive® is driven by a V-belt connected to a variable speed DC motor (Figure 8). The rotational speed is measured by a non-contact laser tachometer connected to a computer which records speed vs. time during each test.

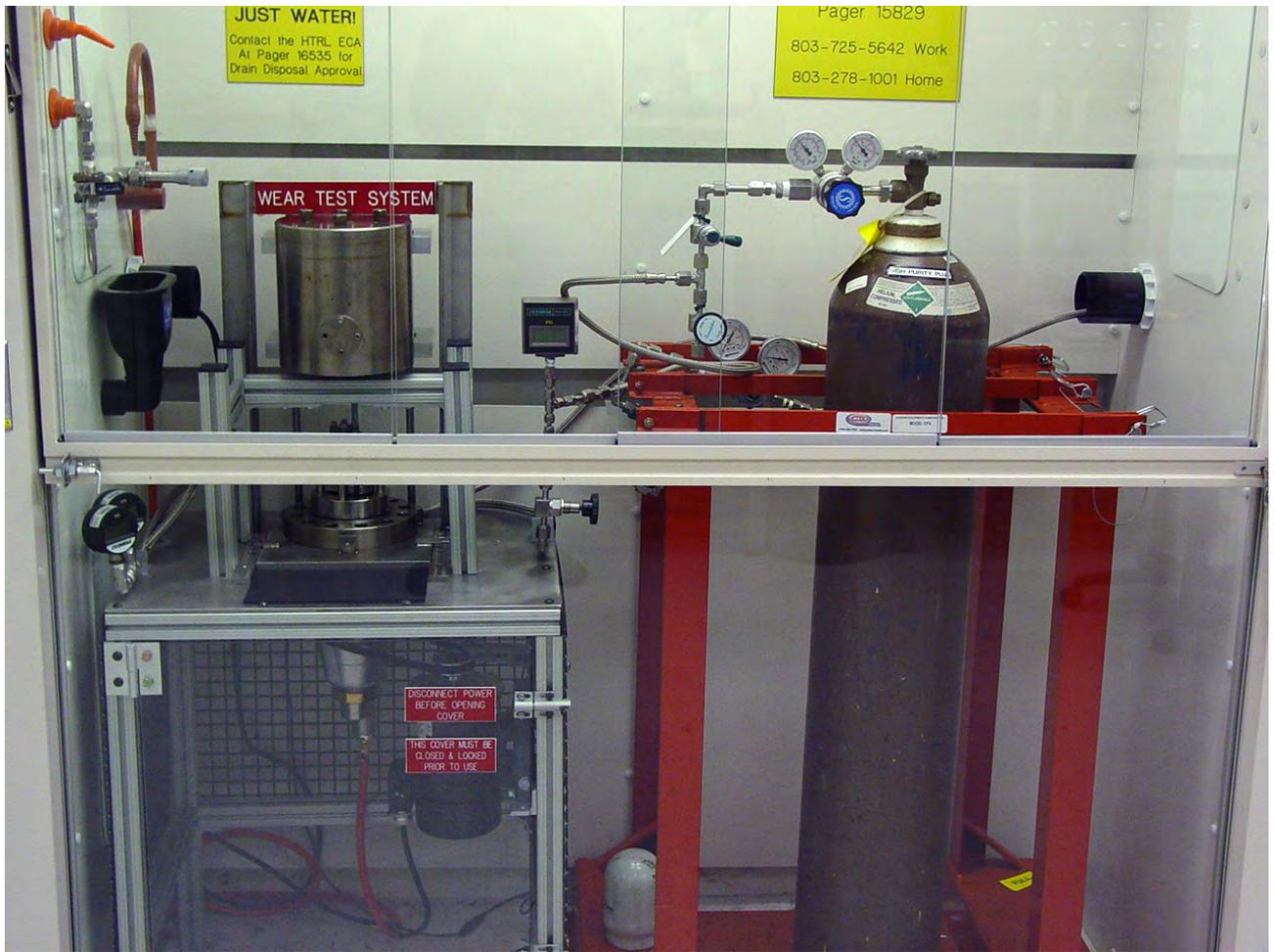


Figure 7 Wear test facilities: Disk-Pin System (left) and hydrogen gas supply cylinder (right).



Figure 8. Wear hydrogen test system from top to bottom: pressure vessel enclosure for hydrogen test (to be lowered when testing), disk-pin holding assembly (samples are mounted), and Magnedrive® with V-belt connected to a DC motor (a non-contact laser tachometer is visible above the motor and behind the V-belt).



Figure 9. Sample disk on a rotating platform and the sample pin mounted in a fixed position.



Figure 10. Sample pin in contact with the disk.



Figure 11. Pressure vessel lowered to enclose the wear test mechanism ready for testing in hydrogen gaseous environment.

7.0 WEAR MEASUREMENT

After the samples were run in the test apparatus at a specified speed and travel distance (nominally 2000 m) under the load of 45.4 g (0.1 lb) or 91 g (0.2 lb), the wear track on the disk and the wear area on the pin were visually examined and photographed (Appendix F). The wear track diameter on the disk and the wear spot diameter on the pin were measured with a Ram Omis II Video Microscope. The wear surface morphology is obtained from a contact profilometer (Model: Rank Taylor-Hobson S5 Form Talysurf Profilometer). For this measurement, a diamond tip stylus is moved in a straight line from a virgin undisturbed area, across the wear region, and ending in the virgin undisturbed area on the other side of the wear track. The trace of each surface profile can also be found in Appendix F. From the trace on the chart, the wear width may be identified by noting a difference in surface finish. Typically, the wear region exhibits higher peak to valley variations, signaling that the surface has been disturbed as a result of testing. However, for some coated disks, especially DLC coated samples, visual examination and the profilometer traces were unable to identify a distinct wear region due to insignificant wear.

The individual disk and pin sample weight changes were obtained by direct measurement (i.e., with a four-place balance). The weighing procedure was as follows:

1. Weigh the test pin and record the weight.
2. Weigh the standard 50 gram weight and record the weight.
3. Weigh the “standard” pin and record the weight.
4. Repeat these three measurements a total of five times.
5. Weigh the test disk and record the weight.
6. Weigh the standard 100 gram weight and record the weight.
7. Weigh the “standard” disk and record the weight.
8. Repeat these three measurements a total of five times

After each test, a repeat of the above measurement process was performed to develop the wear test weight change data.

The wear volume of the samples after wear testing was obtained from the weight change data combined with typical mass density for the stainless steels (7.98 g/cc). In the case of DLC and nitride coatings, their densities are approximated by the pure diamond density (3.5 g/cc) and the typical density for nitriding steels (H7 g/cc).

Volume loss was unable to be measured from the profilometer scans because for most of the samples the surface roughness of the wear track and the virgin area outside of the wear track were very similar—indicating minimal wear. However, the volume loss for the pins could be calculated from the measured wear spot diameter by assuming that it is nearly circular and using the following relationship:

$$\text{Volume Loss} = (\pi R^3/3) \cdot (2 - 3\cos\theta + \cos^3\theta)$$

with $\theta = \sin^{-1}(b/R)$, where R is the pin tip radius (3/16 inch.), and b is the wear spot radius.

The accuracy of the above relationship was evaluated and is demonstrated by the comparison shown Figure 12. This also indicates that the weight measurements of the pin (and the disk) are accurate.

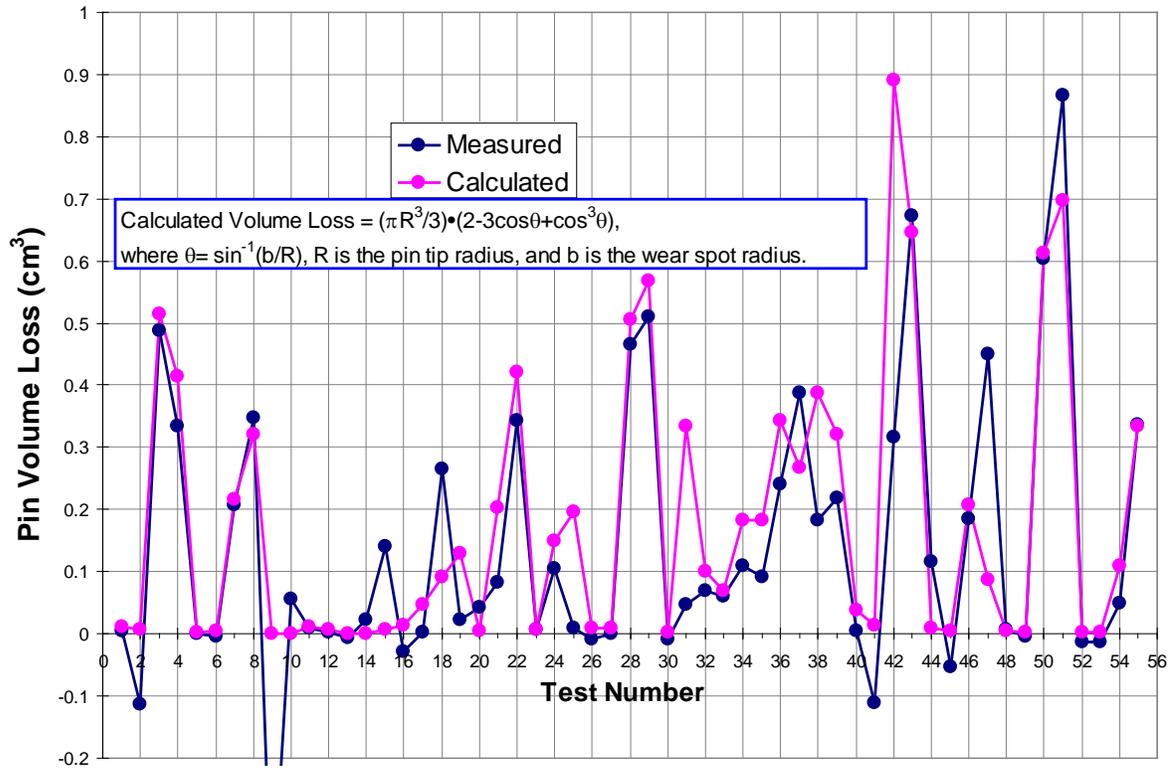


Figure 12. Comparison of pin volume change by weight measurement and by calculation.

Tables 2 to 4 list the test matrix for the Phase II study and the requested test parameters. For the Phase II testing, the nominal test speed is 800 mm/s and the total traveling distance is 2000 m. Note that the actual disk speed and the actual travel distance may vary slightly from the nominal (or target) values, because the actual distance from the center of the disk to the center of the wear track may not coincide exactly with the designed distance of R= 2.54 cm (1 in.).

Table 2. Test Matrix for Non-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Priority | Disk Material | Pin Material | Test Environment | Disk Number | Pin Number |
|----------|----------|------------------|-----------------|-----------------------|-------------|------------|
| 1 | 1 | DLC AUM10 | 316L | Air | AUM10-02 | 316L-06 |
| 2 | 1 | DLC AUM10 | 316L | Air | AUM10-01 | 316L-05 |
| 3 | 1 | Nitride AUM10 | 316L | Air | AUM10-05 | 316L-01 |
| 4 | 1 | Nitride AUM10 | 316L | Air | AUM10-06 | 316L-35 |
| 5 | 1 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-04 | 316L-08 |
| 6 | 1 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-03 | 316L-07 |
| 7 | 1 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-08 | 316L-04 |
| 8 | 1 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-07 | 316L-03 |
| 9 | 2 | DLC AUM10 | DLC 316L | Air | AUM10-09 | 316L-41 |
| 10 | 2 | DLC AUM10 | DLC 316L | Air | AUM10-10 | 316L-42 |
| 11 | 2 | Nitride AUM10 | Nitride 316L | Air | AUM10-12 | 316L-48 |
| 12 | 2 | Nitride AUM10 | Nitride 316L | Air | AUM10-16 | 316L-47 |
| 13 | 2 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | AUM10-33 | 316L-43 |
| 14 | 2 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | AUM10-34 | 316L-44 |
| 15 | 2 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | AUM10-15 | 316L-46 |
| 16 | 2 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | AUM10-14 | 316L-45 |

Table 3. Test Matrix for Pre-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Priority | Disk Material | Pin Material | Test Environment | Disk Number | Pin Number |
|----------|----------|------------------|--------------|-----------------------|-------------|------------|
| 17 | 1 | 316L | 316L | Hydrogen 2000 psig | 316L-04 | 316L-34 |
| 18 | 1 | 316L | 316L | Hydrogen 2000 psig | 316L-02 | 316L-14 |
| 19 | 1 | 316L | 316L | Hydrogen 2000 psig | 316L-03 | 316L-15 |
| 20 | 1 | AUM10 | 316L | Hydrogen 2000 psig | AUM10-60 | 316L-25 |
| 21 | 1 | AUM10 | 316L | Hydrogen 2000 psig | AUM10-61 | 316L-26 |
| 22 | 1 | AUM10 | 316L | Hydrogen 2000 psig | AUM10-62 | 316L-18 |
| 23 | 1 | AUM10 | A286 | Hydrogen 2000 psig | AUM10-63 | A286L-14 |
| 24 | 1 | AUM10 | A286 | Hydrogen 2000 psig | AUM10-41 | A286L-19 |
| 25 | 1 | AUM10 | A286 | Hydrogen 2000 psig | AUM10-42 | A286L-01 |
| 26 | 2 | DLC AUM10 | 316L | Air | AUM10-13 | 316L-22 |
| 27 | 2 | DLC AUM10 | 316L | Air | AUM10-22 | 316L-23 |
| 28 | 2 | Nitride AUM10 | 316L | Air | AUM10-28 | 316L-20 |
| 29 | 2 | Nitride AUM10 | 316L | Air | AUM10-27 | 316L-21 |
| 30 | 2 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-25 | 316L-29 |
| 31 | 2 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-26 | 316L-30 |
| 32 | 2 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-31 | 316L-27 |
| 33 | 2 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-32 | 316L-28 |

Table 4. Test Matrix for Pre-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 91 g or 0.2 lb)

| Test No. | Priority | Disk Material | Pin Material | Test Environment | Disk Number | Pin Number |
|----------|----------|------------------|--------------|-----------------------|-------------|------------|
| 34 | 1 | 316L | 316L | Hydrogen 2000 psig | 316L-01 | 316L-16 |
| 35 | 1 | 316L | 316L | Hydrogen 2000 psig | 316L-05 | 316L-17 |
| 36 | 1 | AUM10 | 316L | Hydrogen 2000 psig | AUM10-43 | 316L-32 |
| 37 | 1 | AUM10 | 316L | Hydrogen 2000 psig | AUM10-44 | 316L-33 |
| 38 | 1 | AUM10 | A286 | Hydrogen 2000 psig | AUM10-45 | A286-02 |
| 39 | 1 | AUM10 | A286 | Hydrogen 2000 psig | AUM10-46 | A286-03 |
| 40 | 2 | DLC AUM10 | A286 | Air | AUM10-11 | A286-11 |
| 41 | 2 | DLC AUM10 | A286 | Air | AUM10-17 | A286-12 |
| 42 | 2 | Nitride AUM10 | A286 | Air | AUM10-19 | A286-13 |
| 43 | 2 | Nitride AUM10 | A286 | Air | AUM10-40 | A286-04 |
| 44 | 2 | DLC AUM10 | A286 | Hydrogen 2000 psig | AUM10-18 | A286-05 |
| 45 | 2 | DLC AUM10 | A286 | Hydrogen 2000 psig | AUM10-21 | A286-06 |
| 46 | 2 | Nitride AUM10 | A286 | Hydrogen 2000 psig | AUM10-20 | A286-07 |
| 47 | 2 | Nitride AUM10 | A286 | Hydrogen 2000 psig | AUM10-24 | A286-08 |
| 48 | 2 | DLC AUM10 | 316L | Air | AUM10-29 | 316L-24 |
| 49 | 2 | DLC AUM10 | 316L | Air | AUM10-30 | 316L-11 |
| 50 | 2 | Nitride AUM10 | 316L | Air | AUM10-23 | 316L-12 |
| 51 | 2 | Nitride AUM10 | 316L | Air | AUM10-37 | 316L-13 |
| 52 | 2 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-36 | 316L-31 |
| 53 | 2 | DLC AUM10 | 316L | Hydrogen 2000 psig | AUM10-35 | 316L-52 |
| 54 | 2 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-38 | 316L-49 |
| 55 | 2 | Nitride AUM10 | 316L | Hydrogen 2000 psig | AUM10-39 | 316L-50 |

8.0 POST-TEST WEAR CHARACTERIZATION

8.1 WEAR SURFACE CHARACTERIZATION

Following the initial visual examination of the disk and pin samples after testing, photographs were used to document the wear tracks and post-test surfaces. They are displayed along with the traces from contact profilometer in Appendix F. Selected sample photographs and profilometer traces are shown in Figure 13-Figure 26. From these micrographs a few general conclusions can be drawn:

1. Tests conducted with non-coated disk and non-coated pin combinations displayed the most significant wear tracks visually
2. Coated disk-pin samples—either coated disk-coated pin or coated disk-non-coated pin—displayed minimal to no wear tracks visually

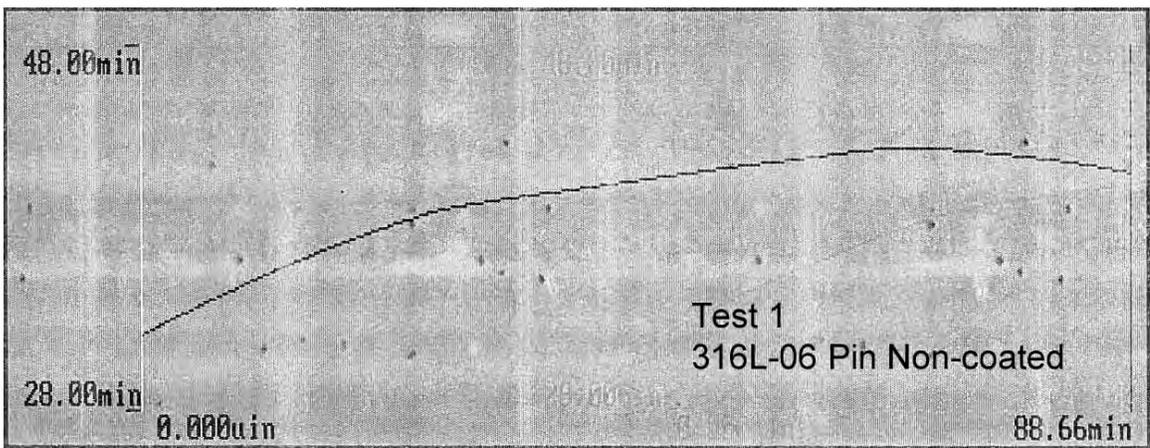
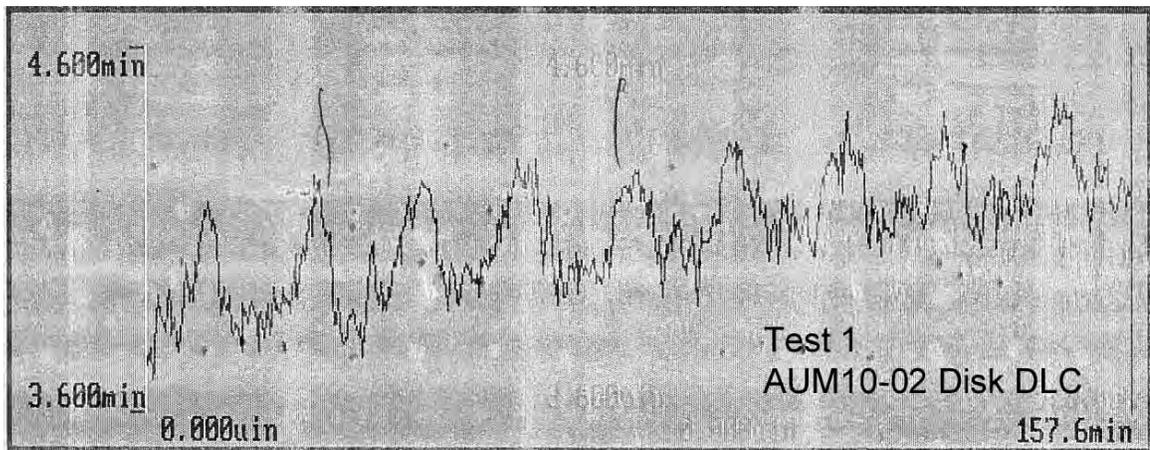
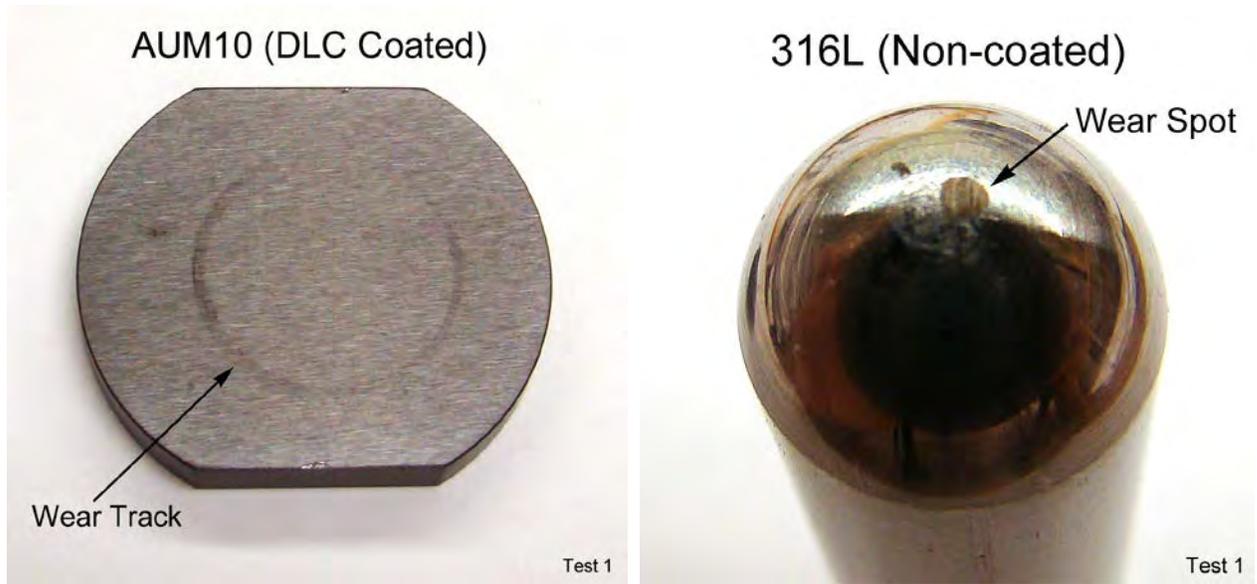


Figure 13. Post-Test Wear Surface Photographs and Profilometer Data for Test #1

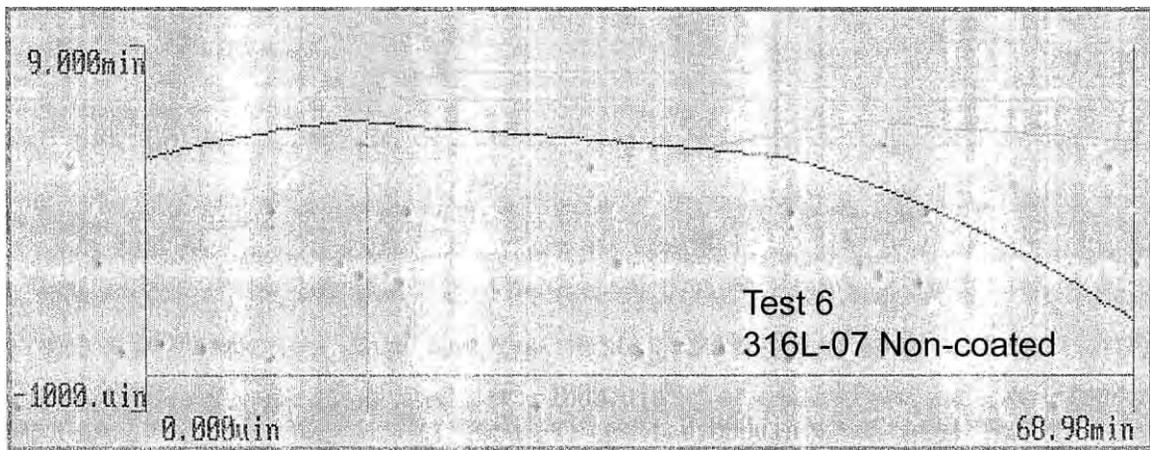
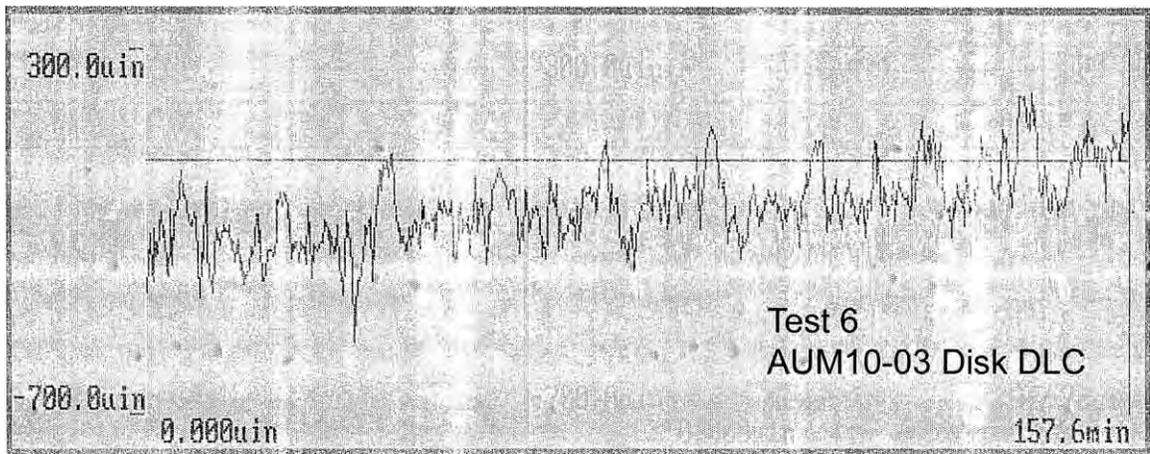


Figure 14. Post-Test Wear Surface Photographs and Profilometer Data for Test #6

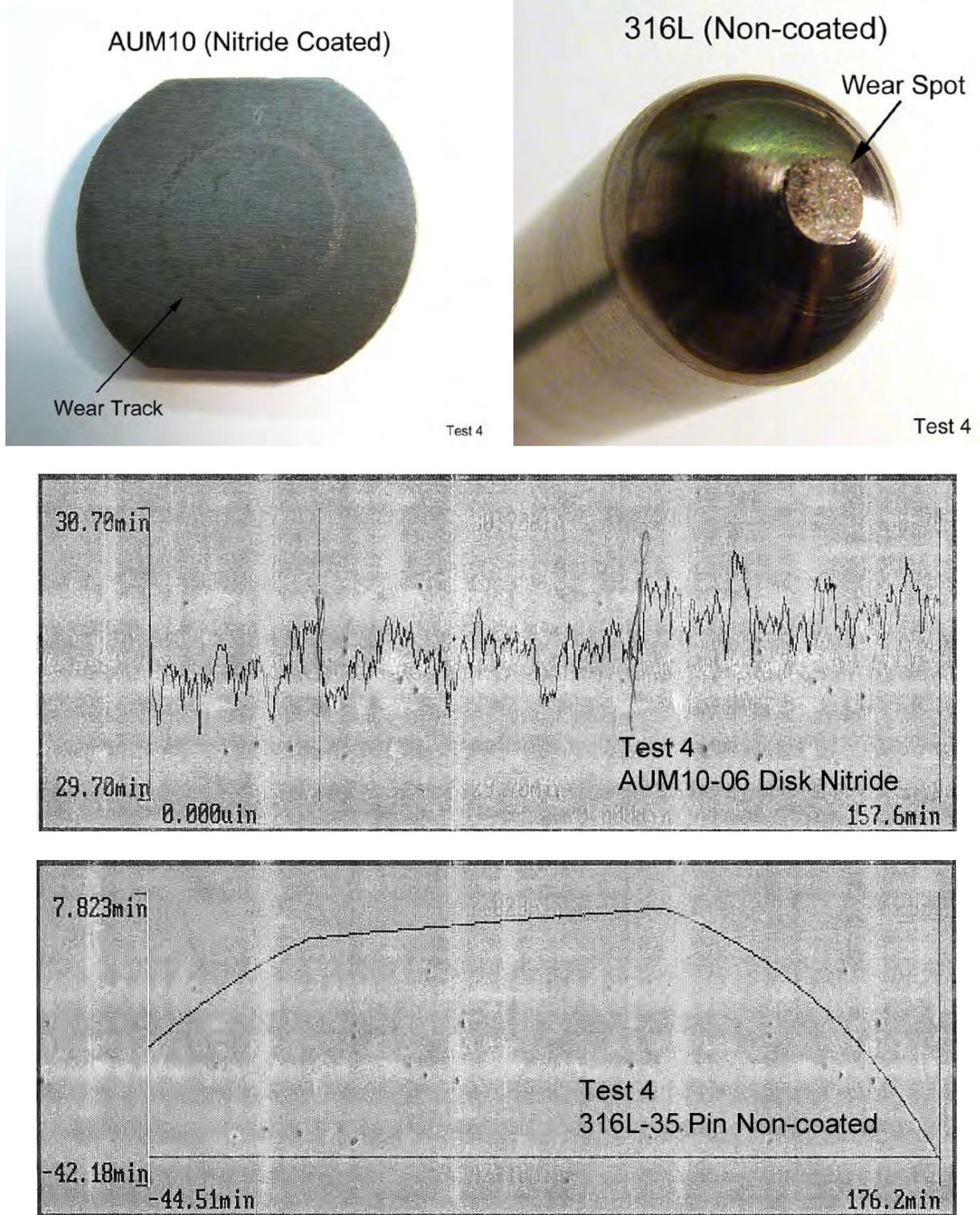


Figure 15. Post-Test Wear Surface Photographs and Profilometer Data for Test #4

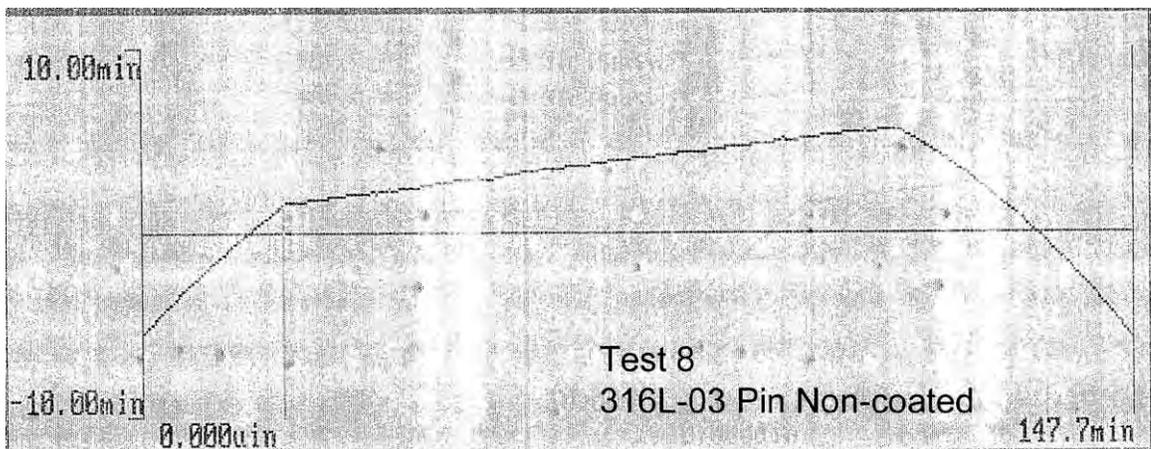
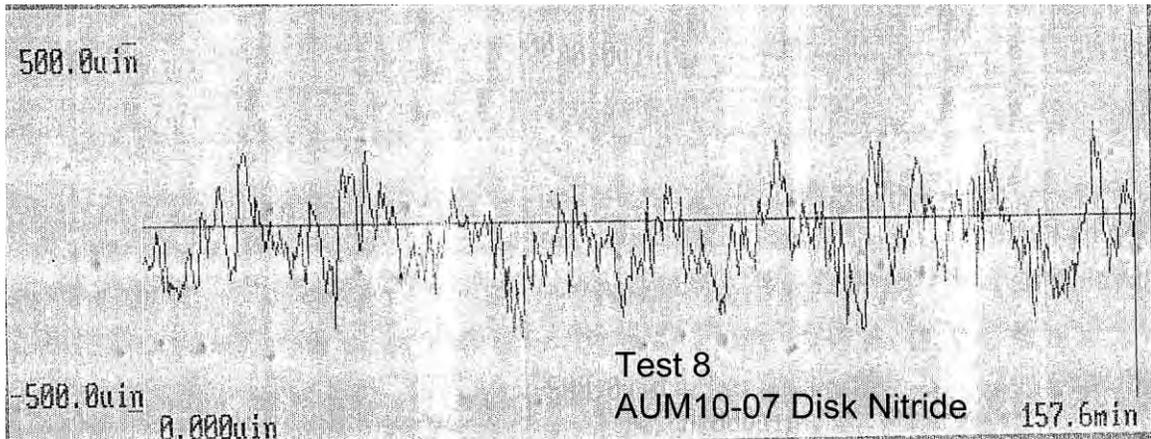
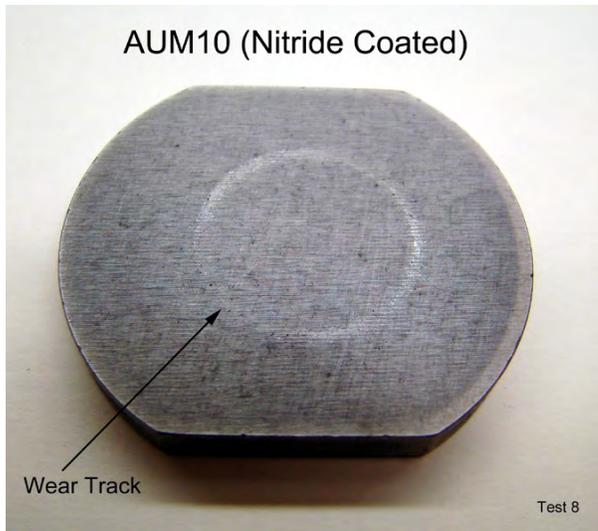


Figure 16. Post-Test Wear Surface Photographs and Profilometer Data for Test #8

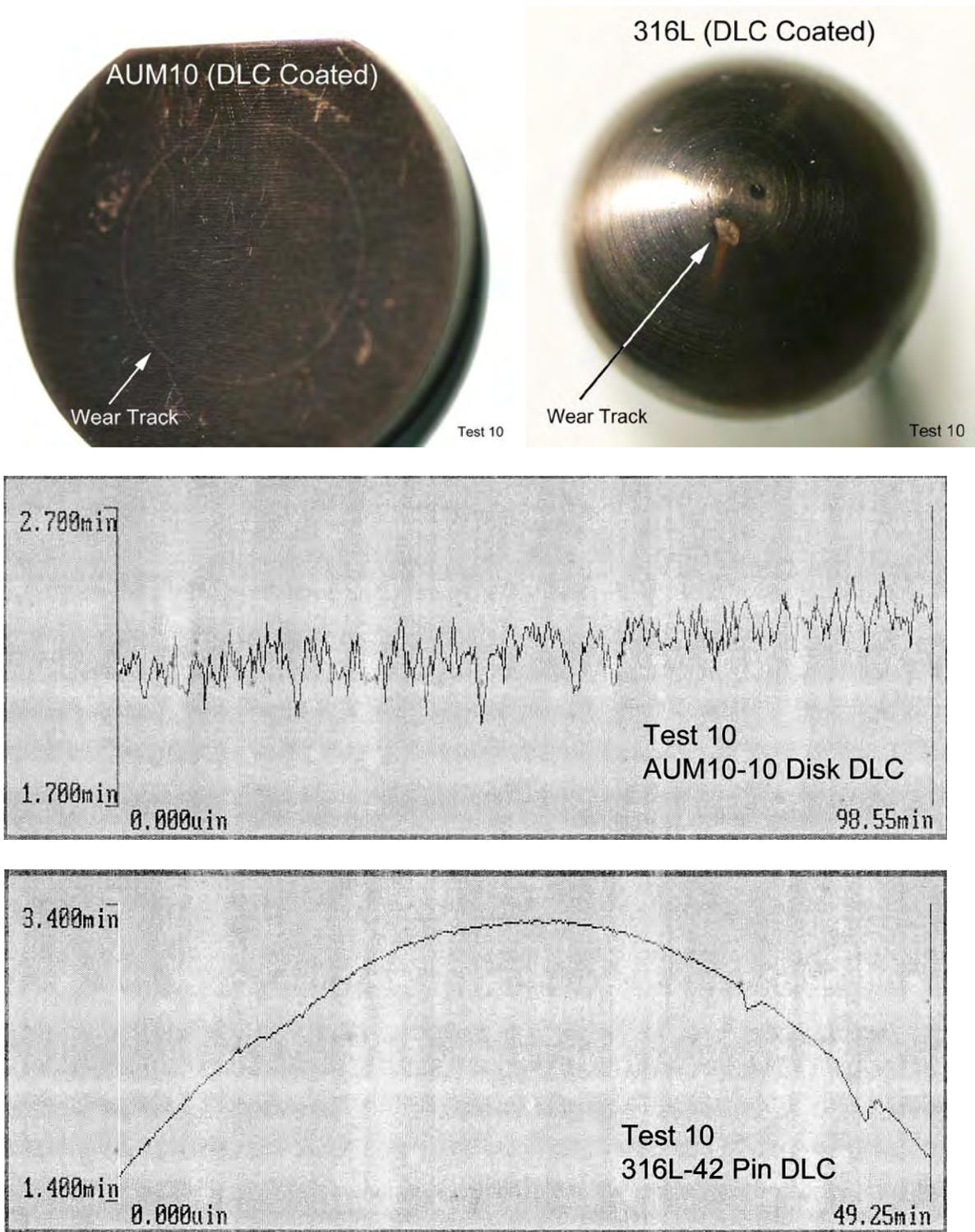


Figure 17. Post-Test Wear Surface Photographs and Profilometer Data for Test #10

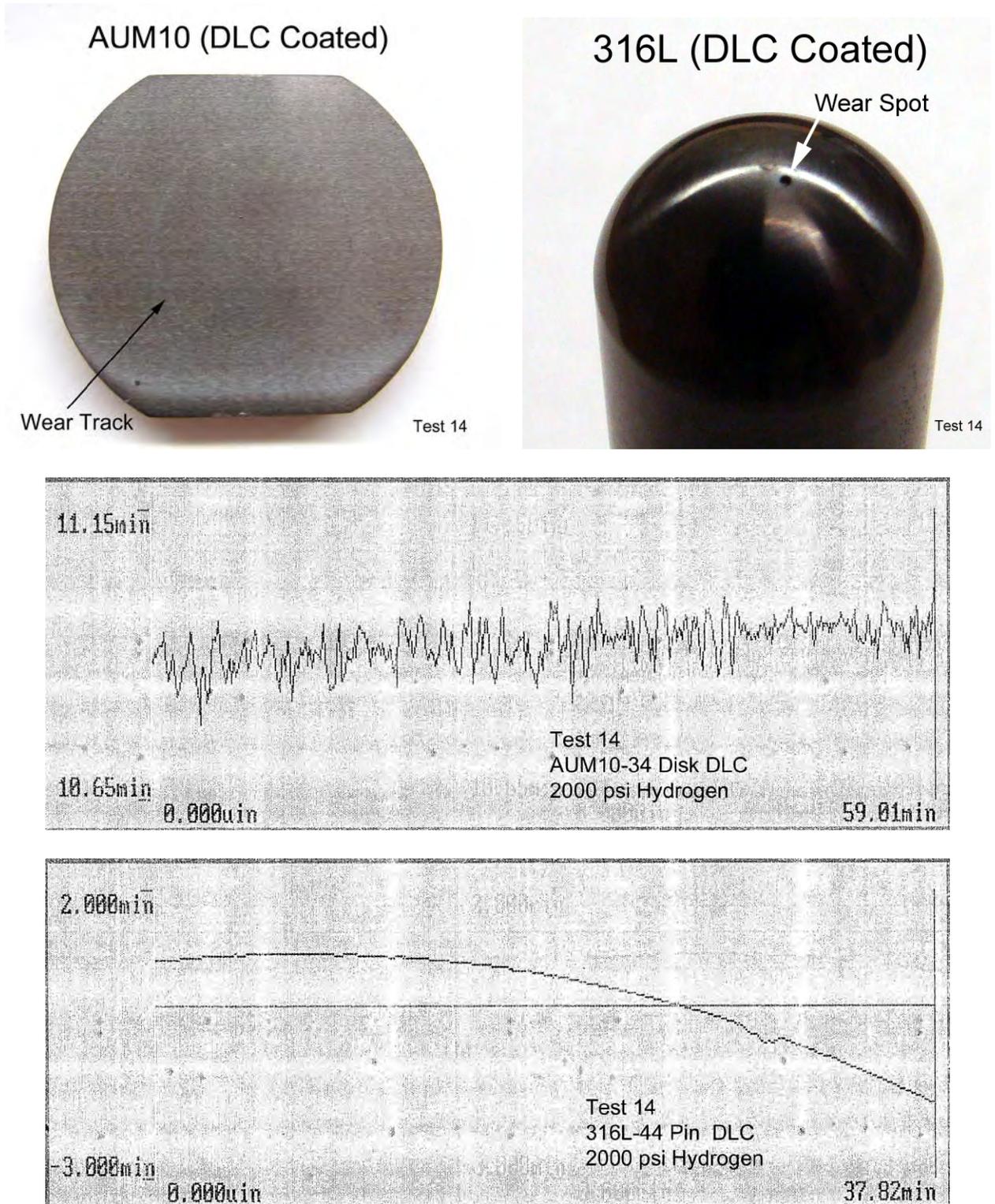


Figure 18. Post-Test Wear Surface Photographs and Profilometer Data for Test #14

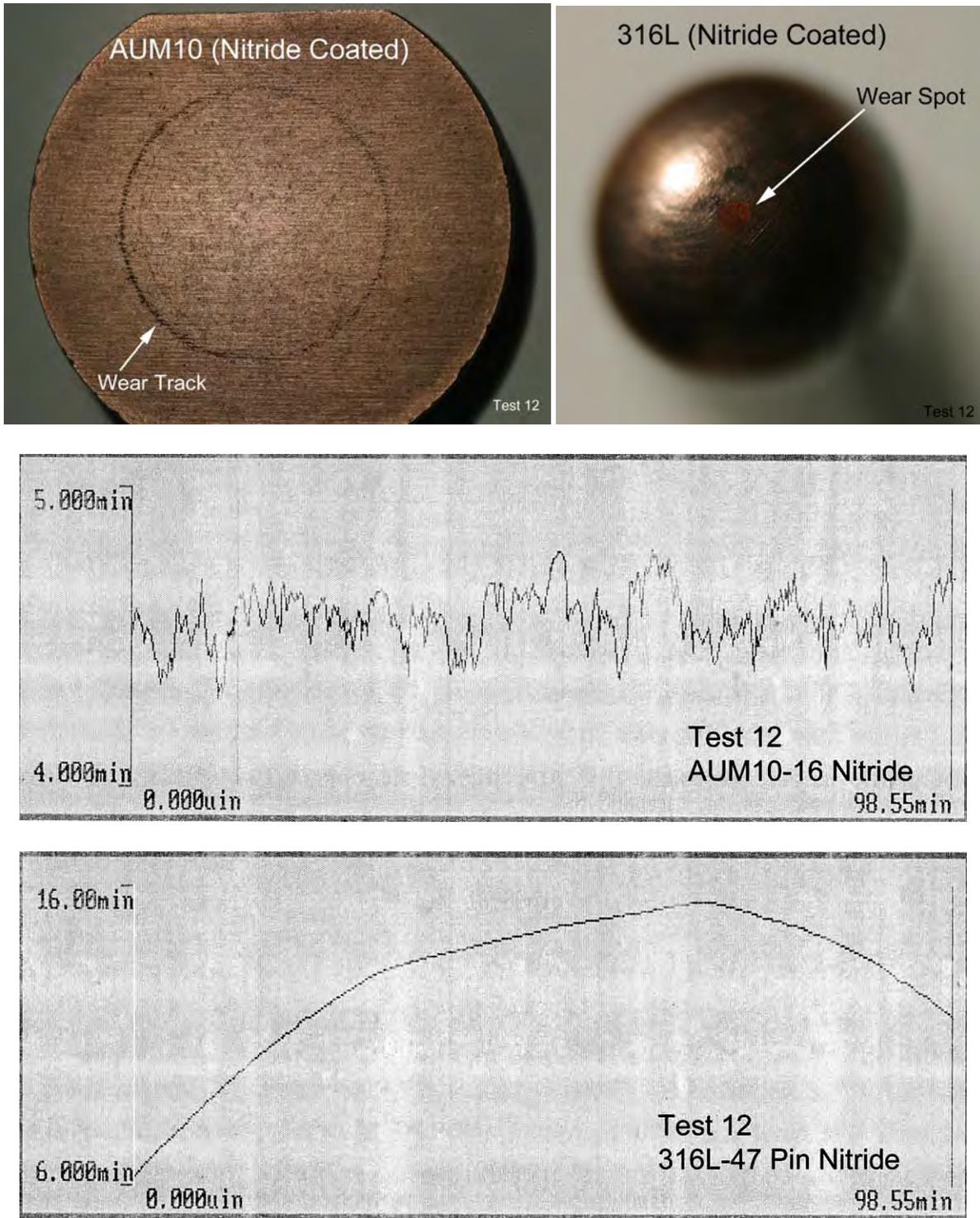


Figure 19. Post-Test Wear Surface Photographs and Profilometer Data for Test #12

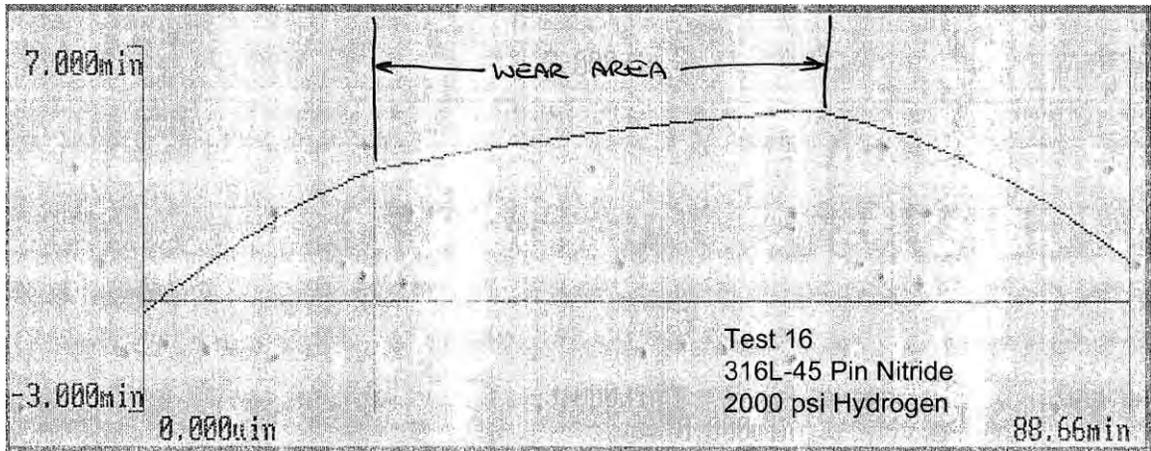
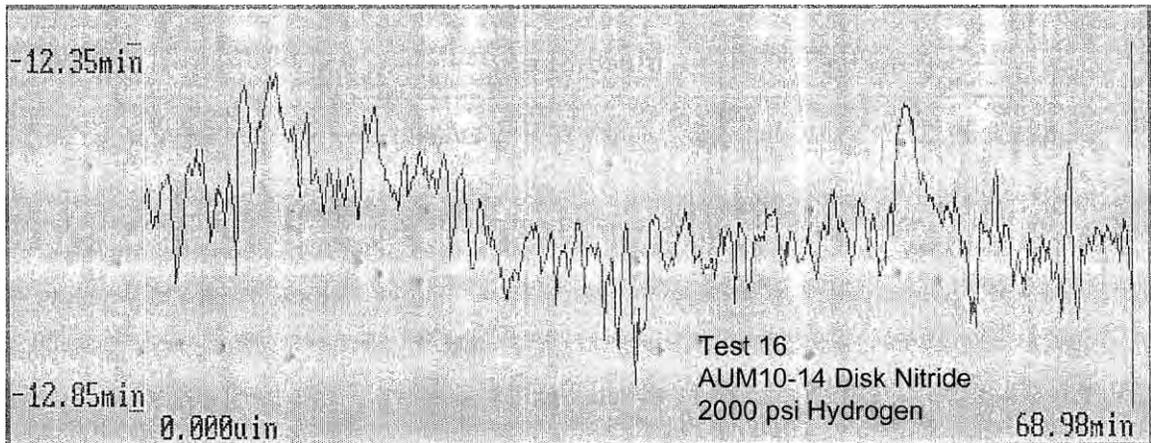
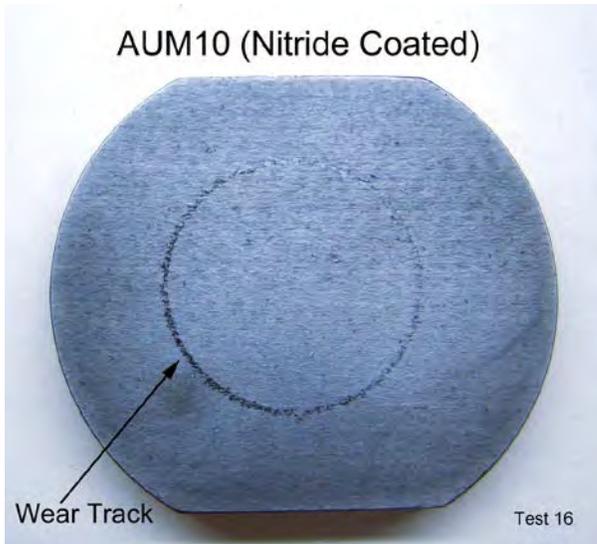


Figure 20. Post-Test Wear Surface Photographs and Profilometer Data for Test #16

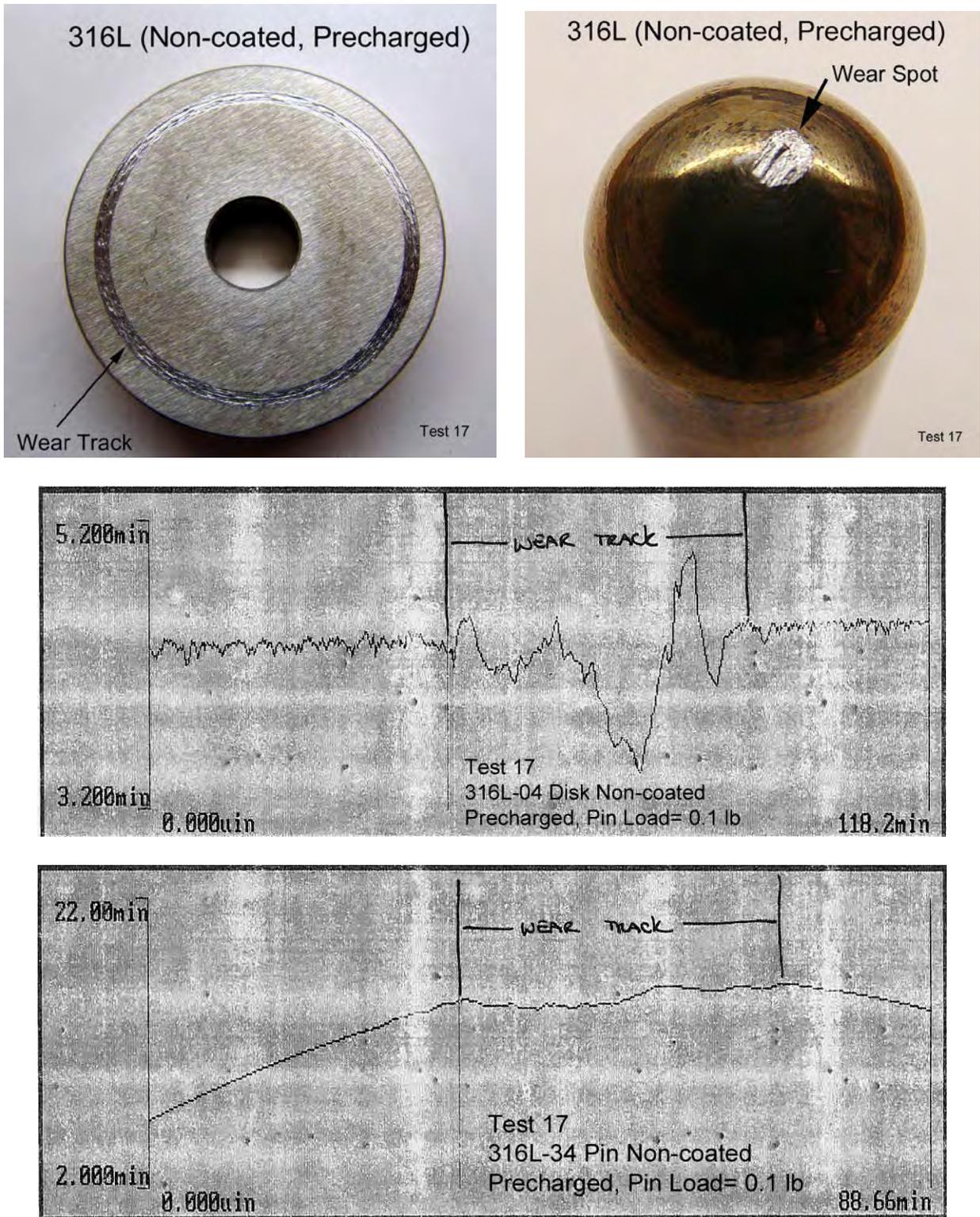


Figure 21. Post-Test Wear Surface Photographs and Profilometer Data for Test #17

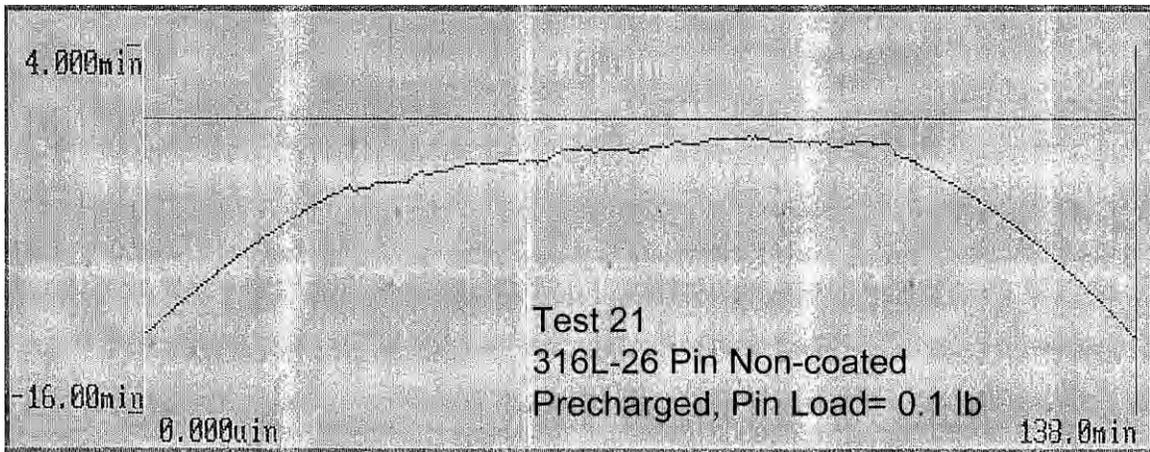
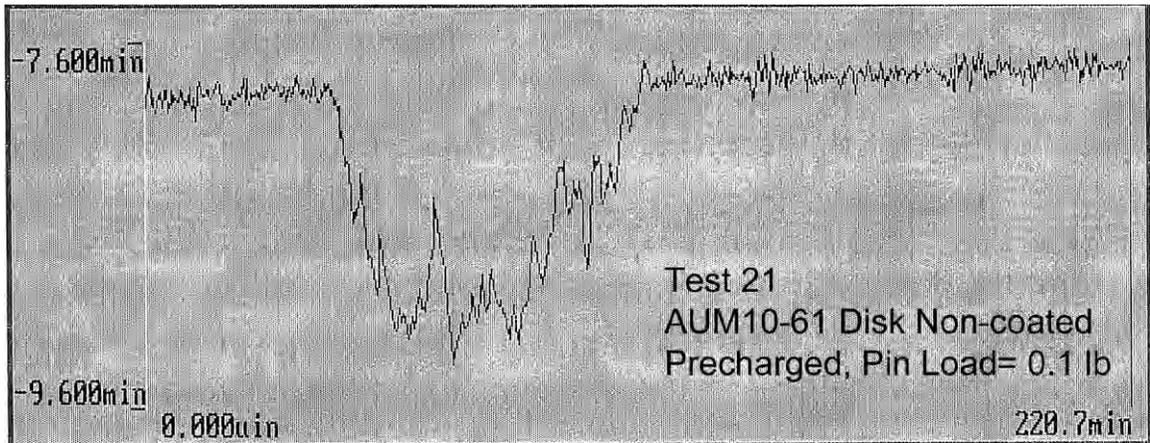
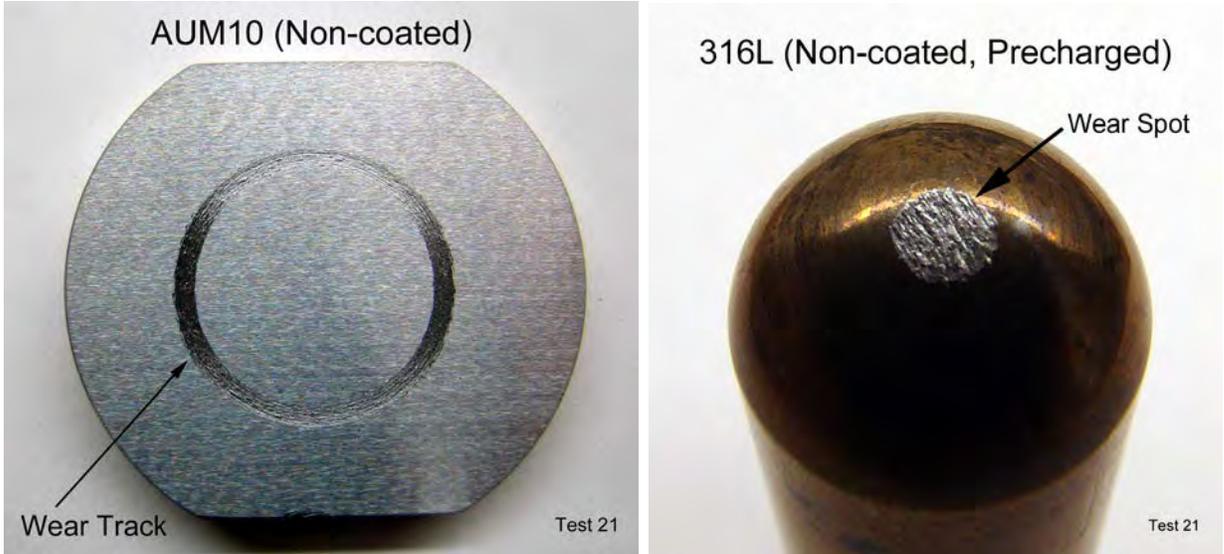


Figure 22. Post-Test Wear Surface Photographs and Profilometer Data for Test #21

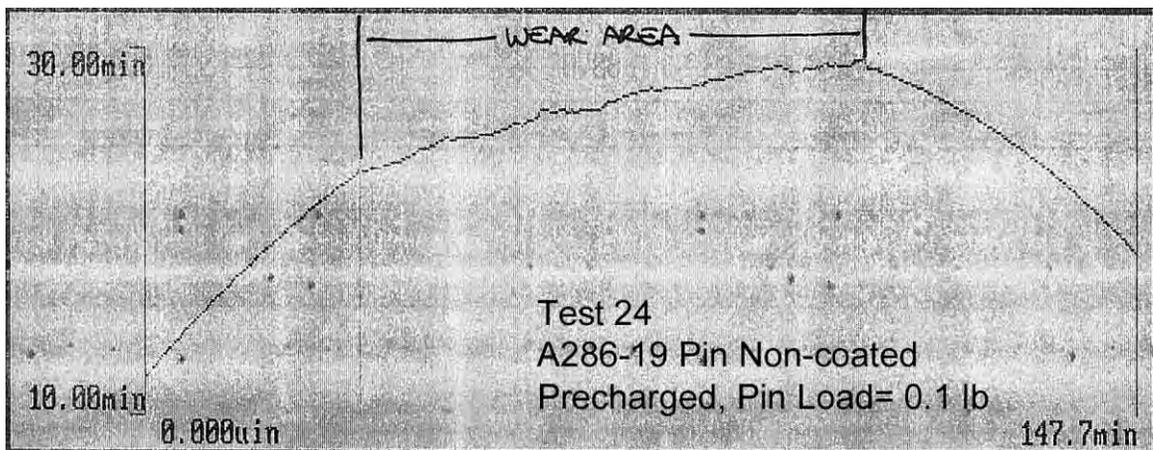
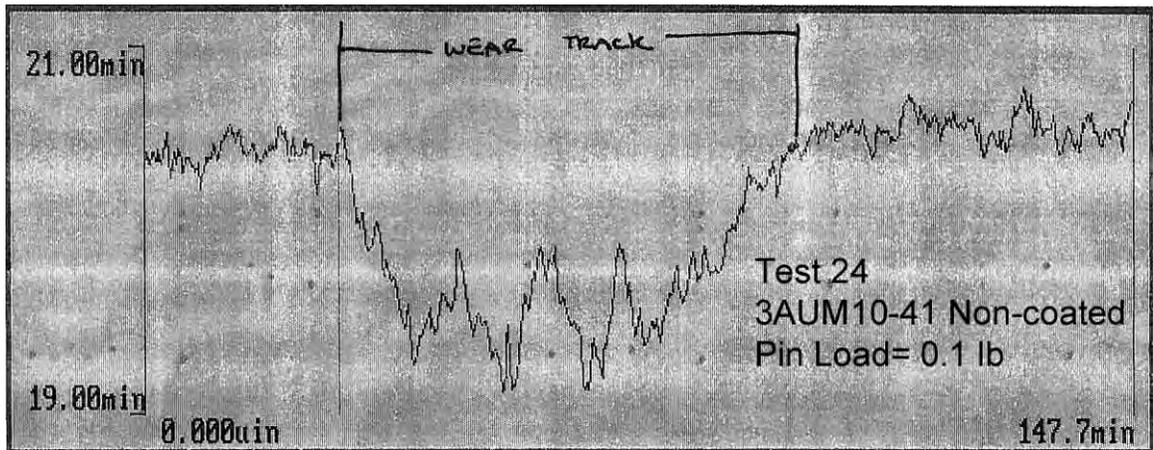
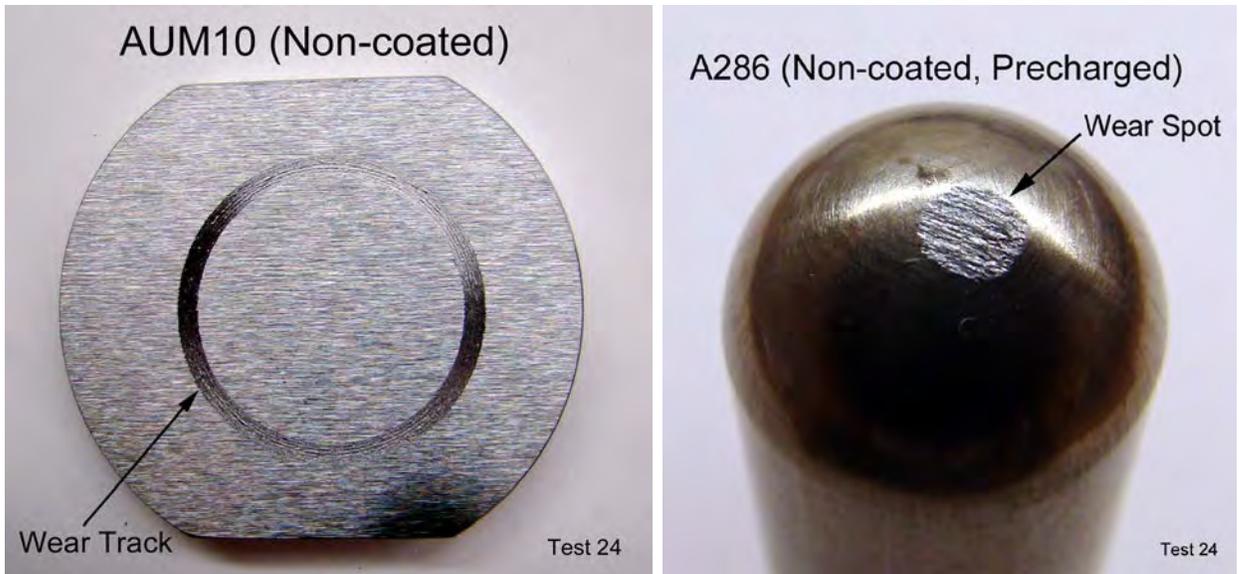


Figure 23. Post-Test Wear Surface Photographs and Profilometer Data for Test #24

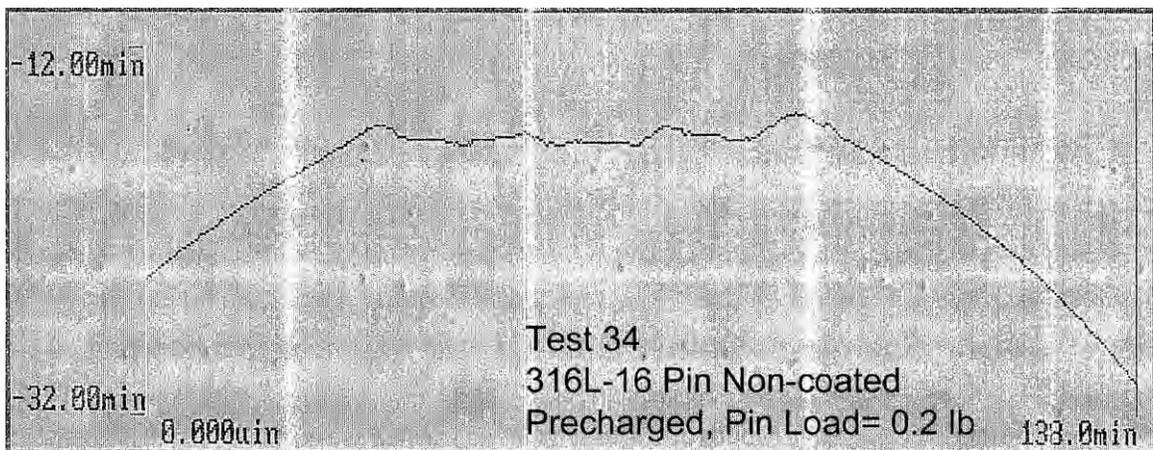
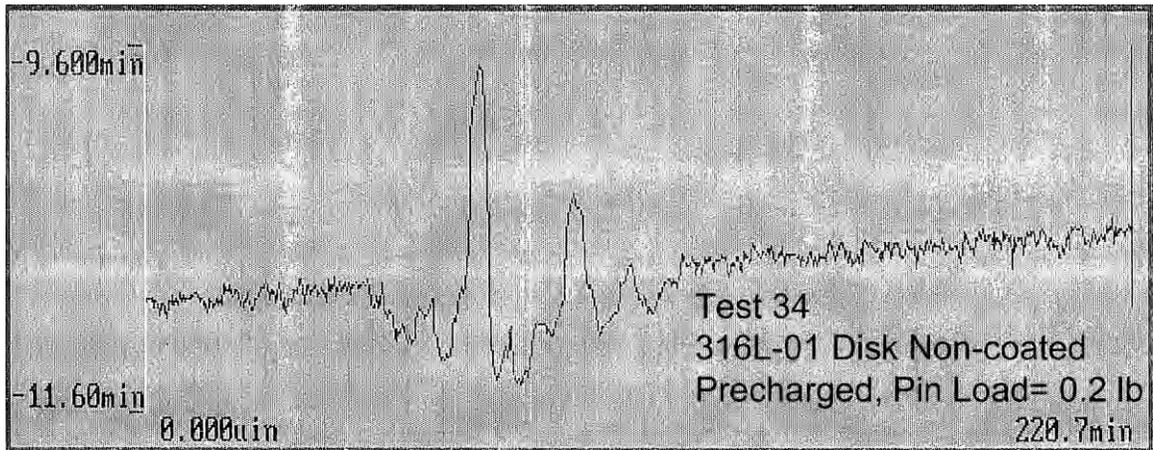


Figure 24. Post-Test Wear Surface Photographs and Profilometer Data for Test #34

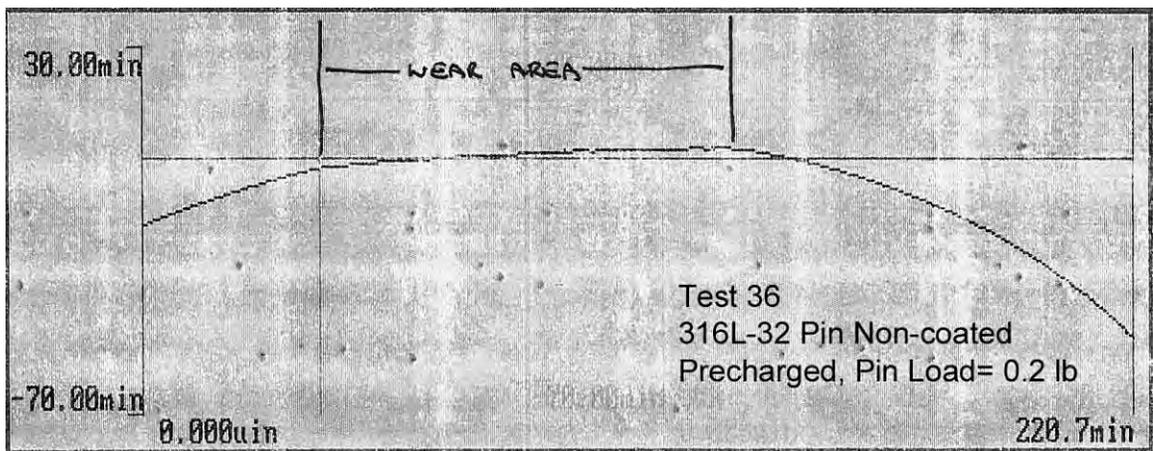
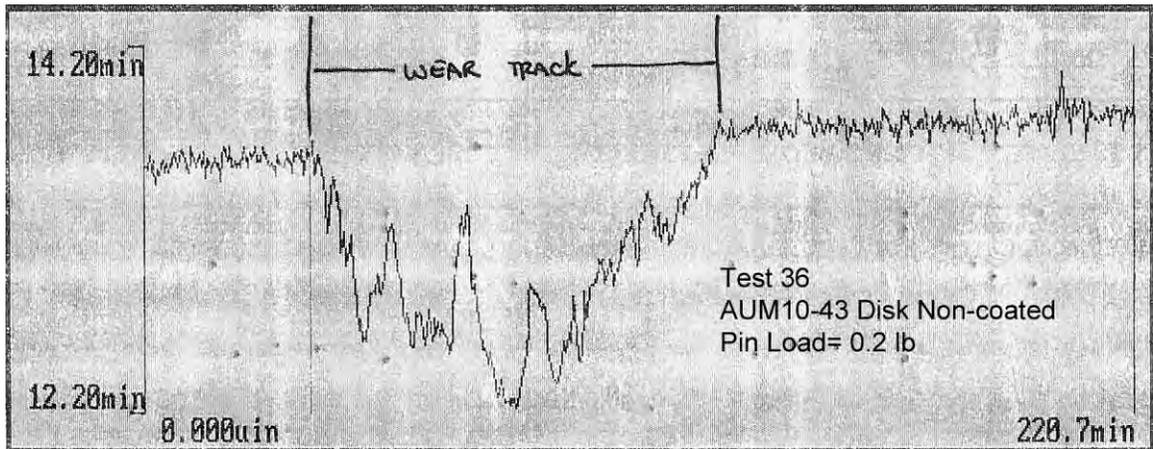
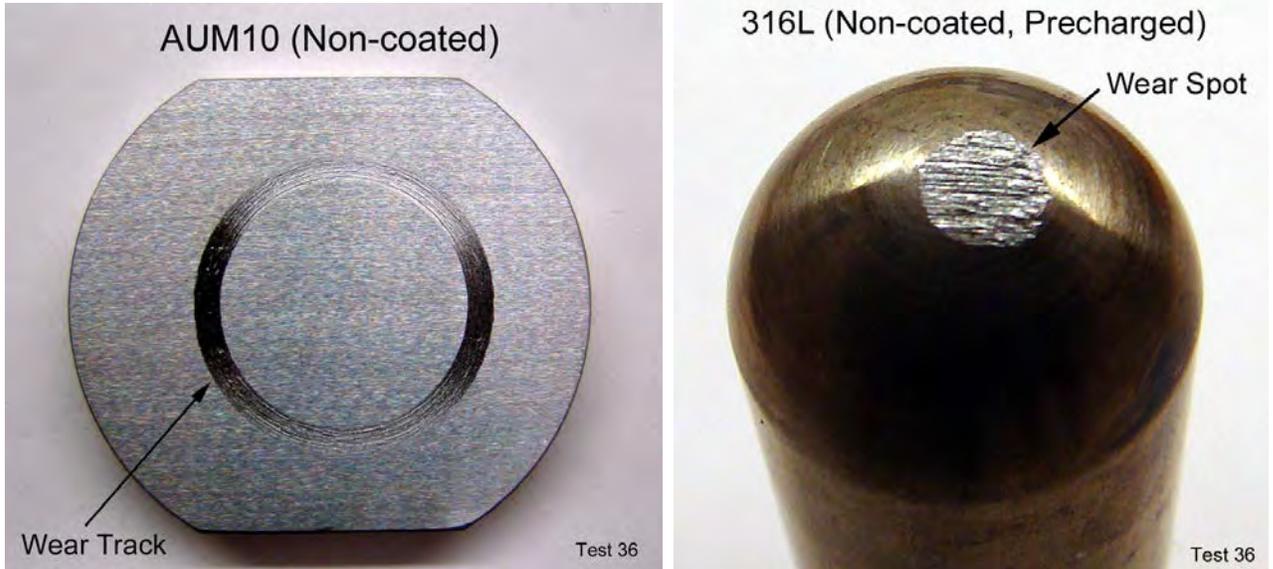


Figure 25. Post-Test Wear Surface Photographs and Profilometer Data for Test #36

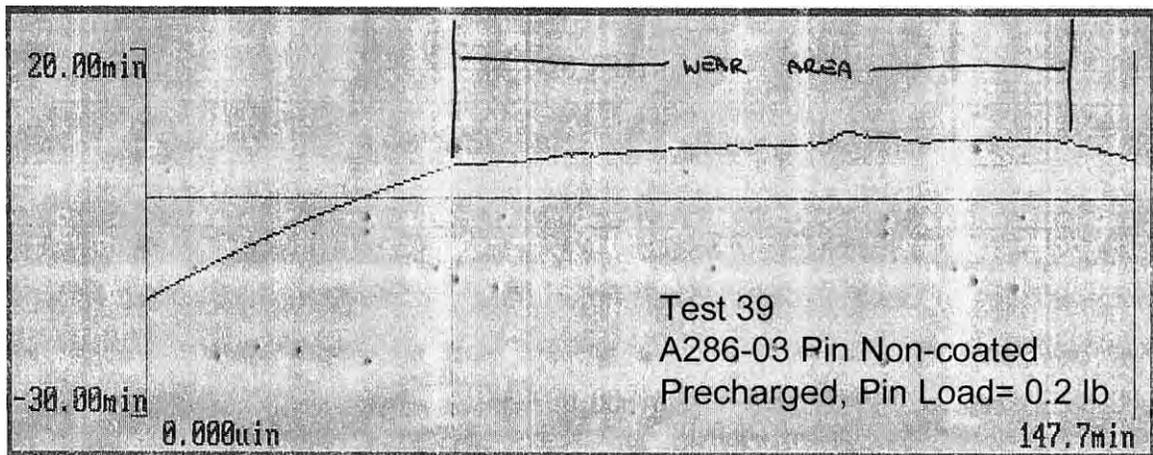
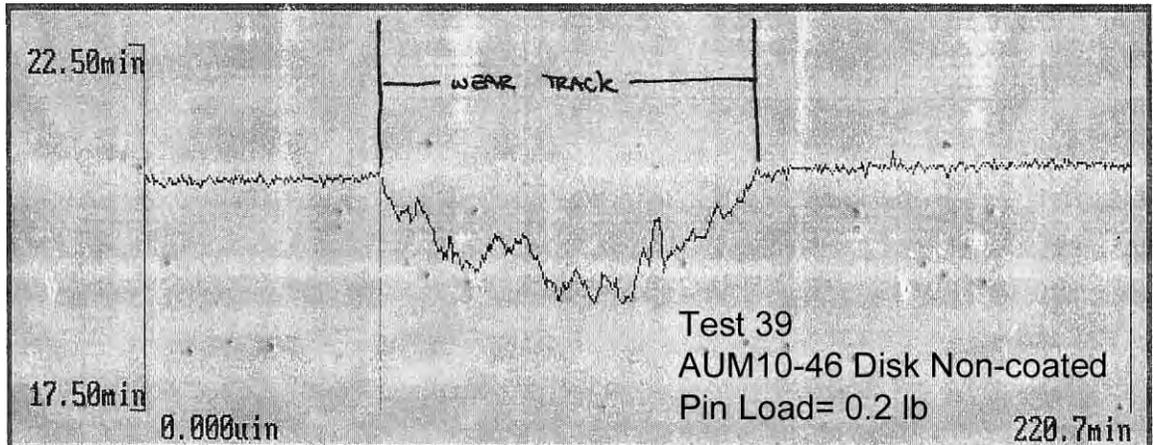
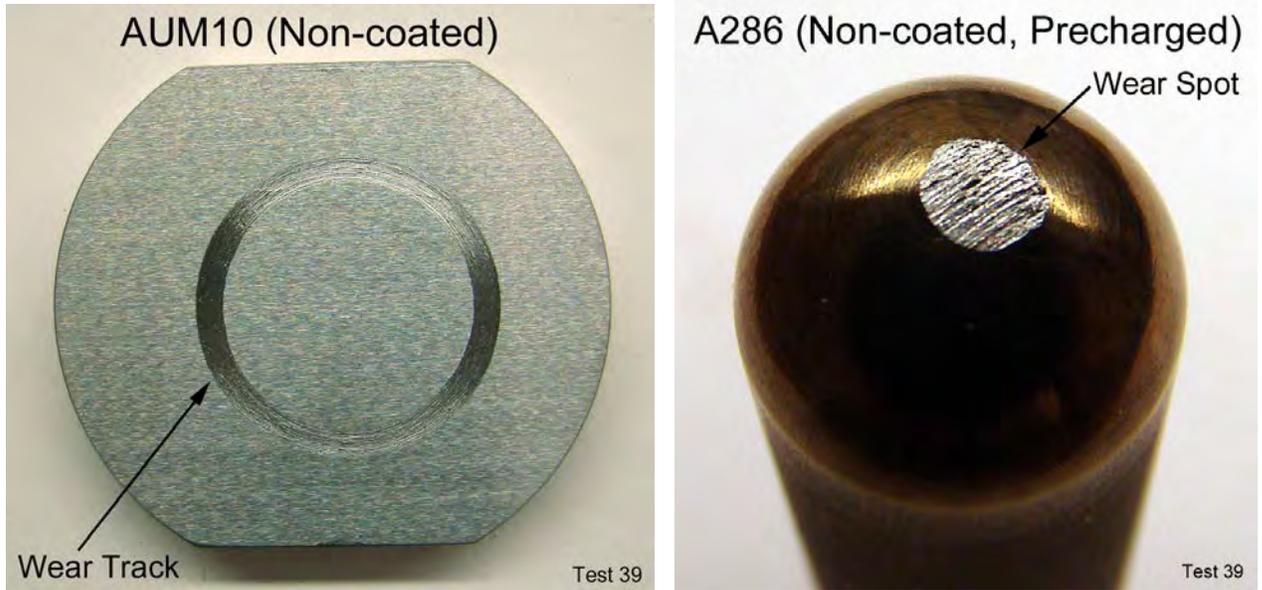


Figure 26. Post-Test Wear Surface Photographs and Profilometer Data for Test #39

8.2 SCANNING ELECTRON MICROSCOPY

Selected post-test samples were chosen for detailed characterization with scanning electron microscope (SEM). The wear tracks on nitride coated disks are similar for air and hydrogen tests when non-charged 316L pins were used (Figure 27-Figure 28). The wear spots on these pins appear to be smoother and unidirectional in the air test when Figure 29 - Figure 32 are compared. When the coated disks were tested with pre-charged 316L pins, Figure 33 shows a possible tearing in the DLC coating while the rough nitrided disk surface appears to be flattened (reduced roughness) by the pin (Figure 34). The wear patterns on the 316L pins are similar in Figure 35 and Figure 36; only the wear spot is much larger when it was tested with the nitride coated disk. Figure 37-Figure 40 show the wear details of DLC and nitride coated disks tested with pre-charged A286 pins in hydrogen. The wear track on the DLC coated disk is smoother but shows possible tearing. The nitrided disk shows that wear took place mainly on the ridges of the surface finish. The wear spot on the A286 pin in Figure 40 is not smooth but shows fracturing/chipping. It is possibly caused by the higher hardness of A286 (compared to 316L) and the inherent nature of the surface of the nitrided disk. Figure 41 and Figure 42 are the wear tracks on the DLC and nitrided coated disks and the pre-charged 316L pins with increasing load (0.2 lb). The test environment was 2000 psig hydrogen. Similar features can be seen earlier in Figure 33-Figure 34. The corresponding wear spots on pre-charged 316L pins are shown in Figure 43 and Figure 44, which can be compared with Figure 35 and Figure 36 when the pin load was 0.1 lb.

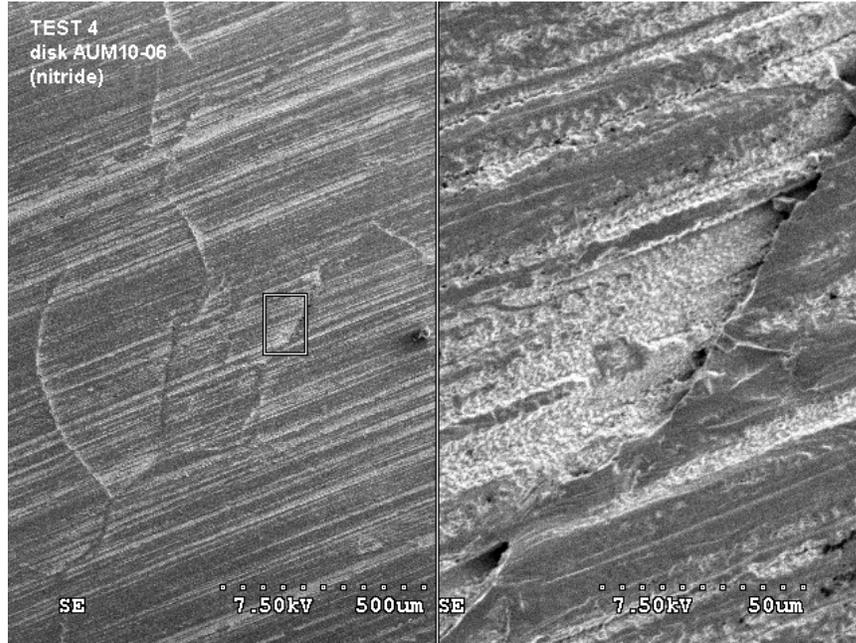


Figure 27. Wear track on nitride coated disk tested in air using non-charged 316L pin loaded with 0.1 lb.



Figure 28. Wear track on nitride coated disk tested in hydrogen using non-charged 316L pin loaded with 0.1 lb.

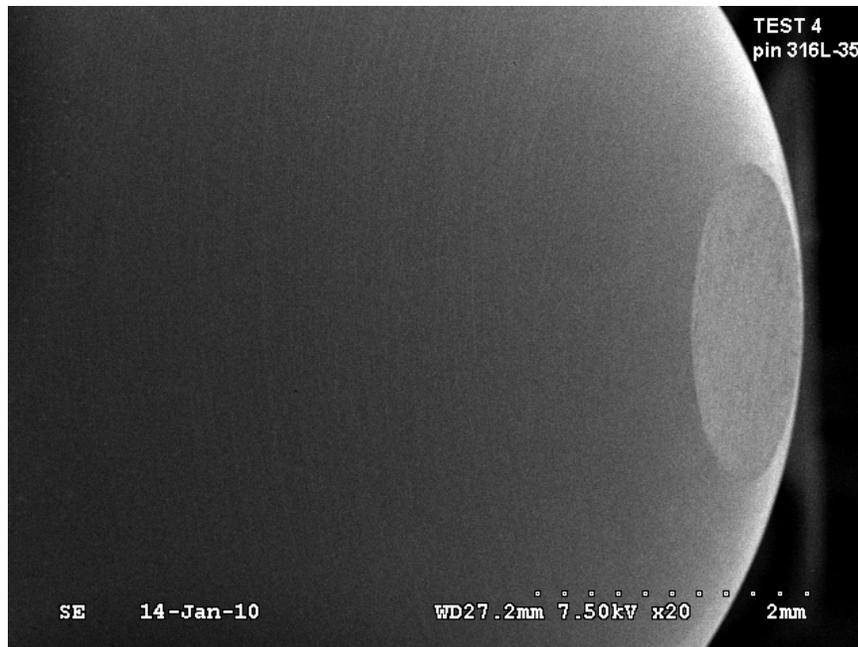


Figure 29. Wear spot on non-charged 316L pin loaded with 0.1 lb and tested in air with nitride coated disk.

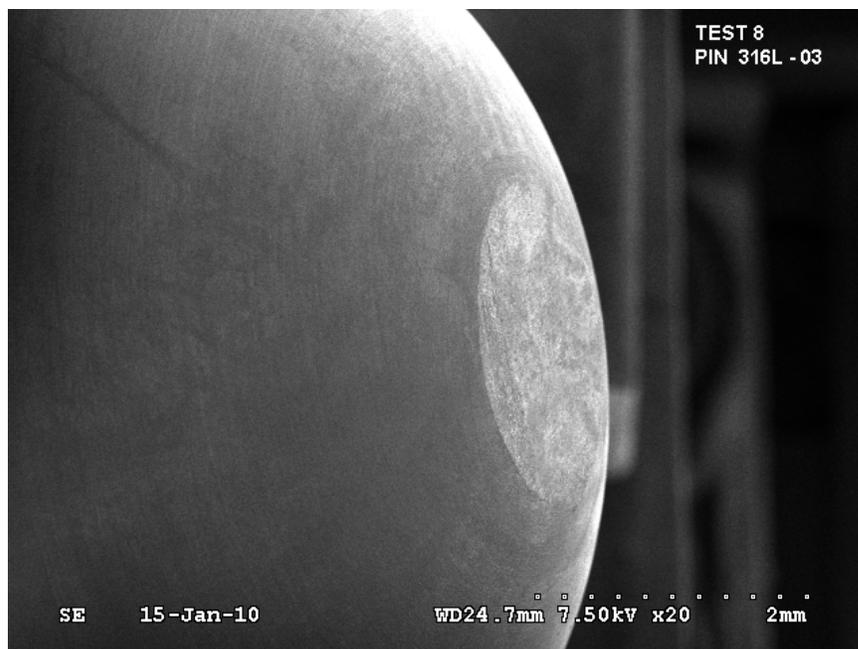


Figure 30. Wear spot on non-charged 316L pin loaded with 0.1 lb and tested in hydrogen with nitride coated disk.

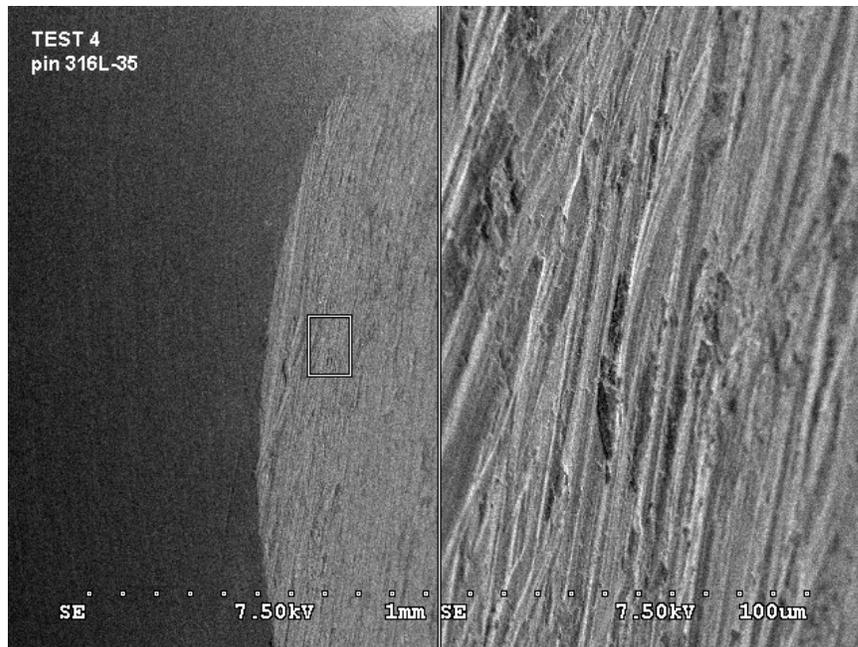


Figure 31. Wear spot details on non-charged 316L pin loaded with 0.1 lb and tested in air with nitride coated disk.

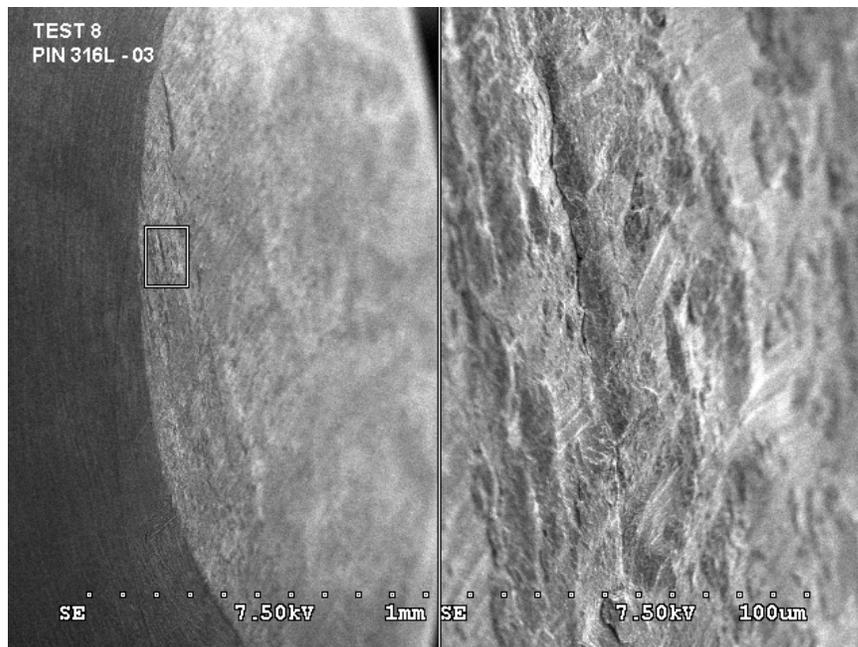


Figure 32. Wear spot details on non-charged 316L pin loaded with 0.1 lb and tested in hydrogen with nitride coated disk.

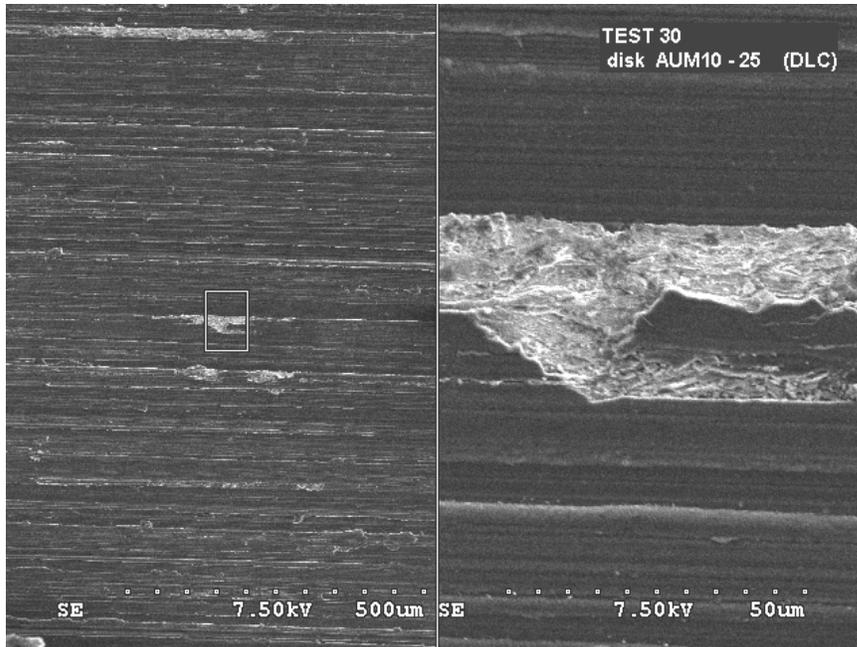


Figure 33. Wear track on DLC coated disk tested in hydrogen using pre-charged 316L pin loaded with 0.1 lb.

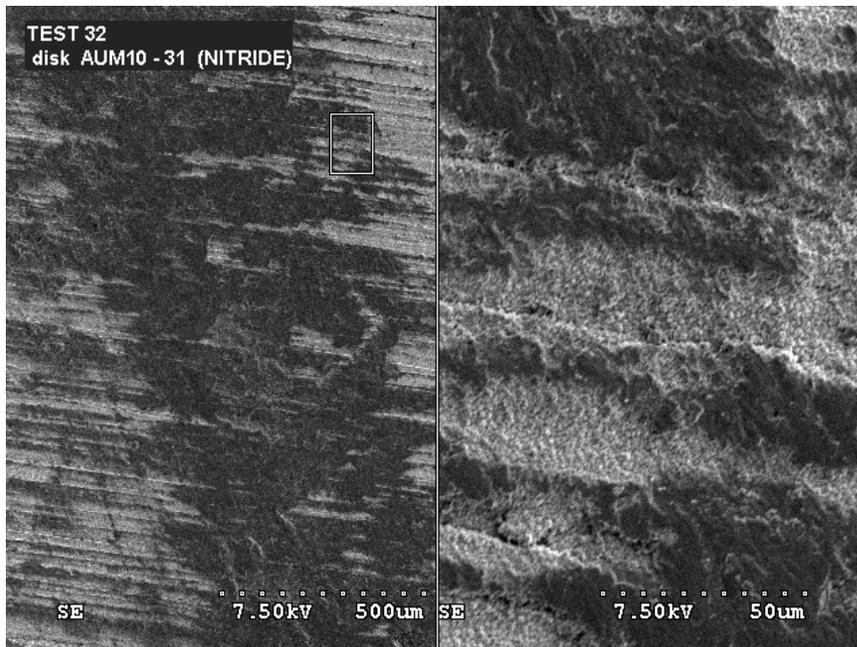


Figure 34. Wear track on nitride coated disk tested in hydrogen using pre-charged 316L pin loaded with 0.1 lb.



Figure 35. Wear spot on pre-charged 316L pin loaded with 0.1 lb and tested in hydrogen with DLC coated disk.

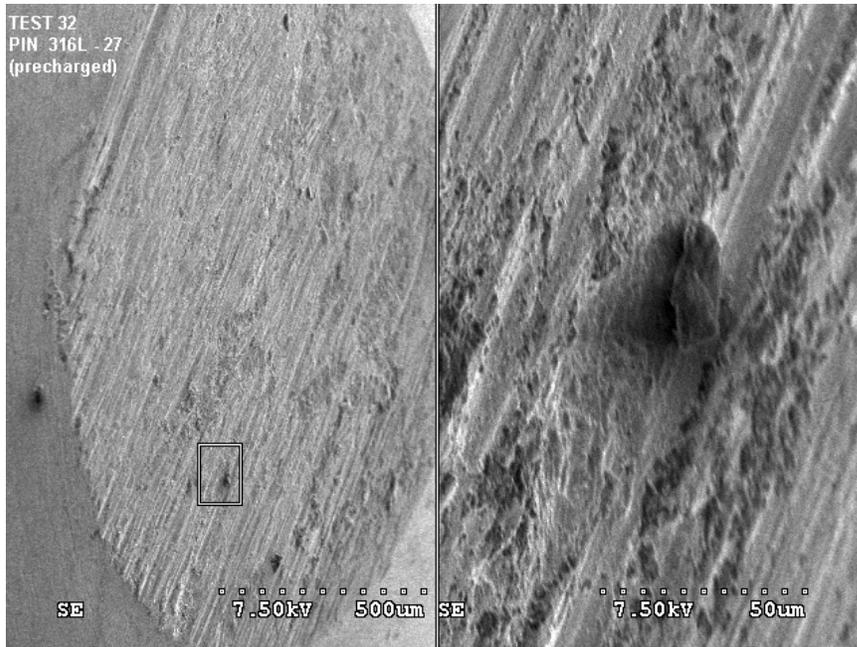


Figure 36. Wear spot on pre-charged 316L pin loaded with 0.1 lb and tested in hydrogen with nitride coated disk.

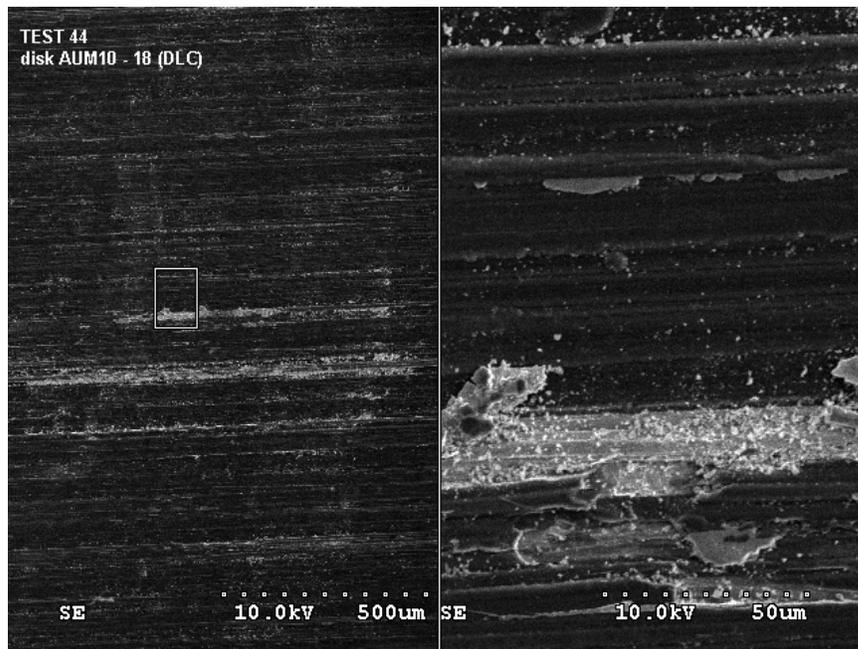


Figure 37. Wear track on DLC coated disk tested in hydrogen using pre-charged A286 pin loaded with 0.2 lb.

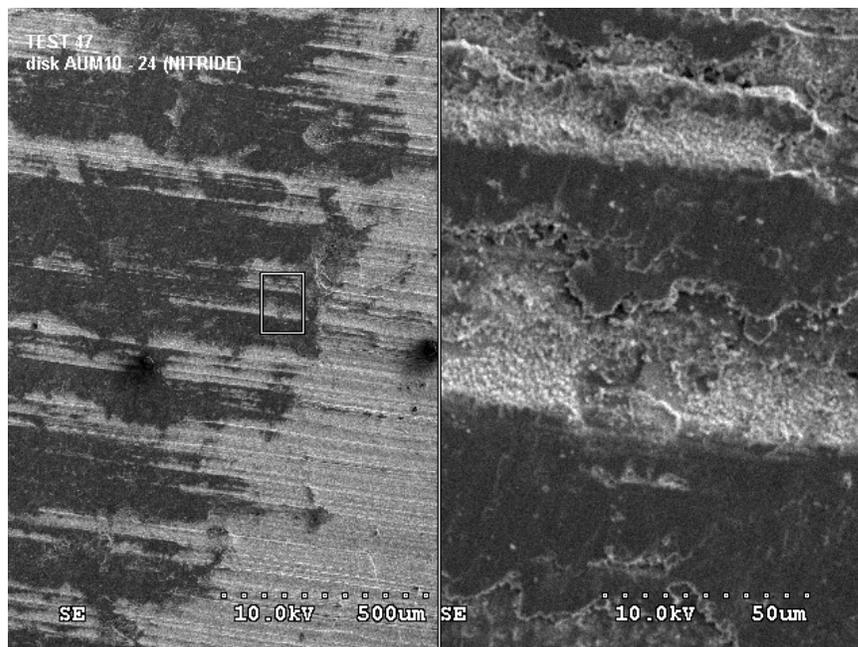


Figure 38. Wear track on Nitride coated disk tested in hydrogen using pre-charged A286 pin loaded with 0.2 lb.

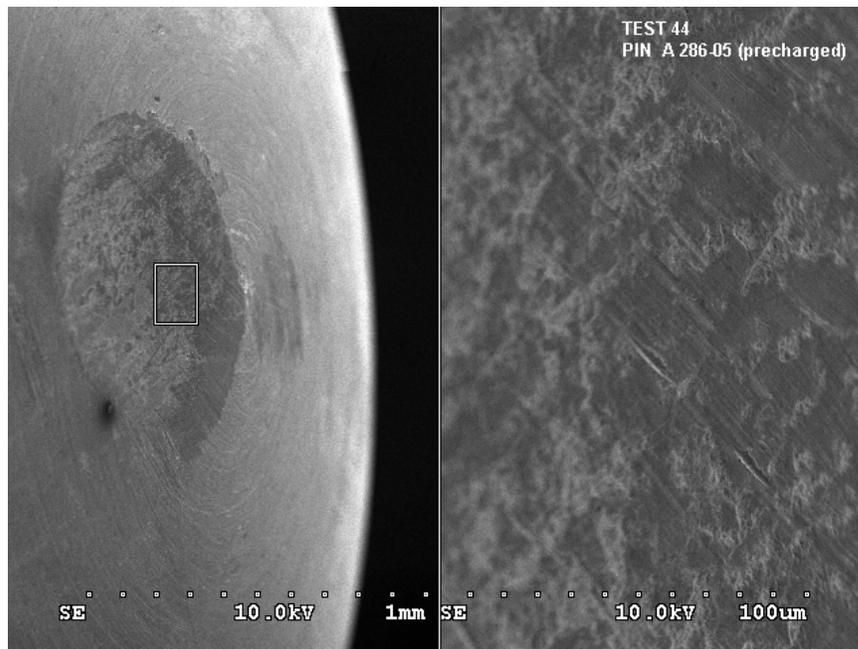


Figure 39. Wear spot on pre-charged A286 pin loaded with 0.2 lb and tested in hydrogen with DLC coated disk.



Figure 40. Wear spot on pre-charged A286 pin loaded with 0.2 lb and tested in hydrogen with nitride coated disk.

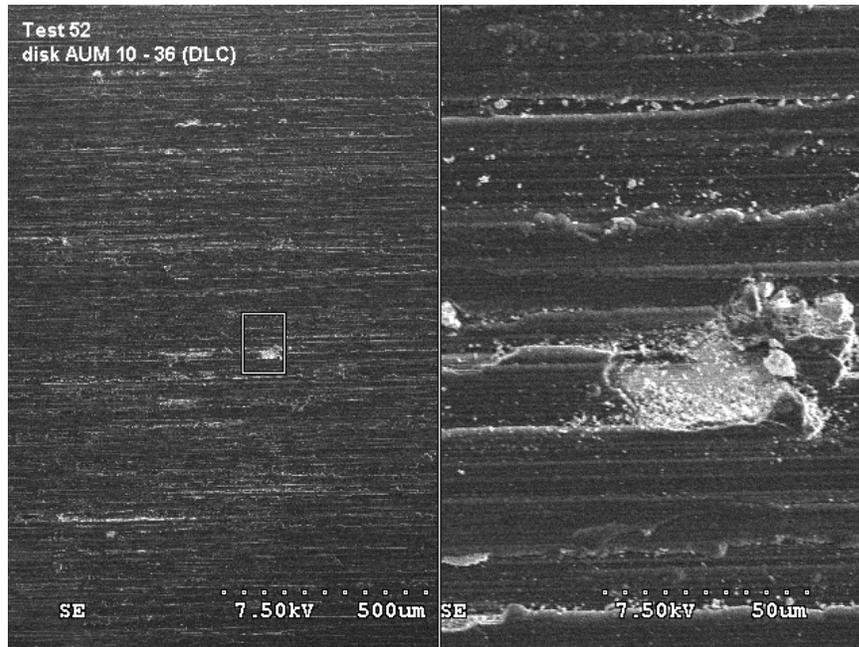


Figure 41. Wear track on DLC coated disk tested in hydrogen using pre-charged 316L pin loaded with 0.2 lb.



Figure 42. Wear track on nitride coated disk tested in hydrogen using pre-charged 316L pin loaded with 0.2 lb.

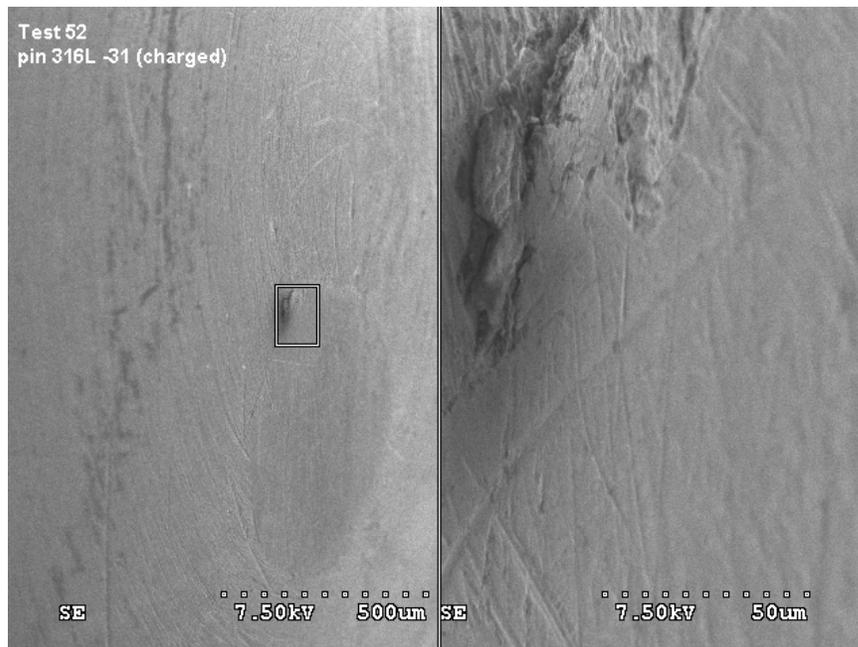


Figure 43. Wear spot on pre-charged 316L pin loaded with 0.2 lb and tested in hydrogen with DLC coated disk.

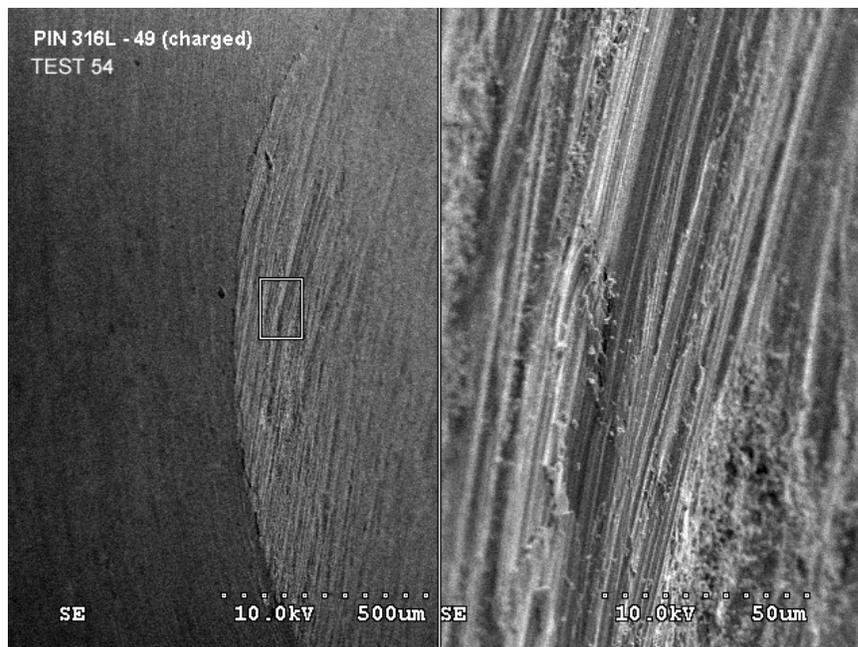


Figure 44. Wear spot on pre-charged 316L pin loaded with 0.2 lb and tested in hydrogen with nitride coated disk.

9.0 RESULTS

9.1 WEIGHT CHANGE

As discussed in a previous section, the weight change of the samples was obtained by weighing the sample with a four-place balance before and after the test. The weight changes and wear area measurements are shown in Table 5-Table 10 for the disks and for the pins. The results are shown graphically in Figure 45. From the weigh change results displayed in Figure 45 the following general conclusions can be made:

1. Wear for disk-pin combinations in which both are coated either with nitride or DLC tested in air and 2000 psig hydrogen shows minimal wear.
2. Wear for disk-pin combinations involving coated disk-non-coated pin combinations displays increased wear for the nitride coated disk cases and not the DLC, but also shows no sensitivity to environment—air vs. hydrogen.
3. For uncoated disk-pin test combination pre-charged with hydrogen and then tested in 2000 psig hydrogen, a small increase in wear is documented in comparison to similar previously tested samples in Phase I of this study
4. Samples with coated disk (nitride and DLC) tested with non-coated pre-charged pins tested at both 0.1lbs and 0.2 lbs show no significant difference between air and hydrogen environments and sample loading for tested disk-pin combinations.

Table 5. Test Results for Non-charged Samples (Disks)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Track Finish (μ -inch) | Inside Track Finish (μ -inch) | Averaged Track Diameter (inch) | Track width (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) |
|----------|------------------|-----------------|-----------------------|-------------------------------------|------------------------------------|--------------------------------|--------------------|--------------------|---|
| 1 | DLC AUM10 | 316L | Air | 82.4 | 82.4 | 1.0175 | 0.0355 | - 0.00040 | -0.11428 |
| 2 | DLC AUM10 | 316L | Air | 57.5 | 57.5 | 1.0965 | 0.0355 | 0 | 0 |
| 3 | Nitride AUM10 | 316L | Air | 33.5 | 29.5 | 0.974 | 0.078 | 0.00072 | 0.10285 |
| 4 | Nitride AUM10 | 316L | Air | 58.9 | 55.3 | 0.941 | 0.074 | 0.00222 | 0.31714 |
| 5 | DLC AUM10 | 316L | Hydrogen 2000 psig | not measured | not measured | not measured | not measured | 0.00030 | 0.08571 |
| 6 | DLC AUM10 | 316L | Hydrogen 2000 psig | 58.9 | not measured. | not measured | not measured | 0.00018 | 0.05142 |
| 7 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 64 | 73.4 | 0.862 | 0.058 | - 0.00010 | -0.01428 |
| 8 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 86 | 67.4 | 0.8425 | 0.0335 | - 0.00006 | -0.00857 |
| 9 | DLC AUM10 | DLC 316L | Air | 55.5 | not measured. | 1.0845 | 0.0135 | - 0.00130 | -0.37142 |
| 10 | DLC AUM10 | DLC 316L | Air | 43.8 | not measured | 1.008 | 0.01 | 0.00070 | 0.2 |
| 11 | Nitride AUM10 | Nitride 316L | Air | 49.5 | not measured | 1.022 | 0.023 | 0.00076 | 0.10858 |
| 12 | Nitride AUM10 | Nitride 316L | Air | 47.8 | not measured | 1.0275 | 0.0215 | 0.00046 | 0.06571 |
| 13 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | 32 | 25 | 1.1425 | 0.0175 | 0.00026 | 0.07428 |
| 14 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | 23 | 23 | 1.08 | 0.015 | - 0.00038 | -0.10857 |
| 15 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 39 | 23 | 1.0535 | 0.0245 | 0.00068 | 0.09714 |
| 16 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 32 | 32 | 0.9255 | 0.0275 | 0.00102 | 0.14571 |

Note: The negative value under Weight Loss or Volume Loss indicates that the sample gained weight or volume after testing. This may be caused by material transfer or measurement imprecision.

Table 6. Test Results for Non-charged Samples (Pins)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Wear Finish (μ -inch) | Inside Wear Finish (μ -inch) | Averaged Wear Spot Dia. (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) | Calculated Volume Loss (mm^3) |
|----------|------------------|-----------------|-----------------------|------------------------------------|-----------------------------------|--------------------------------|--------------------|---|--|
| 1 | DLC AUM10 | 316L | Air | 8.75 | 3.96 | 0.04 | 0.00004 | 0.005013 | 0.01102 |
| 2 | DLC AUM10 | 316L | Air | 5.4 | 8.6 | 0.035 | -0.00090 | -0.11278 | 0.00646 |
| 3 | Nitride AUM10 | 316L | Air | 3.4 | 17.6 | 0.104 | 0.00390 | 0.48872 | 0.51534 |
| 4 | Nitride AUM10 | 316L | Air | 4.44 | 21.38 | 0.0985 | 0.00266 | 0.33333 | 0.41351 |
| 5 | DLC AUM10 | 316L | Hydrogen 2000 psig | 5.89 | 5.12 | 0.03 | 0.00000 | 0 | 0.00348 |
| 6 | DLC AUM10 | 316L | Hydrogen 2000 psig | 9.8 | 4.3 | 0.0325 | -0.00004 | -0.00501 | 0.00480 |
| 7 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 2.3 | 28.2 | 0.084 | 0.00166 | 0.20802 | 0.21727 |
| 8 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 2.5 | 17.9 | 0.0925 | 0.00278 | 0.34837 | 0.32068 |
| 9 | DLC AUM10 | DLC 316L | Air | 9.38 | not measured | 0.0155 | -0.00160 | -0.45714 | 0.00025 |
| 10 | DLC AUM10 | DLC 316L | Air | 10.6 | 3.7 | 0.0165 | 0.00020 | 0.057143 | 0.00032 |
| 11 | Nitride AUM10 | Nitride 316L | Air | 11.7 | 2.9 | 0.041 | 0.00006 | 0.00857 | 0.01217 |
| 12 | Nitride AUM10 | Nitride 316L | Air | 12 | 6 | 0.0361 | 0.00002 | 0.00286 | 0.00730 |
| 13 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | 5.7 | not measured | 0.015 | -0.00002 | -0.00571 | 0.00022 |
| 14 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | 6 | 6 | 0.013 | 0.00008 | 0.02286 | 0.00012 |
| 15 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 8 | 7 | 0.037 | 0.00098 | 0.14 | 0.00807 |
| 16 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 11.5 | 3.8 | 0.0415 | -0.00020 | -0.02857 | 0.01278 |

Table 7. Test Results for Pre-charged Samples (Disks)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Track Finish (μ -inch) | Inside Track Finish (μ -inch) | Averaged Track Diameter (inch) | Track width (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) |
|----------|---------------|--------------|--------------------|-------------------------------------|------------------------------------|--------------------------------|--------------------|--------------------|---|
| 17 | 316L | 316L | Hydrogen 2000 psig | 20 | 59 | 0.962 | 0.054 | 0.00314 | 0.39348 |
| 18 | 316L | 316L | Hydrogen 2000 psig | 19.7 | 180 | 0.8435 | 0.0635 | 0.00296 | 0.37092 |
| 19 | 316L | 316L | Hydrogen 2000 psig | 22 | 90 | 1.0205 | 0.0675 | 0.00334 | 0.41854 |
| 20 | AUM10 | 316L | Hydrogen 2000 psig | 26.2 | 39.8 | 0.8385 | 0.0175 | 0.00182 | 0.22807 |
| 21 | AUM10 | 316L | Hydrogen 2000 psig | 31 | 131.1 | 0.9105 | 0.0695 | 0.02058 | 2.57895 |
| 22 | AUM10 | 316L | Hydrogen 2000 psig | 21 | 132 | 1.032 | 0.103 | 0.03600 | 4.51128 |
| 23 | AUM10 | A286 | Hydrogen 2000 psig | 21 | 49 | 1.0195 | 0.0295 | 0.00010 | 0.01253 |
| 24 | AUM10 | A286 | Hydrogen 2000 psig | 44 | 76 | 1.0165 | 0.0745 | 0.01830 | 2.29323 |
| 25 | AUM10 | A286 | Hydrogen 2000 psig | 29.4 | 138.3 | 0.9955 | 0.0745 | 0.00028 | 0.03508 |
| 26 | DLC AUM10 | 316L | Air | not measured | not measured | not measured | not measured | -0.00004 | -0.01143 |
| 27 | DLC AUM10 | 316L | Air | not measured | not measured | not measured | not measured | 0.00014 | 0.04 |
| 28 | Nitride AUM10 | 316L | Air | 32.2 | 21.7 | 0.9985 | 0.0505 | 0.00092 | 0.13143 |
| 29 | Nitride AUM10 | 316L | Air | 76.3 | 60.9 | 1.023 | 0.06 | 0.00016 | 0.02286 |
| 30 | DLC AUM10 | 316L | Hydrogen 2000 psig | 43 | 43 | 1.003 | 0.028 | -0.00004 | -0.01143 |
| 31 | DLC AUM10 | 316L | Hydrogen 2000 psig | 65 | 65 | 0.9745 | 0.1565 | -0.00004 | -0.01143 |
| 32 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 31 | 23 | 0.9775 | 0.0595 | 0.00082 | 0.11714 |
| 33 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 37 | 15 | 0.9435 | 0.0515 | 0.00024 | 0.03429 |

Table 8. Test Results for Pre-charged Samples (Pins)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Wear Finish (μ -inch) | Inside Wear Finish (μ -inch) | Averaged Wear Spot Dia. (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) | Calculated Volume Loss (mm^3) |
|----------|------------------|--------------|--------------------|------------------------------------|-----------------------------------|--------------------------------|--------------------|---|--|
| 17 | 316L | 316L | Hydrogen 2000 psig | 4 | 71 | 0.0575 | 0.00002 | 0.002506 | 0.04727 |
| 18 | 316L | 316L | Hydrogen 2000 psig | 3.6 | 53.6 | 0.068 | 0.00212 | 0.265664 | 0.09276 |
| 19 | 316L | 316L | Hydrogen 2000 psig | 5 | 117 | 0.074 | 0.00018 | 0.022556 | 0.13035 |
| 20 | AUM10 | 316L | Hydrogen 2000 psig | 5.9 | 103.8 | 0.034 | 0.00034 | 0.042607 | 0.00575 |
| 21 | AUM10 | 316L | Hydrogen 2000 psig | 3.8 | 58.6 | 0.0825 | 0.00066 | 0.082707 | 0.20204 |
| 22 | AUM10 | 316L | Hydrogen 2000 psig | 5 | 70 | 0.099 | 0.00274 | 0.343358 | 0.42208 |
| 23 | AUM10 | A286 | Hydrogen 2000 psig | 13 | 82 | 0.036 | 0.00006 | 0.007519 | 0.00723 |
| 24 | AUM10 | A286 | Hydrogen 2000 psig | 4.5 | 52.6 | 0.0765 | 0.00084 | 0.105263 | 0.14902 |
| 25 | AUM10 | A286 | Hydrogen 2000 psig | 5.1 | 75.9 | 0.082 | 0.00008 | 0.010025 | 0.19714 |
| 26 | DLC AUM10 | 316L | Air | 23.3 | 12.5 | 0.038 | -0.00006 | -0.00752 | 0.00898 |
| 27 | DLC AUM10 | 316L | Air | 5.18 | 11.34 | 0.038 | 0.00000 | 0 | 0.00898 |
| 28 | Nitride AUM10 | 316L | Air | 5.18 | 13.7 | 0.1035 | 0.00372 | 0.466165 | 0.50537 |
| 29 | Nitride AUM10 | 316L | Air | 4.97 | 13.7 | 0.1065 | 0.00408 | 0.511278 | 0.56746 |
| 30 | DLC AUM10 | 316L | Hydrogen 2000 psig | 8 | 6 | 0.0295 | -0.00006 | -0.00752 | 0.00326 |
| 31 | DLC AUM10 | 316L | Hydrogen 2000 psig | 8 | 22 | 0.0935 | 0.00038 | 0.047619 | 0.33496 |
| 32 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 4 | 48 | 0.0695 | 0.00056 | 0.070175 | 0.10127 |
| 33 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 4 | 23 | 0.0635 | 0.00048 | 0.06015 | 0.07044 |

Table 9. Test Results for Pre-charged Samples (Disks)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 91 g or 0.2 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Track Finish (μ -inch) | Inside Track Finish (μ -inch) | Averaged Track Diameter (inch) | Track width (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) |
|----------|---------------|--------------|--------------------|-------------------------------------|------------------------------------|--------------------------------|--------------------|--------------------|---|
| 34 | 316L | 316L | Hydrogen 2000 psig | 25.9 | 307.9 | 0.808 | 0.073 | 0.00380 | 0.47619 |
| 35 | 316L | 316L | Hydrogen 2000 psig | 21.7 | 59.8 | 0.817 | 0.076 | 0.00396 | 0.49624 |
| 36 | AUM10 | 316L | Hydrogen 2000 psig | 32 | 182 | 0.9665 | 0.0985 | 0.02774 | 3.47619 |
| 37 | AUM10 | 316L | Hydrogen 2000 psig | 27 | 96 | 0.9765 | 0.0915 | 0.02456 | 3.07769 |
| 38 | AUM10 | A286 | Hydrogen 2000 psig | 28 | 176 | 0.9615 | 0.1015 | 0.04106 | 5.14536 |
| 39 | AUM10 | A286 | Hydrogen 2000 psig | 33 | 138 | 0.9195 | 0.0895 | 0.02822 | 3.53634 |
| 40 | DLC AUM10 | A286 | Air | not measured | not measured | not measured | not measured | -0.00016 | -0.04571 |
| 41 | DLC AUM10 | A286 | Air | not measured | not measured | not measured | not measured | -0.00004 | -0.01143 |
| 42 | Nitride AUM10 | A286 | Air | 59.1 | 40.8 | 0.985 | 0.059 | 0.00208 | 0.29714 |
| 43 | Nitride AUM10 | A286 | Air | 24 | 26 | 0.9665 | 0.0695 | 0.00084 | 0.12 |
| 44 | DLC AUM10 | A286 | Hydrogen 2000 psig | 45 | 45 | 1.0215 | 0.0295 | -0.00016 | -0.04571 |
| 45 | DLC AUM10 | A286 | Hydrogen 2000 psig | 35 | 35 | 1.04 | 0.015 | -0.00012 | -0.03428 |
| 46 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 35 | 23 | 0.943 | 0.046 | 0.00210 | 0.3 |
| 47 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 57 | 42 | 0.984 | 0.043 | 0.00006 | 0.00857 |
| 48 | DLC AUM10 | 316L | Air | 29.5 | 28.5 | 0.978 | 0.026 | 0.00026 | 0.07429 |
| 49 | DLC AUM10 | 316L | Air | not measured | not measured | not measured | not measured | -0.00006 | -0.01714 |
| 50 | Nitride AUM10 | 316L | Air | 83.8 | 74.6 | 1.0005 | 0.0875 | -0.00028 | -0.04 |
| 51 | Nitride AUM10 | 316L | Air | 101.2 | 65.1 | 1.0205 | 0.0695 | 0.00042 | 0.06 |
| 52 | DLC AUM10 | 316L | Hydrogen 2000 psig | 28 | 28 | 1.1055 | 0.0285 | -0.00006 | -0.01714 |
| 53 | DLC AUM10 | 316L | Hydrogen 2000 psig | 21 | 21 | 1.024 | 0.025 | -0.00022 | -0.06286 |
| 54 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 30 | 17 | 0.992 | 0.064 | 0.00054 | 0.07714 |
| 55 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 53 | 46 | 0.971 | 0.083 | 0 | 6.3E-18 |

Table 10. Test Results for Pre-charged Samples (Pins)

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 91 g or 0.2 lb)

| Test No. | Disk Material | Pin Material | Test Environ. | Outside Wear Finish (μ -inch) | Inside Wear Finish (μ -inch) | Averaged Wear Spot Dia. (inch) | Weight Loss (gram) | Estimated Volume Loss (mm^3) | Calculated Volume Loss (mm^3) |
|----------|---------------|--------------|--------------------|------------------------------------|-----------------------------------|--------------------------------|--------------------|---|--|
| 34 | 316L | 316L | Hydrogen 2000 psig | 9.1 | 76.2 | 0.0805 | 0.00088 | 0.110276 | 0.18300 |
| 35 | 316L | 316L | Hydrogen 2000 psig | 8.7 | 68.1 | 0.0805 | 0.00074 | 0.092732 | 0.18300 |
| 36 | AUM10 | 316L | Hydrogen 2000 psig | 8 | 116 | 0.094 | 0.00192 | 0.240602 | 0.34223 |
| 37 | AUM10 | 316L | Hydrogen 2000 psig | 3 | 101 | 0.0885 | 0.00310 | 0.388471 | 0.26822 |
| 38 | AUM10 | A286 | Hydrogen 2000 psig | 5 | 123 | 0.097 | 0.00146 | 0.182957 | 0.38861 |
| 39 | AUM10 | A286 | Hydrogen 2000 psig | 5 | 70 | 0.0925 | 0.00174 | 0.218045 | 0.32067 |
| 40 | DLC AUM10 | A286 | Air | 10.4 | 13.6 | 0.0545 | 0.00004 | 0.005013 | 0.03812 |
| 41 | DLC AUM10 | A286 | Air | 10.5 | 2.6 | 0.042 | 0.00088 | -0.11028 | 0.01341 |
| 42 | Nitride AUM10 | A286 | Air | 7.2 | 9.3 | 0.119 | 0.00252 | 0.315789 | 0.89095 |
| 43 | Nitride AUM10 | A286 | Air | 6 | 14 | 0.11 | 0.00536 | 0.671679 | 0.64706 |
| 44 | DLC AUM10 | A286 | Hydrogen 2000 psig | 6 | 7 | 0.0385 | 0.00092 | 0.115288 | 0.00946 |
| 45 | DLC AUM10 | A286 | Hydrogen 2000 psig | 4 | 5 | 0.033 | 0.00042 | -0.05263 | 0.00510 |
| 46 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 7 | 25 | 0.083 | 0.00148 | 0.185464 | 0.20702 |
| 47 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 8 | 33 | 0.067 | 0.00360 | 0.451128 | 0.08739 |
| 48 | DLC AUM10 | 316L | Air | 19.99 | 4.01 | 0.0315 | 0.00006 | 0.007519 | 0.00423 |
| 49 | DLC AUM10 | 316L | Air | 9.1 | 11.3 | 0.03 | 0.00004 | -0.00501 | 0.00348 |
| 50 | Nitride AUM10 | 316L | Air | 13.8 | 8.3 | 0.1085 | 0.00482 | 0.60401 | 0.61197 |
| 51 | Nitride AUM10 | 316L | Air | 6.3 | 19.1 | 0.112 | 0.00692 | 0.867168 | 0.69620 |
| 52 | DLC AUM10 | 316L | Hydrogen 2000 psig | 6 | 3 | 0.0295 | 0.00010 | -0.01253 | 0.00326 |
| 53 | DLC AUM10 | 316L | Hydrogen 2000 psig | 10 | 4 | 0.028 | 0.00010 | -0.01253 | 0.00264 |
| 54 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 5 | 32 | 0.071 | 0.00040 | 0.050125 | 0.11035 |
| 55 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 4 | 20 | 0.0935 | 0.00268 | 0.33584 | 0.33492 |

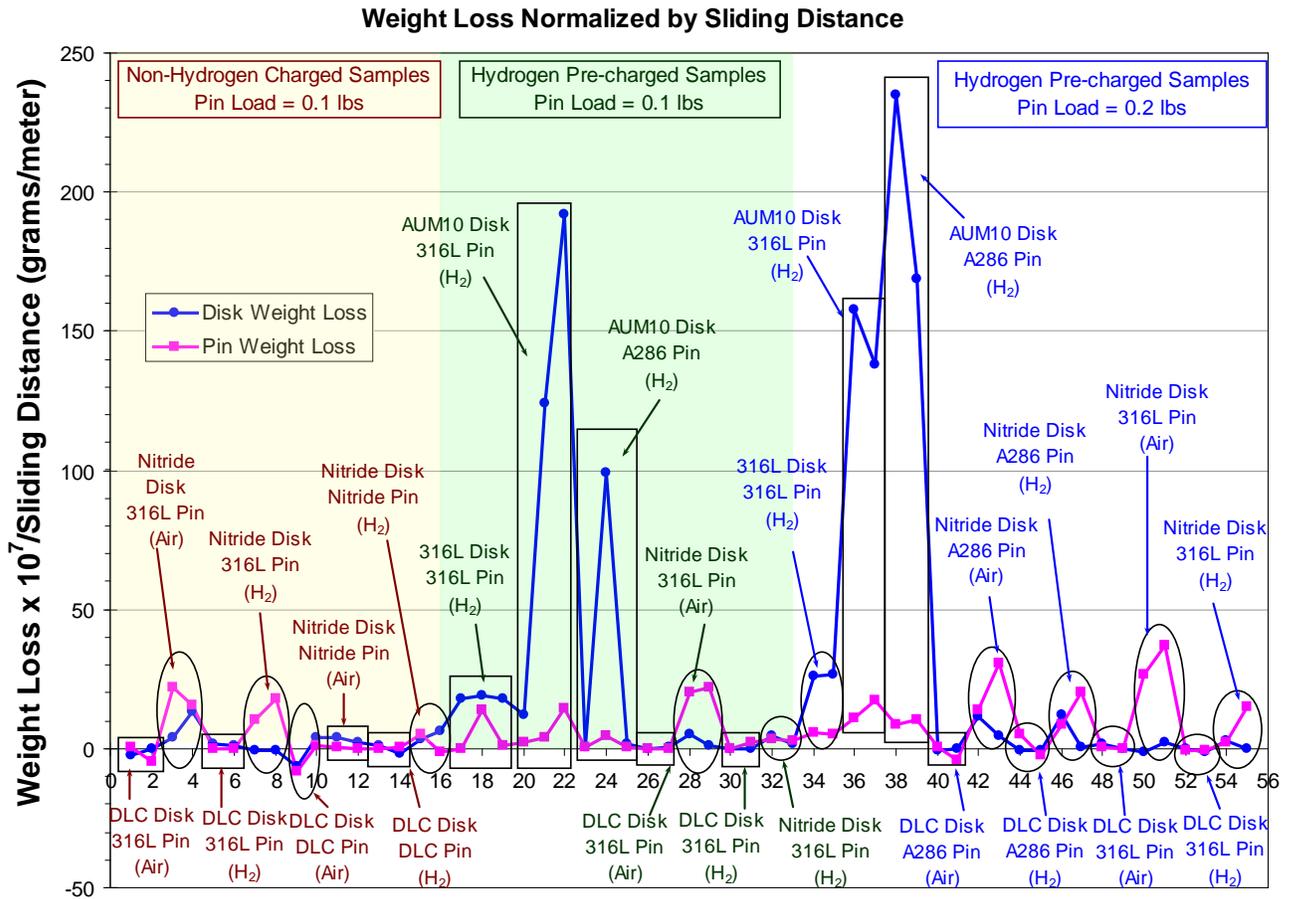


Figure 45. Disk weight change (loss) after 2000 m of travel distance and under a pin load of 45.4 g (0.1 lb) or 91 g (0.2lb)—non-pre-charged, pre-charged, non-coated, and coated.

9.2 WEAR COEFFICIENT (K_w)

A wear coefficient was calculated based on the Archard Equation [5-8] for each test where the volume loss took place by the wear. This coefficient is the proportionality between the wear rate (volume loss per unit sliding distance) and the ratio of the external load to the flow pressure of the material. Mathematically, it can be regarded as the probability of any material surface junctions leading to transferred fragments [5]. The majority of the wear coefficients based on the SRNL test data are within the range of those for the stainless steels as reported in the literature, on the order of 0.01 [5].

A wear equation was proposed by Holm in 1946 [6] and later was derived analytically by Archard in 1953 [7,8] as

$$W=kL/3p,$$

where W is the wear rate (volume loss per unit sliding distance), L is the external loading acting on the flat plate, p is the flow pressure of the metal (the Brinell Hardness can be used as an approximation), the “3” in the denominator is a shape factor for the assumed circular junctions and hemispheric fragments or debris (for a square junctions and cubical fragment, the factor would be 1), and k is the probability of any material surface junctions leading to transferred fragments [5].

The above Archard Equation is rearranged to incorporate the test data for calculation the wear coefficient K_w , in which the shape factor has been absorbed:

$$K_w = \frac{\text{Hardness (kg/mm}^2\text{)} \times (\text{Wear Volume per unit Sliding Distance, mm}^2\text{)}}{\text{Total Normal Load (kg)}}$$

Typical density of 7.98 g/cc was used in estimating the wear volume for the stainless steels (316L, A285, and AUM10). For the DLC coating, the pure diamond density 3.5 g/cc [9] was used in the Archard equation for DLC-coated samples. Because its hardness is from HV 1000 to 3500 [3], which exceeds the Brinell hardness scale HB 739 (at HV 832) [10], a value of 1000 was used in the Archard equation for DLC coating by extrapolating from charts provided in the literature [11].

The maximum density of the nitriding steels is 7.7g/cc (typical stainless steel is 7.98 g/cc) [12]. Therefore, a density of 7 g/cc was used for the nitride coating in the analysis for K_w . Because nitriding is a thermo-chemical diffusion process and its properties depend on the substrate material, the hardness of the nitriding steel in general increases with the chromium content in the steel. The surface hardness of 17% Cr stainless steel was used (Note that the chromium contents in the materials investigated in this report are 16.7, 14.0, and 12.5% for, respectively, 316L pins, A286 pins, and AUM10 disks). The surface hardness value is HV 1050 [3], which quickly drops to HV 200 at 0.2 mm (0.008 inch.) below the surface, typical for the stainless steels.

The wear coefficients for the test samples are listed in Table 11-Table 13 below, and is shown graphically in Figure 46. Many of the tests resulted in $K_w < 0.001$, which may be consistent with the observation by Rabinowicz (Table 6.1 of Ref. 5), in which he reported that $k = 21 \times 10^{-3}$ (equivalently, $K_w = 7 \times 10^{-3}$) for stainless steels. Rabinowicz also reported that the values of k above 0.1 are very rare in his 172 data points, with most of them are in between 10^{-4} and 10^{-2} (Figure 6.15 in Ref. 5) on non-lubricated surfaces for metals and non-metals.

Table 11. K_w for Non-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test Number | Disk Material | Pin Material | Test Environ. | K_w (Disk) | K_w (Pin) |
|-------------|------------------|--------------|-----------------------|--------------|-------------|
| 1 | DLC AUM10 | 316L | Air | -0.00136 | 0.00001 |
| 2 | DLC AUM10 | 316L | Air | 0.00000 | -0.00026 |
| 3 | Nitride AUM10 | 316L | Air | 0.00094 | 0.00129 |
| 4 | Nitride AUM10 | 316L | Air | 0.00301 | 0.00091 |
| 5 | DLC AUM10 | 316L | Hydrogen 2000 psig | 0.00094 | 0.00000 |
| 6 | DLC AUM10 | 316L | Hydrogen 2000 psig | 0.00056 | -0.00001 |
| 7 | Nitride AUM10 | 316L | Hydrogen 2000 psig | -0.00015 | 0.00062 |
| 8 | Nitride AUM10 | 316L | Hydrogen 2000 psig | -0.00009 | 0.00106 |
| 9 | DLC AUM10 | DLC 316L | Air | -0.00414 | -0.00510 |
| 10 | DLC AUM10 | DLC 316L | Air | 0.00240 | 0.00069 |
| 11 | Nitride AUM10 | Nitride 316L | Air | 0.00095 | 0.00007 |
| 12 | Nitride AUM10 | Nitride 316L | Air | 0.00057 | 0.00002 |
| 13 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | 0.00079 | -0.00006 |
| 14 | DLC AUM10 | DLC 316L | Hydrogen 2000 psig | -0.00122 | 0.00026 |
| 15 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 0.00082 | 0.00119 |
| 16 | Nitride AUM10 | Nitride 316L | Hydrogen 2000 psig | 0.00141 | -0.00028 |

Note: The negative values of K_w are associated with negative wear volume in the previous section

Table 12. K_w for Pre-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 45.4 g or 0.1 lb)

| Test Number | Disk Material | Pin Material | Test Environ. | K_w (Disk) | K_w (Pin) |
|-------------|------------------|--------------|--------------------|--------------|-------------|
| 17 | 316L | 316L | Hydrogen 2000 psig | 0.00091 | 0.00001 |
| 18 | 316L | 316L | Hydrogen 2000 psig | 0.00098 | 0.00081 |
| 19 | 316L | 316L | Hydrogen 2000 psig | 0.00092 | 0.00006 |
| 20 | AUM10 | 316L | Hydrogen 2000 psig | 0.00057 | 0.00013 |
| 21 | AUM10 | 316L | Hydrogen 2000 psig | 0.00596 | 0.00023 |
| 22 | AUM10 | 316L | Hydrogen 2000 psig | 0.00920 | 0.00085 |
| 23 | AUM10 | A286 | Hydrogen 2000 psig | 0.00003 | 0.00003 |
| 24 | AUM10 | A286 | Hydrogen 2000 psig | 0.00475 | 0.00040 |
| 25 | AUM10 | A286 | Hydrogen 2000 psig | 0.00007 | 0.00004 |
| 26 | DLC AUM10 | 316L | Air | -0.00012 | -0.00002 |
| 27 | DLC AUM10 | 316L | Air | 0.00044 | 0.00000 |
| 28 | Nitride AUM10 | 316L | Air | 0.00118 | 0.00120 |
| 29 | Nitride AUM10 | 316L | Air | 0.00020 | 0.00128 |
| 30 | DLC AUM10 | 316L | Hydrogen 2000 psig | -0.00014 | -0.00002 |
| 31 | DLC AUM10 | 316L | Hydrogen 2000 psig | -0.00014 | 0.00013 |
| 32 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 0.00107 | 0.00018 |
| 33 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 0.00032 | 0.00016 |

Note: The negative values of K_w are associated with negative wear volume in the previous section

Table 13. K_w for Pre-charged Samples

(Sliding Distance: 2000 m, Speed: 800 mm/s, Pin Load: 91 g or 0.2 lb)

| Test Number | Disk Material | Pin Material | Test Environ. | K_w (Disk) | K_w (Pin) |
|-------------|------------------|--------------|-----------------------|--------------|-------------|
| 34 | 316L | 316L | Hydrogen 2000 psig | 0.00066 | 0.00017 |
| 35 | 316L | 316L | Hydrogen 2000 psig | 0.00068 | 0.00015 |
| 36 | AUM10 | 316L | Hydrogen 2000 psig | 0.00378 | 0.00032 |
| 37 | AUM10 | 316L | Hydrogen 2000 psig | 0.00331 | 0.00051 |
| 38 | AUM10 | A286 | Hydrogen 2000 psig | 0.00563 | 0.00037 |
| 39 | AUM10 | A286 | Hydrogen 2000 psig | 0.00404 | 0.00046 |
| 40 | DLC AUM10 | A286 | Air | -0.00025 | 0.00001 |
| 41 | DLC AUM10 | A286 | Air | -0.00006 | -0.00019 |
| 42 | Nitride AUM10 | A286 | Air | 0.00135 | 0.00062 |
| 43 | Nitride AUM10 | A286 | Air | 0.00055 | 0.00135 |
| 44 | DLC AUM10 | A286 | Hydrogen 2000 psig | -0.00027 | 0.00022 |
| 45 | DLC AUM10 | A286 | Hydrogen 2000 psig | -0.00020 | -0.00010 |
| 46 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 0.00142 | 0.00038 |
| 47 | Nitride AUM10 | A286 | Hydrogen 2000 psig | 0.00004 | 0.00089 |
| 48 | DLC AUM10 | 316L | Air | 0.00046 | 0.00001 |
| 49 | DLC AUM10 | 316L | Air | -0.00009 | -0.00001 |
| 50 | Nitride AUM10 | 316L | Air | -0.00018 | 0.00077 |
| 51 | Nitride AUM10 | 316L | Air | 0.00026 | 0.00109 |
| 52 | DLC AUM10 | 316L | Hydrogen 2000 psig | -0.00009 | -0.00001 |
| 53 | DLC AUM10 | 316L | Hydrogen 2000 psig | -0.00037 | -0.00002 |
| 54 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 0.00035 | 0.00006 |
| 55 | Nitride AUM10 | 316L | Hydrogen 2000 psig | 0.00000 | 0.00044 |

Note: The negative values of K_w are associated with negative wear volume in the previous section

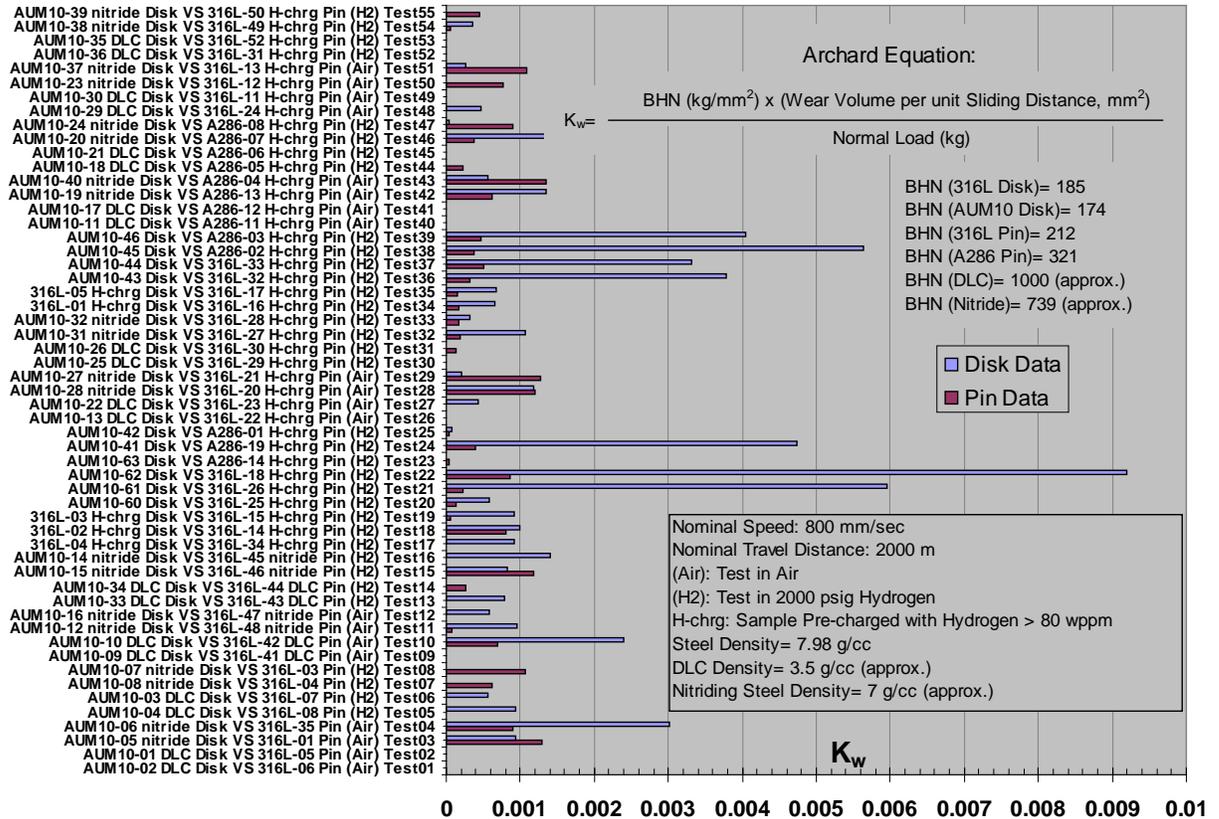


Figure 46. The wear coefficients calculated for all wear test cases.

Examination of the wear coefficient data displayed in Figure 46 indicates that consistent with the previous Phase I wear testing study AUM10 disk material exhibits more wear in comparison to the 316L disk material in the uncoated disk-uncoated pin condition for samples tested in hydrogen in the pre-charged condition and at both 0.1 and 0.2 lb loads. Additional observations from the wear coefficient data are as follows:

1. Samples tested with nitride coated disk display highest pin wear—this can be attributed to the high hardness and increased surface roughness associated with the nitride coatings.
2. There is minimal influence associated with hydrogen pre-charge and increasing load on K_w.
3. Samples involving the DLC coating typically display the lowest K_w value which is consistent both with visual surface examination and wear track measurements.

10.0 CONCLUSIONS

Based on the data collected for a travel distance of 2000 m and loads of both 45.4 g and 91 g, the tribological effects of the stainless disks (316L and AUM10) and the pins (316L and A286) in the pre-charged or coated conditions are not significant. Consistent with Phase I test results, the Phase II results do show a tendency for somewhat higher wear of non-coated materials sliding against each other in a gaseous hydrogen environment. Results for coated samples either nitride or DLC show that the coatings reduce the overall sample wear for samples tested in both air and hydrogen. It should be noted that for test conducted with nitride coated disks and non-coated pins, that increased wear was seen on the pins when compared to similar samples tested with DLC coated disks. This increased pin wear is most likely due to the increased roughening of the disk surface associated with the nitride coating application process.

Testing conducted in Phase II which was aimed at helping understand the previously collected results in Phase I; several critical questions were addressed: 1) does increasing the wear distance increase wear and aid weight change measurement sensitivity, 2) will pre-charging in hydrogen exacerbate the effect of gaseous hydrogen on surface wear, 3) will increased load increase the wear in hydrogen, and 4) can surface coatings reduce wear in both air and hydrogen.

Results from Phase II testing indicate that increasing the load from 45g to 91g (0.1 to 0.2 lb) does show some increase in wear. Additionally, increased test load also shows a trend of increasing wear for nitride coated samples. The increased load, however, does not appear to influence the wear behavior of DLC coated materials. Analysis of the test data also indicates that pre-charging with hydrogen has a slight effect for uncoated samples but shows essentially no effect for coated samples.

The major result from Phase II testing is the significant impact of surface coatings on wear. Both surface coating materials—nitride or DLC—reduce wear in air and in hydrogen (compared to non-coated disks), and they performed equally well in resisting wear. However, as previously mentioned, the non-coated materials (pins) tested against nitride coated materials (disks) exhibited more wear than the non-coated materials tested against DLC coated materials resulting from the inherent nature of the surface due to nitriding. From the Phase II test results, it may be concluded that DLC coating appears to be the most versatile coatings for reducing wear.

11.0 REFERENCES

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- [11] Steels Hardness Conversions, www.grantadesign.com/images/hardness.fe3.gif.
- [12] Nitriding Steels, www.thomasnet.com/products/nitriding-steel-80033004-1.html

APPENDIX A STAINLESS STEEL TYPE 316L (FOR DISKS)



Where Quality Isn't Expensive.....It's Priceless!

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INCORPORATED

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 WEBSITE: WWW.DIVERSIFIEDMETALS.NET
 49 MAIN STREET MONSON, MA 01057-0065

FAX: 413-267-3151
 PHONE: 413-267-5101
 CAGE CODE: 57067
 PO BOX 65

| TEST CERTIFICATE | | | | | | |
|---|--------------|----------------|----------|--|----------|-----------|
| Customer: Washington Savannah River Co Building 723-A Aiken, SC 29808 | | | | Customer Order: 7P5896 | | |
| Ship To Address: Washington Savannah River Co Operations Receiving 731-1N Aiken, SC 29808 | | | | Item No.: 3 | | |
| | | | | PN: NONE | | |
| | | | | Dimensions: 2.000 IN DIA X 27.1250 INCHES | | |
| | | | | Alloy/Grade: STAINLESS STEEL 316 COND A | | |
| Specification/Grade: QQ-S-763 REV F | | | | | | |
| No. Of Boxes | No of Pieces | Length | Weight | Packing Slip No. | Heat No. | Lot No. |
| 1 | 1 | 27.1250 INCHES | 24.0 LBS | 59849 | 415377 | 426500950 |
| MILL TEST REPORT ATTACHED X | | | | | | |
| MATERIAL HAS NOT BEEN EXPOSED TO MERCURY WHILE IN DIVERSIFIED METALS INC. FACILITY. | | | | | | |
| This is to certify that the material covered by this report and shipped on the above order has been inspected in accordance with, and has been found to meet the applicable requirements described herewith, involving any specifications forming a part of this description. | | | | | | |
| We certify that the Chemical Analysis and Physical Test Results applying on the above order number are correct and true. | | | | | | |
| QUALITY APPROVAL: Sharon Brown Quality Control Administrator Date: November 29, 2007 | | | | | | |

Mill Cert. - REV 2. - 2/27/2007

Scanned
Acciaierie Valbruna S.p.A.



**CERTIFICATO DI COLLAUDO
 ABNAHMEPRUEFZEUGNIS
 INSPECTION CERTIFICATE
 CERTIFICAT DE RECEPTION
 EN 10204 , 3.1.B**

36100 VICENZA (Italia) - Viale della scienza, 25 z.l.
 Stab.: 39100 BOLZANO (Italia) - Via A. Volta, 4
 Cliente / Kunden / Purchaser / Client
 VALBRUNA CORP.
 31 IRON HORSE ROAD
 USA-OA/LAND NJ 07436-USA
 Produttore: **STABILIMENTO DI BOLZANO**
 Hersteller / Fabrik / producer

Avviso di Spedizione: D-BZ05001142
 Lieferungs-/Packungsauftrag

Certificato nr: MEST184033/2005/ 2
 Prüfung/Taufschein

Ordine nr: 12779/STOCK (1)
 Bestell/Vorbestellung/Bestellnummer

Conferma online nr: E104007851
 Werk/Our Order/Nr.

Marchio di Fabbrica:
 Zeichen des Lieferwerkes
 Trade mark
 Signe de l'usine / producer



Funzione del Collaudatore:
 Dienst der Werksschwermetalle
 Inspector / stamp/Prüfer von Festzeug

Oggetto Prove: Polished Annealed Peeled
 Prüfgegenstand: Item / Inventar / Probe

Tipo di Elaborazione: E+AOD
 Erzeugnisart/Herstellung / Prozess/Arbeitsverfahren

Specifiche:
 Anforderungen / Requirements / Conditions
 FIRST METALS 2003 316/316L A
 ASME (1) SA182 2001 S31600/03 (a)
 ASME (1) SA479 2001 S31600/03 (p)
 ASTM A193 2004C 88M
 ASTM A276 2004 S31600/03
 ASTM A370 2003A
 MIL-S-882 B/1 316
 AMS-QQ-S-763 98 316/316L
 (2) SEC. II PT. A 2001 EDITION ADD.2002
 (1) SEC. II PT. A 2001 EDITION ADD.2002
 (3) SEC. II PT. A 2001 EDITION ADD.2002

AMS 5648 K S31600
 ASME (1) SA193 2001 88M (1)
 ASME (1) SA484 2001
 ASTM A194 99 8M
 ASTM A314 97 S31600/03
 ASTM A473 99 S31600/03
 NACE MR0175 2002 S31600/03
 QQ-S-763 E/1 316/316L

AMS 5653 F S31603
 ASME (1) SA320 2001 88M (2)
 ASTM A182 2002 S31600/03 (4)
 ASTM A262 2002A
 ASTM A320 2004 88M
 ASTM A479 2004 S31600/03
 QQ-S-763 F 316/316L

(1) Chemical analysis only and mechanical properties.
 (2) SEC. II PT. A 2001 EDITION ADD.2002
 (4) Chemical analysis only.

APPROVED BY CR
 DATE 1/6/06
CAROL RALPH
 Q.C. MANAGER

Qualità: S31603
 Werkstoff/Grade/Name

Punzonatura: S31803
 Kennzeichnung/Marking/Markierung

Marca: MVAPML MAXIVAL
 Markenbezeichnung/Brand/Markenname

| Pos. nr. Pos. nr. Nr. der Probe | Oggetto Description Design. der Probe | Dimensioni - in Abmessungen Dimension | Tolleranza Tolerance Abweichung Toleranz | Lunghezza - FI Length Länge Longueur | Colata Schmelze Heat Coulée | Pezzi Stückzahl Pieces | Peso - LB Gewicht Poids | Lotto nr. Lose Lot Nr. |
|---------------------------------------|---|---|---|---|--------------------------------------|------------------------------|-------------------------------|------------------------------|
| 0180 | Round | 2,0000 | A484 | 12 / 13 | 415377 | | 4,113 | 426500950 |

Sono state soddisfatte tutte le condizioni richieste
 Die gefällten Anforderungen sind erfüllt. Anlage erfüllt
 The material has been inspected in accordance with the requirements
 Le matériel a été inspecté conformément aux exigences

Controllo antisolfatazione: OK
 Verwechslungsprüfung: keine Verwechslung durchgeführt
 Antisulfating test: performed OK
 Corrosion resistance test: i.s.s.

Controllo visivo e dimensionale: soddisfatta le esigenze:
 Beschauung und Abmessung: ohne Beanstandung
 Visual inspection and dimensional check: satisfactory
 Contrôle visuel et dimensionnel: satisfaisant

| TEST | Prove/versuche Examination/Prüfung Long Short, Dia nach DIN, Thickness Läng. nach, mm | ° F | Preis. Stärke Temperatur | Svernamento Schweißnaht Weld Seam Line Rp 0,2% k.s.l. | Svernamento Schweißnaht Weld Seam Line Rp 0,2% k.s.l. | Resistenza Zugfestigkeit Tensile strength Rupture & Yield Rm k.s.l. | Allungamento Zugdehnung Elongation Mangement E 4d % | Strizione Zugverschiebung Reduction of Area 31603 RA % | Resistenza Korrosionsbest. Korrosion Resistance | Durezza Härte Festigkeit Stoß HB |
|---|---|------------|--------------------------------|--|--|--|--|---|--|--|
| Valori richiesti 1 Anforderungen/Prüfungswerte Values dimension | | min max | | 30 | - | 75 115 | - 40 | - 50 | - | 140 223 |
| A | 12.50 | 68 L | | 39 | | 86 | 66 | 73 | | 185 |

Grain size for ASTM E112 : 5

Analisi chimica
 Chemisches Zusammensetzungs/Chemical Analysis/Analyse der Legierung

| Colata / Heat Schmelze/Coûlée | min - max | 1,25 2,00 | 18,20 18,20 | 5,00 3,00 | 1,00 1,00 | 10,20 14,00 | 0,040 | 0,020 0,030 | 0,100 | - | - | - | - |
|----------------------------------|--------------|--------------|----------------|---------------|--------------|----------------|---------------|----------------|--------------|--------------|---|---|---|
| 415377 | C % 0,018 | Si % 0,70 | Mn % 1,57 | Cr % 17,11 | Mo % 2,01 | Cu % 0,42 | Ni % 10,23 | P % 0,028 | S % 0,026 | N % 0,078 | | | |

Produced without class I-II Ozone depleting substances.
 Solution heat treated free from continuous carbide network.

Annealing temperature: 1960° F for 2,50h/H2O,
 Micro and macro etch test: OK

Reduction ratio > 4 : 1
 Interganular corrosion test per ASTM A262,02, pract. A&E:ok

Melted and manufactured in Italy No welding or weld repair Material free from Mercury or radio-activity contamination
 The Quality Management System is Certified acc. Pressure Equipment Directive [97/23/EC] Annex 1.4., 4.3 by TÜV and ULDYD'S

DIVERSIFIED METALS, INC.
 49 MAIN STREET, MONSON, MA 01057
 PH 413-267-5101/FAX 413-267-3151
 SOLD TO:
 CUSTOMER: WASHINGTON SAVANNAH Co.
 P.O. # 7P589L
 ITEM# 3

A
V
Valbruna Group

Valbruna Stainless, Inc.

31 Iron Horse Road
Oakland, NJ 07436
Tel. (201) 337-1233
Fax (201) 337-0833
njsales@valbruna.com

TEST CERTIFICATE

BUYER : Diversified Metals
BUYERS P.O. No 42239

11/10/2005
VALBRUNA No: 12779

| GRADE | SIZE / SHAPE | DESCRIPTION AND SPECIFICATION | | QTY (LBS) |
|--------|-----------------|--|---|-----------|
| S31603 | ROUND 2.000" | FIRST METALS 2003 316/316L ASME(1)SA182 2001 S31600/03 (1) ASME (1) SA479 2001 S31600/03 (3) ASTM A193 2004C B8M ASTM A276 2004 S31600/03 ASTM A370 2003A ML-S-862 B/1 316 | AMS-QS-763 98 316/316L (e) SEC. 11PT A 2001 EDITION ADD 2002 (1) SEC. 11PT A 2001 EDITION ADD 2002 (2) SEC. 11PT A 2001 EDITION ADD 2002 | 1,000 |

CHEMICAL ANALYSIS

*Chemical analysis only

| HEAT NUMBER | C | Mn | P | S | Si | Ni | Cr |
|-------------|---|----|---|---|----|----|----|
| 415377 | | | | | | | |

| Mo | Cu | Ti | Co | Cb+Ta | Sn | N |
|----|----|----|----|-------|----|---|
| | | | | | | |

MECHANICAL PROPERTIES

| TENSILE STRENGTH (KSI) | YIELD STRENGTH (KSI) | ELONG % | RED.OF AREA % | HARDNESS | | AS SHIPPED HARDNESS | |
|------------------------|----------------------|---------|---------------|----------|----------|---------------------|-----|
| | | | | BRINELL | ROCKWELL | | |
| | | | | | "B" | | "C" |
| | | | | | | | |

OTHER TEST(S) AND / OR REQUIREMENTS

| | | |
|----------------------------|------------------------------|--|
| AMS 5648 K S31600 | AMS 5653 FS31603 | (1) Chemical analysis only and mechanical properties |
| ASME (1) SA193 2001 B8M(1) | ASME (1) SA320 2001 B8M (2) | (2) SEC 11PT A 2001 EDITION ADD 2002 |
| ASME(1) SA484 2001 | ASTM A162 2002 S31600/03 (4) | (4) Chemical analysis only |
| ASTM A194 99 8M | ASTM A262 2002A | |
| ASTM A314 97 S31600/03 | ASTMA320 2004 B8M | |
| ASTM A479 99 S31600/03 | ASTM A479 2004S31600/03 | |
| NACE MR0175 2002 S31600/03 | QQ-S-763 F 316316L | |
| QQ-S-763 E/1 316/316L | | |

NOTES

"Statement of Compliance"
"This material, is produced by Acciaierie Valbruna and transferred to Valbruna Stainless, Inc., is mercury free and satisfies the requirements of the related specifications and standards."



Timothy P. Cooney
Authorized Representative

APPENDIX B STAINLESS STEEL TYPE AUM10 (FOR DISKS)

No.301

AICHI STEEL


 ハイクオリティスチール

軟磁性ステンレス鋼 AUMシリーズ

特 長

- | | |
|------------|-------------------------|
| 1. 優れた磁気特性 | 磁束密度が高く、保磁力が小さい。 |
| 2. 大きい電気抵抗 | 比抵抗が高く、交流特性が優れている。 |
| 3. 優れた耐食性 | 優れた耐食性を有している。(AUM6を除く) |
| 4. 豊富な品揃え | 用途、加工方法に応じて最適な鋼種選択ができる。 |

種 類

| 分類 | 鋼種 | 特性 | 磁束密度 | 保磁力 | 電気抵抗 | 引張強さ | 耐食性 | 切削性 | 冷鍛性 | 溶接性 | 鋼種選定のポイント |
|-----|--------|----|------|-----|------|------|-----|-----|-----|-----|--|
| 切削用 | AUM6 | ◎ | ◎ | ○ | ◎ | × | ◎ | — | — | — | ステンレス鋼ではない。珪素鉄の電気抵抗、切削性を改善した鋼種。磁束密度が非常に高い。電磁弁の可動鉄心に最適。 |
| | AUM10 | ○ | ◎ | ◎ | ◎ | ○ | — | — | ◎ | — | 磁気特性、電気抵抗、溶接性に優れている。油圧用電磁弁に幅広く使用。 |
| | AUM11 | ○ | ◎ | ◎ | ◎ | ○ | ◎ | — | — | — | AUM10の切削性を改善した鋼種。 |
| 冷鍛用 | AUM12 | ○ | ◎ | ○ | ○ | ○ | — | ◎ | ◎ | — | 軟鋼並の冷間鍛造性を有し、しかも優れた磁気特性、耐食性を有する鋼種。 |
| | AUM15 | ○ | ◎ | ○ | ○ | ○ | ◎ | ◎ | — | — | 冷間鍛造性と切削性を両立させた鋼種。耐食性、電磁特性も優れている。量産タイプの電子制御燃料噴射装置に最適。 |
| | AUM15H | ○ | ◎ | ○ | ○ | ○ | ◎ | ◎ | — | — | AUM15の冷鍛性を改善した鋼種。 |
| | AUM25 | ○ | ◎ | ◎ | ○ | ○ | ◎ | ◎ | — | — | 動的磁気特性が特に優れている。耐食性、冷鍛性、切削性も優れている。パルス駆動型電磁弁に最適。 |
| 耐食用 | AUM20 | ○ | ◎ | ○ | ◎ | ◎ | — | ○ | ◎ | — | SUS316並の優れた耐食性と優れた磁気特性を有する鋼種。 |

注) ◎: 特に優れている ○: 優れている ×: 劣る

用 途

電磁弁、EFI(電子制御式燃料噴射装置)、スピードセンサ など

AUMシリーズ

化学成分

| 分類 | 鋼種名 | 化学成分(%) | | | | | | | |
|-----|--------|---------|-----|------|----|----|----|----|----|
| | | C+N | Si | Cr | Al | Pb | S | Ti | Mo |
| 切削用 | AUM6 | 0.02 | 2.2 | 3.6 | 添加 | 添加 | 調整 | — | — |
| | AUM10 | 0.02 | 2.4 | 12.5 | — | — | 調整 | 添加 | — |
| | AUM11 | 0.02 | 2.2 | 12.5 | — | 添加 | 調整 | 添加 | — |
| 冷鍛用 | AUM12 | 0.02 | 0.2 | 12.0 | — | — | — | — | — |
| | AUM15 | 0.02 | 0.7 | 12.0 | 添加 | 添加 | 調整 | 添加 | — |
| | AUM15H | 0.02 | 0.2 | 12.0 | 添加 | 添加 | 調整 | 添加 | — |
| 加工用 | AUM25 | 0.02 | 0.2 | 10.0 | 添加 | 添加 | 調整 | — | — |
| | AUM20 | 0.02 | 0.2 | 19.0 | — | — | — | — | 添加 |

- (1) 13%Crステンレス鋼をベース成分として低(C+N)化とAl、Ti等の添加により磁気特性、電気抵抗、耐食性、冷鍛性を改善しています。
- (2) 高耐食性用途のために19%Cr鋼をベースとし、必要に応じてMoを添加することにより耐食性を改善しています。
- (3) Pb、Sにより切削性を改善しています。
- (4) AUM6は磁束密度を改善するためにCr量を3.6%にしています。

特性

1. 電磁特性 (代表特性値)

| 分類 | 鋼種名 | 磁束密度 (G) | | | | 保磁力 Hc(10el) | 電気抵抗 μΩ-cm | 透磁率 μmax |
|-----|-----------|----------|-------|-------|-------|-----------------|---------------|-------------|
| | | B1 | B2 | B10 | B20 | | | |
| 切削用 | AUM6 | 4000 | 12000 | 14000 | 14500 | 1.0 | 72 | 4700 |
| | AUM10 | 3000 | 11000 | 12000 | 12800 | 1.0 | 93 | 4200 |
| | AUM11 | 3600 | 11000 | 12100 | 12900 | 1.0 | 90 | 4100 |
| 冷鍛用 | AUM12 | 3300 | 11200 | 12900 | 14000 | 1.5 | 47 | 3500 |
| | AUM15 | 3500 | 10800 | 11800 | 12900 | 1.0 | 72 | 4500 |
| | AUM15H | 5400 | 11000 | 12200 | 13200 | 1.0 | 60 | 5000 |
| | AUM25 | 5400 | 10800 | 11700 | 12700 | 1.0 | 100 | 4900 |
| 加工用 | AUM20 | 6000 | 10500 | 11500 | 12000 | 1.0 | 53 | 5000 |
| 比較材 | SUS(SIOC) | 480 | 9000 | 13200 | 15500 | 1.8 | 15 | 1700 |
| | SUS403 | 30 | 140 | 290 | 660 | 3.3 | 57 | 40 |

2. 耐食性 (耐海水性)



- (1) AUM20はSUS316より耐食性が良好で、特に耐食性が要求される場合に最適です。
- (2) AUM10からAUM25までは、13%Crステンレス鋼より耐食性が優れています。



愛知製鋼株式会社

本社 愛知県東海市荒尾町ワノ割1番地 〒476 ☎(052) 604-1111 (大代表) FAX.052-601-0301
 東京支店 東京都千代田区大手町2丁目6番2号(日本ビル) 〒100 ☎(03) 3270-0854 (代表) FAX.03-3245-0649
 名古屋支店 名古屋市中村区名駅4丁目7番23号(豊田ビル) 〒450 ☎(052) 571-6493 (代表) FAX.052-561-8432
 大阪支店 大阪市中央区南船場4丁目3番11号(大阪豊田ビル) 〒542 ☎(06) 252-7373 (代表) FAX.06-251-9497
 広島営業所 広島市中区八丁堀11番28号(朝日広告ビル) 〒730 ☎(082) 228-0228 (代表) FAX.082-227-5140
 福岡営業所 福岡市博多区博多駅前2丁目11番16号(第2大西ビル) 〒812 ☎(092) 474-1690 (代表) FAX.092-474-0558
 米国駐在員事務所 771 Corporate Drive, Suite 460 Lexington, Kentucky 40503, -5444, U.S.A. ☎(606) 223-7052 FAX.606-224-4736
 工場 知多・刈谷・鍛冶・東浦

特約店

1940611

2008年8月24日
愛知製鋼株
品質保証部
品質・技術サービス室
余語

軟磁性ステンレス鋼AUM10の機械的性質

熱処理 焼鈍 900℃×2Hr での機械的性質を下記に示します。

| | |
|----------------------------|-------------------|
| 0.2%耐力(N/mm ²) | ： 290以上 (代表例 390) |
| 引張強さ(N/mm ²) | ： 490以上 (代表例 530) |
| 伸び(%) | ： 30以上 (代表例 37) |
| 絞り(%) | ： 55以上 (代表例 74) |
| 硬さ(HRB) | ： 83~95 (代表例 88) |

以上

APPENDIX C STAINLESS STEEL TYPE 316L (FOR PINS)

NAS
 6870 HIGHWAY 42 EAST
 Certificate: 434100 4 Mail To:
 Customer: RICKARD METALS, INC. # 1493

Date: 10/23/2008 Page: 1
 Steel: 316L/316
 Finish: CD

METALLURGICAL TEST REPORT

Ship To:
 SAVANNAH RIVER NL P.O. # 4R3679

Corrosion: ASTM A362-02a PRACTICE P-

NAS Order: IN 0052893 01

PRODUCT DESCRIPTION:
 Round bar, annealed, cold drawn. ASTM-A-484-06B UNS 321500 UNS 8216
 03, ASTM A276/04, ASTM A479/04, AMS 5640R, AMS 5653F, QQS-763F, EN
 10206 3.18 ASME SA479/01, AMS QQS-762A

REMARKS:
 Melted & Manufactured in the USA. Product complies w/ ENVDIT
 specs of EU directive 2007.95.EC.ROHS. Material Free from Mer
 cury contamination. No weld repair. NAS certifies the analysi
 is on certification is correct & the material meets specu at
 sted.

| Product Id | Skid # | Diameter | Size | Weight | Length | Mark | Pieces | Commodity Code |
|------------|--------|----------|------|--------|--------|------|--------|----------------|
| BC1389 1 | | .3750 | | 771 | 144.00 | 3 | 1 | |

CHEMICAL ANALYSIS CM(Country of Melt) ES(Spain) US(United States) ZA(South Africa) JP(Japan)

| HEAT | CM | C | CO | CR | CU | MO | N | NI |
|------|----|-------|-------|---------|-------|--------|--------|---------------|
| 4889 | US | .0296 | .1425 | 16.6747 | .4985 | 1.4133 | 2.1839 | .0390 11.0082 |
| | P | S | SI | TI | | | | |
| | | .0309 | .0243 | .4226 | .0053 | | | |

MECHANICAL PROPERTIES

| Product Id | 1 4 | 0 1 | MS | .2 YS | UTS | RA | ELC-4D | Oxalic |
|------------|-----|--------|-------|-------|-------|-------|--------|--------|
| | CT | Mo. | KSI | KSI | % | % | P/F | |
| BC1389 1 | CL | 212.00 | 81.43 | 99.84 | 75.20 | 46.01 | 1.00 | |

NAS hereby certifies that the analysis on this certification is correct and the material meets the specifications stated.

QC ENGINEER ERIC HESS 10/23/2008
Eric Hess Du. 10.24.08

APPENDIX D STAINLESS STEEL TYPE A286 (FOR PINS)

| | | |
|--|-----------------------------------|--------------------------------------|
| <p>CERTIFICATE OF TESTS CERT SERIAL# 000661755</p> <p>CARPENTER Carpenter Technology Corporation 101 West Bern Street, Reading, Pa. 19601 Tel: (610) 208-2000 (800) 338-4592</p> <p>08/30/08 CUSTOMER/BESTELLER/CLIENT RICKARD METALS, INC. # 1493</p> | <p>ABNAHMEPRUEFZEUGNIS</p> | <p>CERTIFICAT DE CONTROLE</p> |
|--|-----------------------------------|--------------------------------------|

- THE RECORDING OF FALSE, FICTITIOUS OR FRAUDULENT STATEMENTS OR ENTRIES ON THIS DOCUMENT MAY BE PUNISHED AS A FELONY UNDER FEDERAL STATUTES INCLUDING FEDERAL LAW, TITLE 18, CHAPTER 47.
- THE VALUES AND OTHER TECHNICAL DATA REPRESENT THE RESULTS OF ANALYSES AND TESTS MADE ON SAMPLES COLLECTED FROM THE TOTAL LOT. ORIGINAL DATA RECORDS CAN BE VIEWED BY REFERENCE TO THE CARPENTER ORDER NUMBER.
- MATERIAL IS MANUFACTURED FREE FROM MERCURY, RADIUM, ALPHA AND GAMMA SOURCE CONTAMINATION.
- THIS DOCUMENT SHALL NOT BE REPRODUCED, EXCEPT IN FULL, WITHOUT THE WRITTEN CONSENT OF CARPENTER TECHNOLOGY CORPORATION.

SELLER/VERKÄUFER/VENDEUR PAGE 1 OF 2
SAVANNAH RIVER NL P.O. # 4R3679

| | | | |
|--|---|----------------|----------------------|
| CUSTOMER ORDER NO./BESTELL.-NR./N° DE COMMANDE | CARPENTER NO./WERKS-NR./N° DE REFERENCE INTERNE | DATE/DATE/DATE | WEIGHT/GEWICHT/POIDS |
| | W94868 | 08/30/08 | 1888.000 |

HEAT NUMBER / SCHMELZE-NR. / N° DE COULEE: 555513

PRODUCT DESCRIPTION: CONSUMET A286 SOLUTION ANNEALED AGED GROUND

SPECIFICATION: ASTM-A638-00 (2004) GRADE 660, TYPE 2

 ASTM-A453-04 GRADE 660 CLASS B (REPORT GRAIN AS SHIPPED)
 AMS 5732 REV J (09/ /06)
 GE S-400 (10/31/07)
 GE S-1000 (01/02/08)
 FAIRCHILD MS 314 "CLASS A" REV 4 (01/17/03)
 DFARS 1998 EDITION

SIZE 0.375000 IN. (9.53 MM) RD BAR INGOT 3

PRIMARY HEAT CHEMISTRY (WT%): (TEST METHOD IS SHOWN IN PARENTHESIS)

| | | | | | |
|----------|----------|----------|----------|----------|----------|
| C (OES) | Mn (XRF) | SI (XRF) | P (OES) | S (OES) | CR (XRF) |
| 0.04 | 0.40 | 0.13 | 0.021 | 0.001 | 14.04 |
| NI (XRF) | MO (XRF) | CU (XRF) | CO (XRF) | AL (OES) | TI (OES) |
| 24.27 | 1.18 | 0.24 | 0.35 | 0.17 | 2.18 |
| V (XRF) | B (OES) | | | | |
| 0.26 | 0.007 | | | | |

GEAG SUPPLIER CODE 21100 / CARPENTER

THE CHEMICAL ANALYSIS OF THIS HEAT HAS BEEN PERFORMED BY THE CARPENTER SPECIALTY ALLOYS CHEMICAL LABORATORY, (CODE NUMBER PRI 100004), WHICH WAS ACCREDITED TO THE ISO/IEC 17025.

MILL HEAT TREATMENT:

| | | |
|----------------------|-----------------|---------------|
| TYPE | SOLUTION ANNEAL | AGE |
| TEMP | 1800F (982C) | 1328F (720C) |
| TIME (BATCH FURNACE) | 1.00 HOURS | 16.00 HOURS |
| QUENCH | WATER | AIR |

HARDNESS AS SHIPPED, HRC - 34 (MIDRADIUS)

CONTINUED ON NEXT PAGE

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555513-3

3/8 rd

CERTIFICATE OF TESTS ABNAHMEPRUEFZEUGNIS CERTIFICAT DE CONTROLE

CERT SERIAL# 000661755



CARPENTER

Carpenter Technology Corporation
101 West Bern Street, Reading, Pa. 19601
Tel: (610) 208-2000 (800) 338-4592

* THE RECORDING OF FALSE, FICTITIOUS OR FRAUDULENT STATEMENTS OR ENTRIES ON THIS DOCUMENT MAY BE PUNISHED AS A FELONY UNDER FEDERAL STATUTES INCLUDING FEDERAL LAW 1113-18 CHAPTER 4.
* THE VALUES AND OTHER TECHNICAL DATA REPRESENT THE RESULTS OF ANALYSES AND TESTS MADE ON SAMPLES COLLECTED FROM THE TOTAL LOT. ORIGINAL DATA RECORDS CAN BE TRACKED BY REFERENCE TO THE CARPENTER ORDER NUMBER.
* MATERIAL IS MANUFACTURED FREE FROM MERCURY, RADIUM, ALPHA AND GAMMA SOURCE CONTAMINATION.
* THIS DOCUMENT SHALL NOT BE REPRODUCED, EXCEPT IN FULL, WITHOUT THE WRITTEN CONSENT OF CARPENTER TECHNOLOGY CORPORATION.

08/30/08
CUSTOMER/BESTELLER/CLIENT
RICKARD METALS, INC. # 1493

SELLER/VERKÄUFER/VENDEUR PAGE 2 OF 2
SAVANNAH RIVER NL P.O. # 4R3679

| | | | |
|---|---|-----------------|----------------------|
| CUSTOMER ORDER NO./BESTELL-NR./N° DE COMMANDE | CARPENTER NO./WFRS-NR./N° DE REFERENCE IN LITRE | DATE/DATUM/DATE | WEIGHT/GEWICHT/POIDS |
| | W94868 | 08/30/08 | 1888.000 |

HEAT NUMBER / SCHMELZE-NR. / N° DE COULEE: 555513
 YIELD STRENGTH (0.20 %) KSI(MPA) 134.0 (924)
 TENSILE STRENGTH, KSI(MPA) 168.0 (1158)
 ELONGATION IN 1.53", % 23.0
 REDUCTION OF AREA, % 39.0

GRAIN SIZE PER ASTM E112: 9 (KALLINGS)

COMBINATION STRESS RUPTURE
 TEST TEMP 1200 F (649 C)
 STRESS, KSI(MPA) 56.0 (386)
 ELONGATION % 31.3
 HOURS 149.2

AFTER MINIMUM TIME UNDER SPECIFIED STRESS, THE STRESS RUPTURE TEST WAS OVERLOADED IN INCREMENTS AS FOLLOWS:

| TOTAL HOURS | STRESS KSI(MPA) |
|-------------|-----------------|
| 103 | 61.0 (421) |
| 111 | 66.0 (455) |
| 119 | 71.0 (490) |
| 127 | 76.0 (524) |
| 135 | 81.0 (558) |
| 143 | 86.0 (593) |

COMBINATION STRESS RUPTURE
 TEST TEMP 1200 F (649 C)
 STRESS, KSI(MPA) 70.0 (483)
 ELONGATION % 27.8
 HOURS 82.2

AFTER MINIMUM TIME UNDER SPECIFIED STRESS, THE STRESS RUPTURE TEST WAS OVERLOADED IN INCREMENTS AS FOLLOWS:

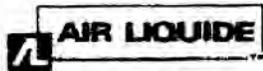
| TOTAL HOURS | STRESS KSI(MPA) |
|-------------|-----------------|
| 55 | 75.0 (517) |
| 66 | 80.0 (552) |
| 79 | 85.0 (586) |

MATERIAL PRODUCED ON THIS ORDER WAS MELTED AND MANUFACTURED IN THE U.S.A. MATERIAL HAS BEEN MELTED IN USA OR QUALIFYING COUNTRY TO DFARS REQUIREMENTS 252.225-7014 WITH ALTERNATE 1 FOR QUALIFYING COUNTRY 225.872.1. CARPENTER'S QUALITY MANAGEMENT SYSTEM WAS REGISTERED AS OF SEPTEMBER 2, 2004 TO THE REQUIREMENTS OF ISO 9001:2000 APPROVAL CERTIFICATE 07-0869 BY PERFORMANCE REVIEW INSTITUTE. CERTIFICATE OF TEST IS PREPARED IN ACCORDANCE WITH PARAGRAPH 3.1 OF EN 10204 (DIN 50049). WE HEREBY CERTIFY THAT THE ABOVE TEST DATA ARE IN ACCORDANCE WITH THE PURCHASE ORDER AND SPECIFICATION REQUIREMENTS.

MICHELE L. HEFFNER
 MET RELEASE/REQUIREMENTS ANALYST
 CARPENTER TECHNOLOGY CORPORATION

This certificate is made to the customer passed on this form. Carpenter neither makes, nor assumes responsibility for, any representation or certification to other parties.
 Die vorliegende Zertifikatsurkunde ist nur für den in diesem Formular genannten Kunden gültig. Carpenter übernimmt gegenüber Dritten keinerlei Haftung für die Aussagegenauigkeit dieser Zertifikatsurkunde.
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APPENDIX E CERTIFICATE OF RESEARCH GRADE HYDROGEN



CERTIFICATE OF BATCH/LOT ANALYSIS

Certification Of Batch/Lot #: 348LAP7698A

Product: Hydrogen Grade: Research

| | | | |
|------------------------|----------|------------------|--------|
| Customer: | WSRC | Test Cylinder #: | K-9017 |
| Date of Certification: | 1/3/2008 | Item Number: | |
| P.O. Number: | KO84592 | Valve: | 350 |
| Document Number: | 27606650 | Cylinder Size: | 44 |

ANALYSIS REPORT

| Major Component | Specification | Purity |
|-------------------|---------------|-----------|
| Hydrogen Research | 99.9995% | >99.9995% |

| Impurities | Specification | Actual Analysis |
|--------------------|---------------|-----------------|
| Moisture | <1 ppm | 0.1 PPM |
| Oxygen | <1 ppm | 0.1 PPM |
| Total Hydrocarbons | <0.2 ppm | N/D |
| Carbon Dioxide | <1 ppm | N/D |
| Carbon Monoxide | <1 ppm | N/D |
| Nitrogen | <1 ppm | N/D |

Notes: SERIAL NUMBERS:

H-73268, W-402637, H-87986, H-2434889, 14869,
 H-2087368, 55961, H-875341, T210623, T241253,
 AF-9053, H1098996, T240371, K118929, 39771,
 250-032814, 84-16414, 998, T-146554, K133399,
 K-071920, 250-278966, K161980, 250-721261, K-78020,
 K-128664, T-180699, K-9017, K-127508

Certified By:

Name



Cure date: 12/14/07

Exp date : 12/14/12

Air Liquide America, L.P.

11426 Fairmont Pkwy LaPorte, TX 77571-6000

Phone: 281-474-8400

ICSC-CYL-0307-W

Revision: 2

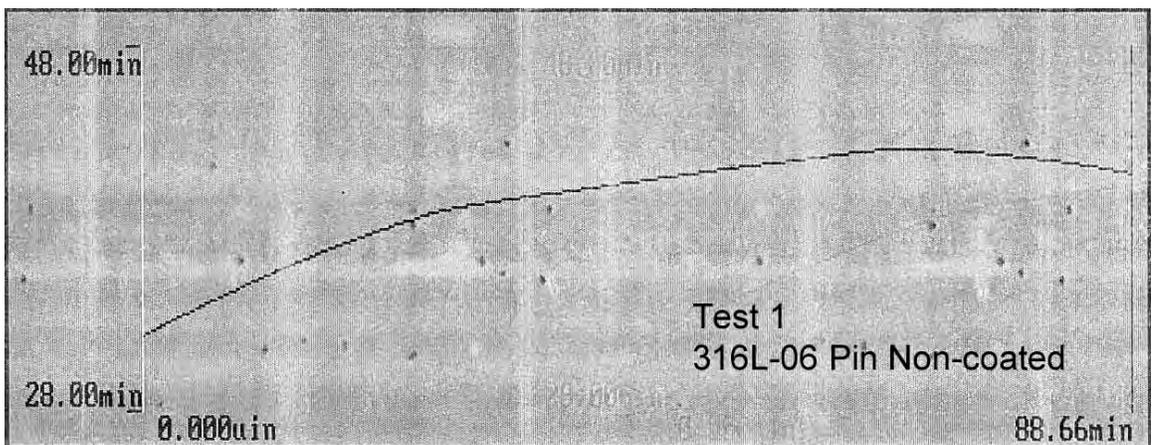
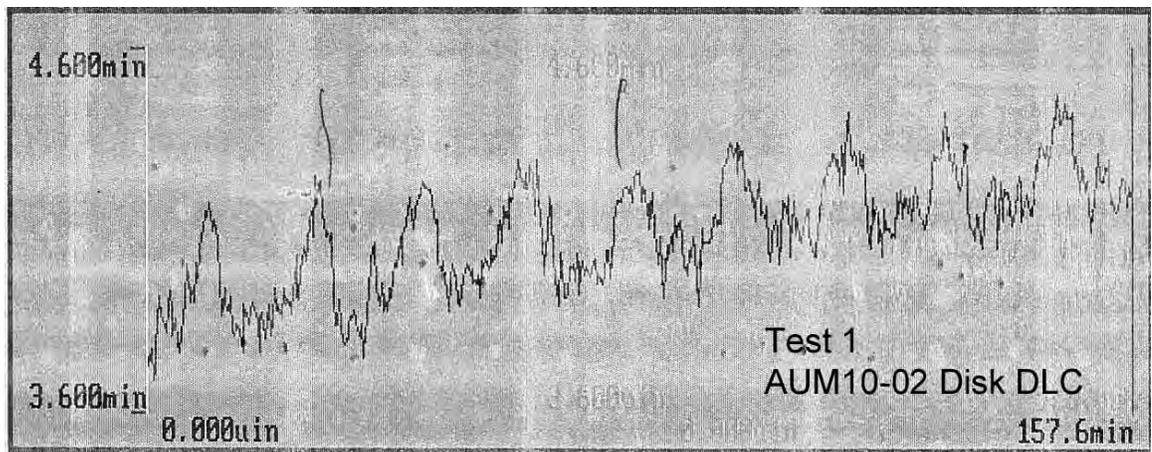
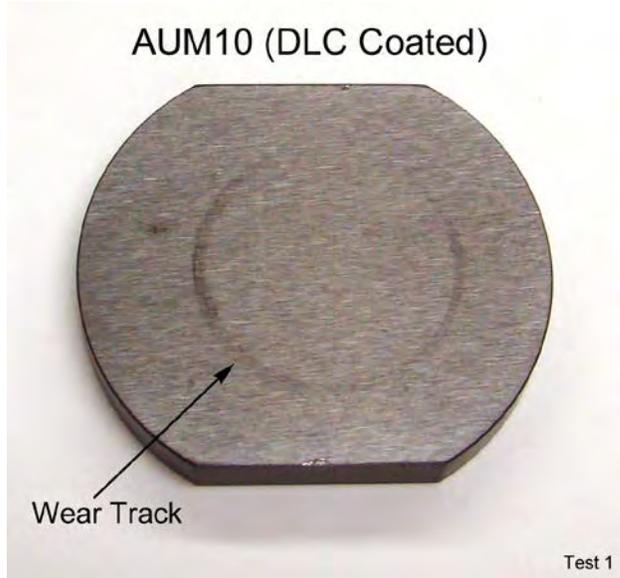
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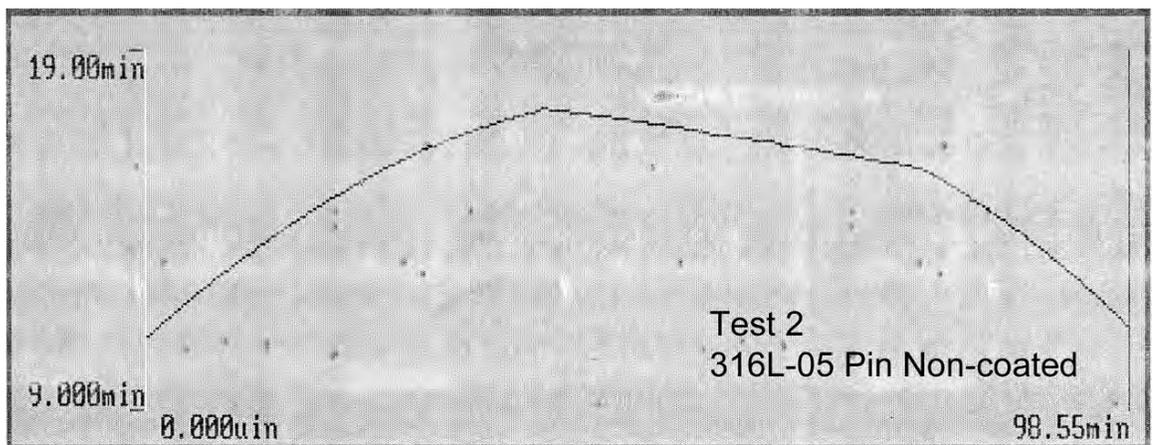
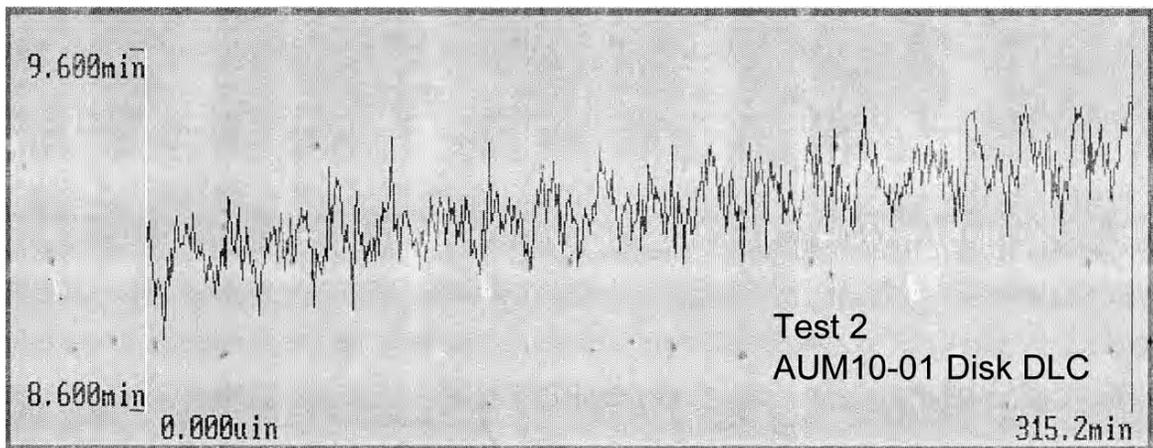
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**APPENDIX F POST-TEST SAMPLE PHOTOGRAPHS AND CONTACT
PROFILOMETER SCANS**

Test 1



Test 2

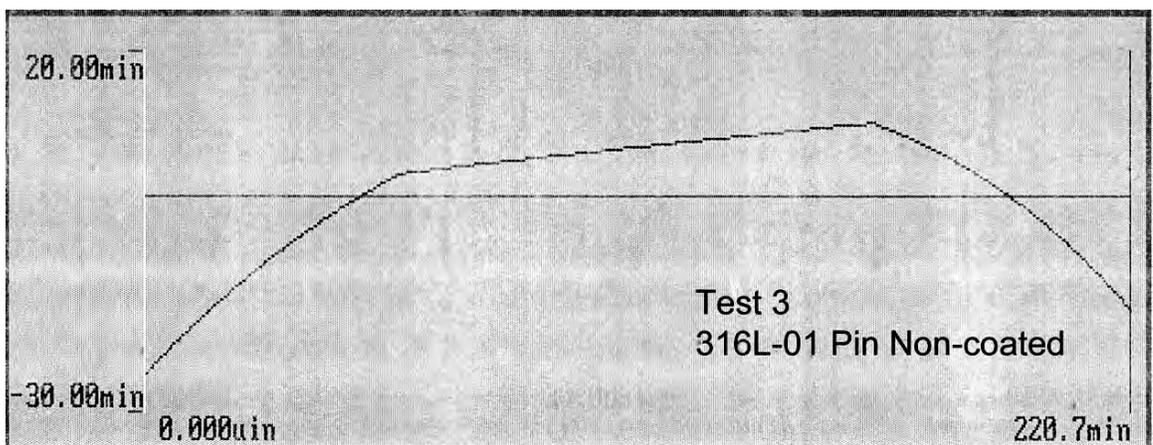
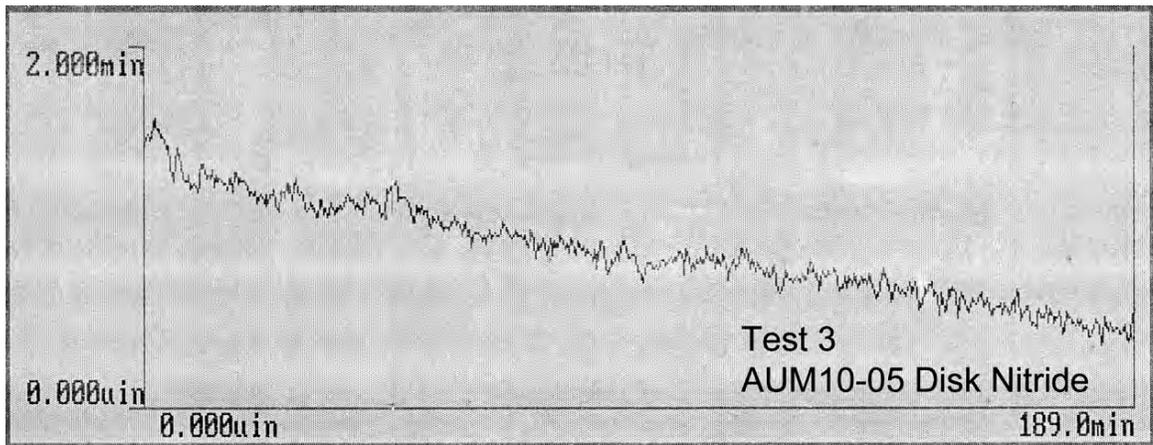


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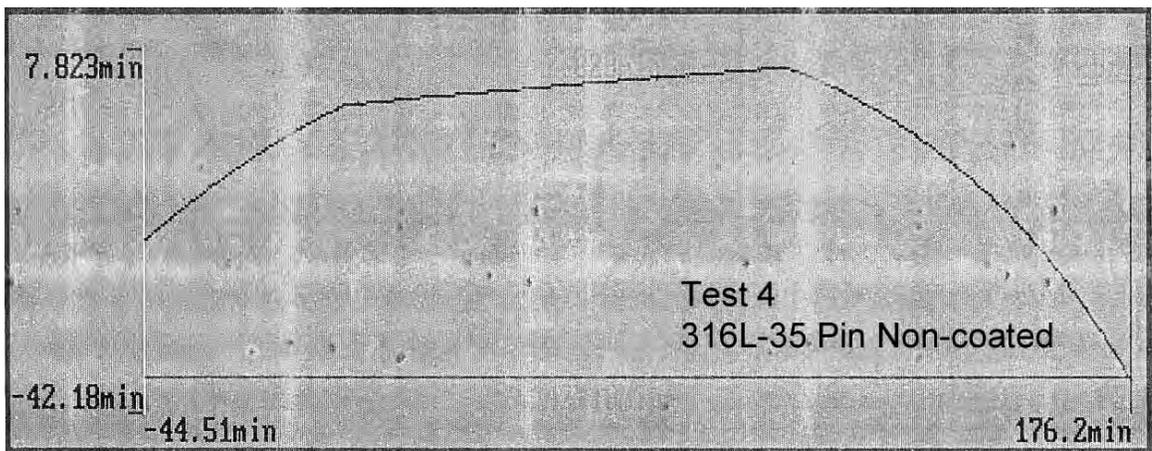
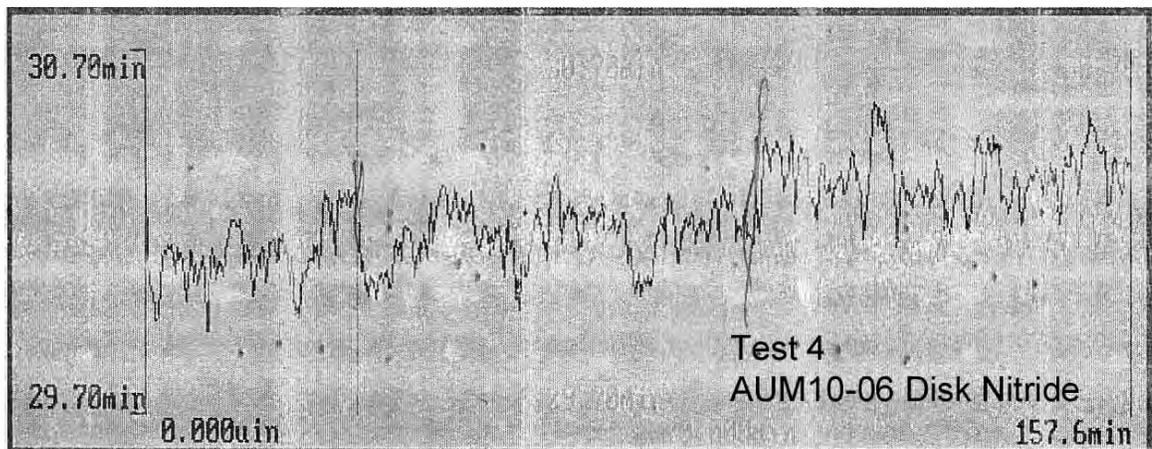
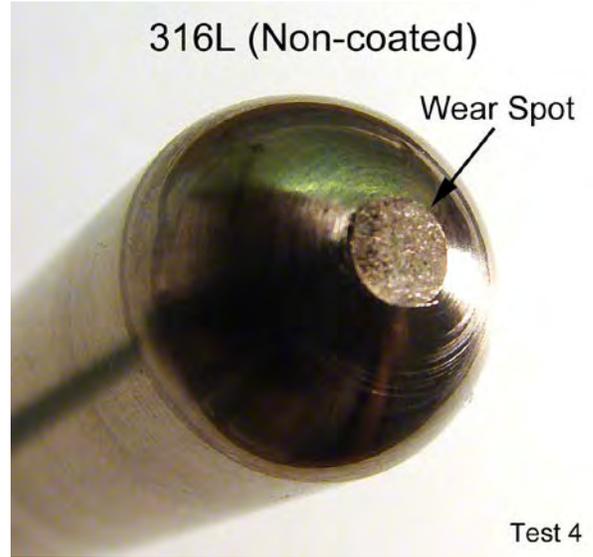
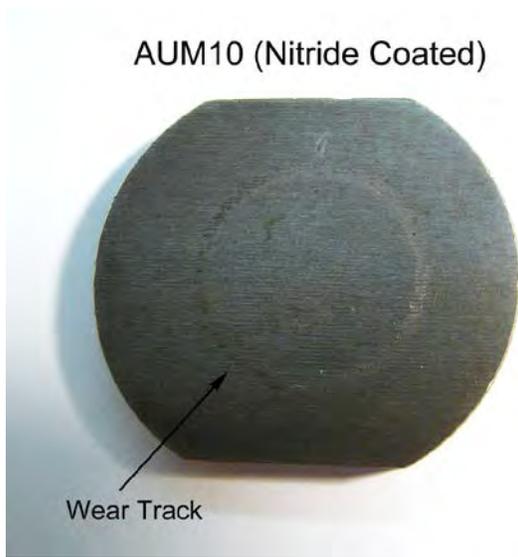
AUM10 (Nitride Coated)



316L (Non-coated)



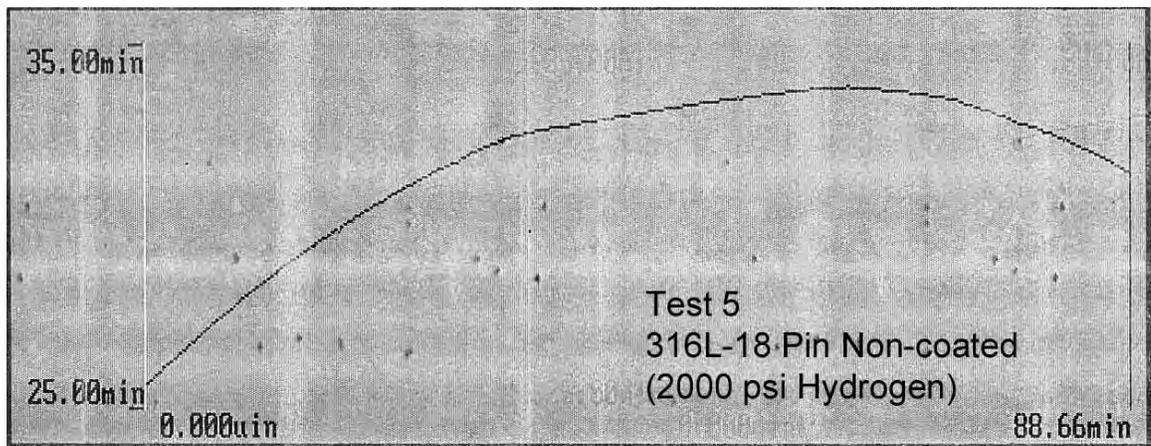
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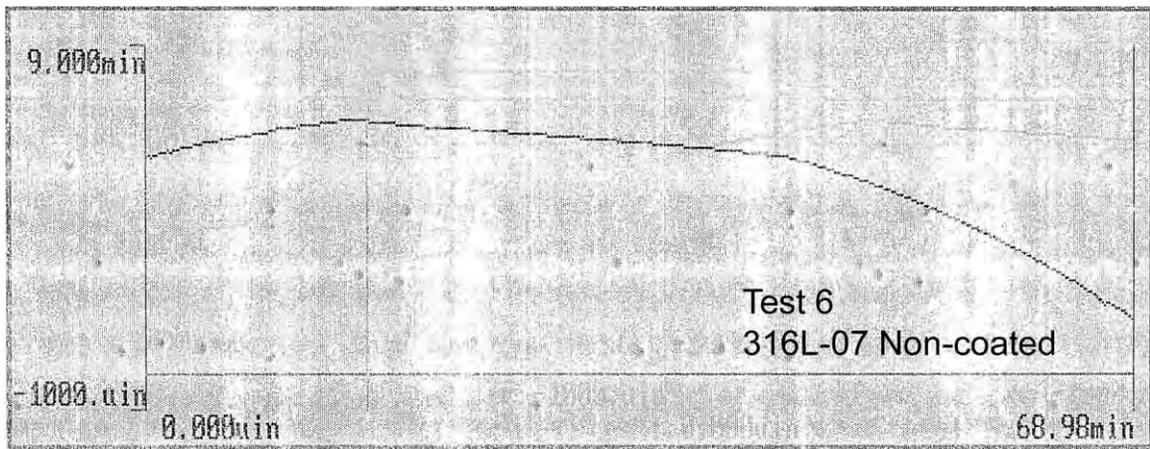
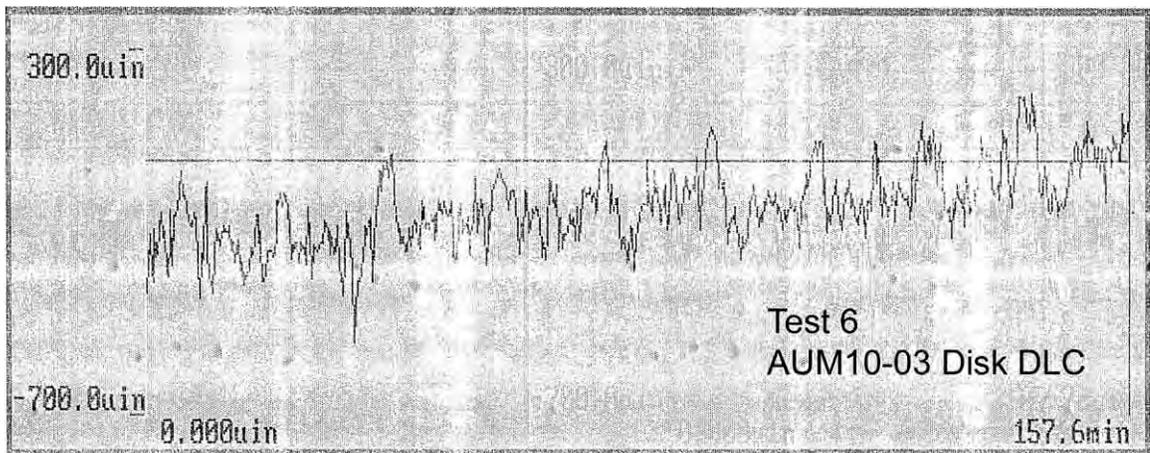
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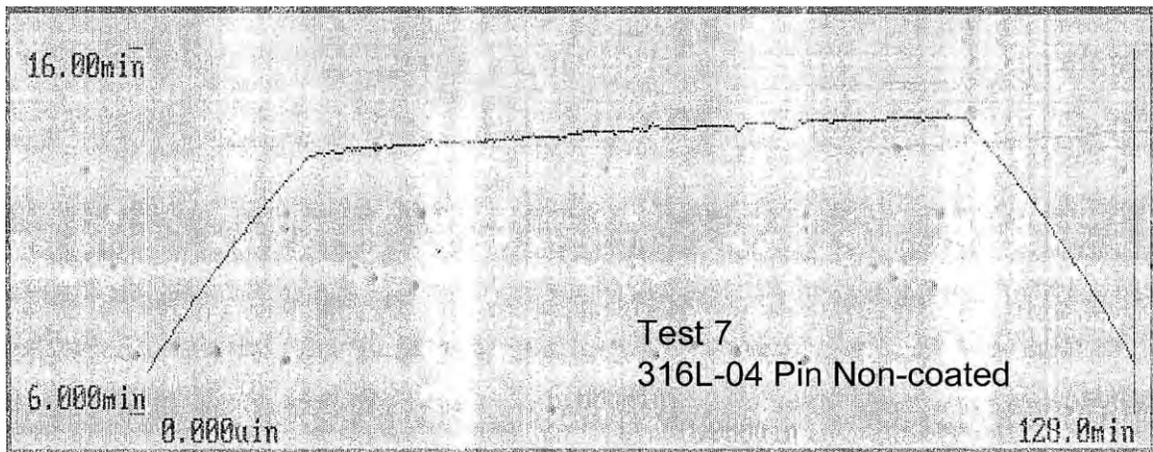
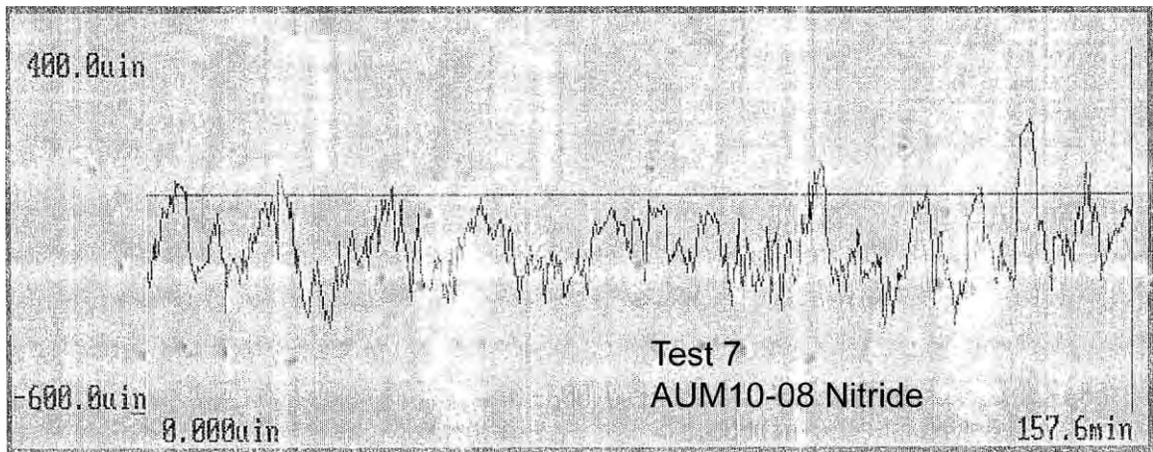
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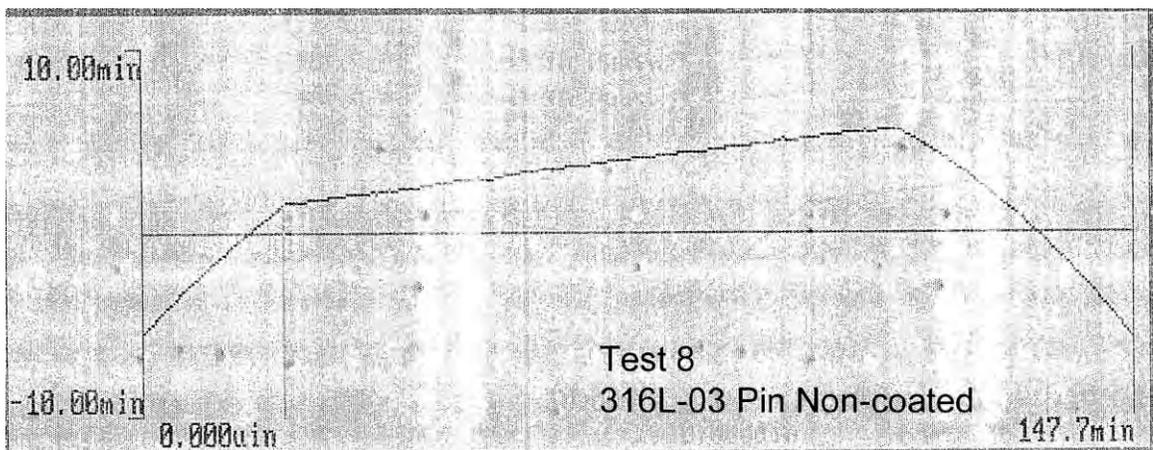
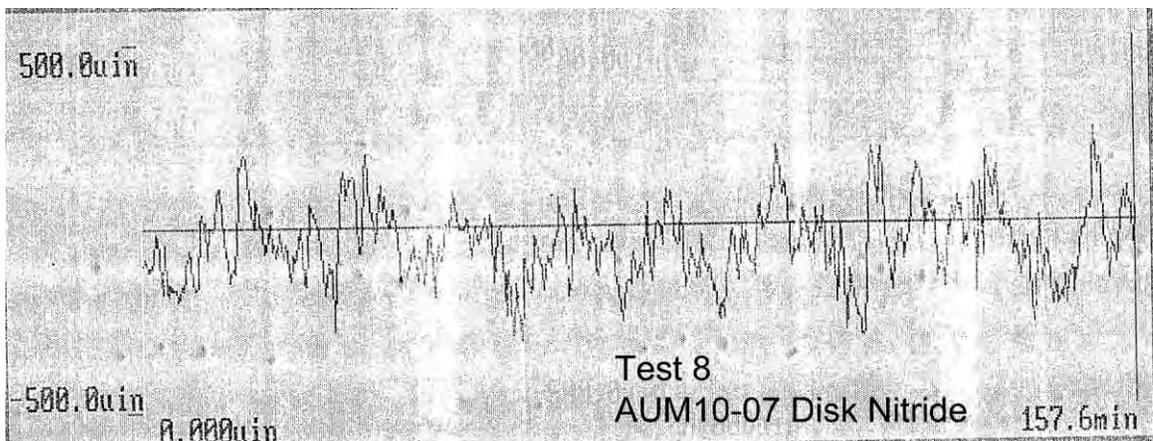
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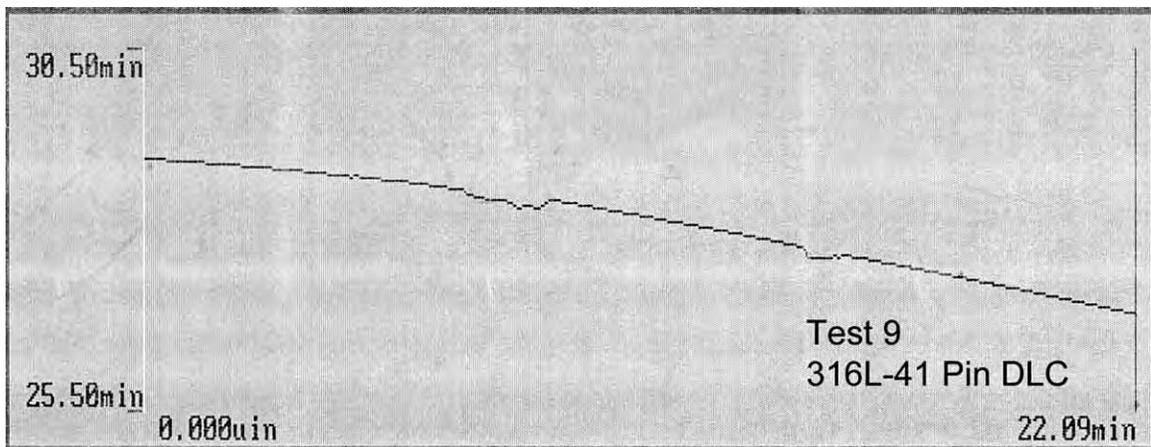
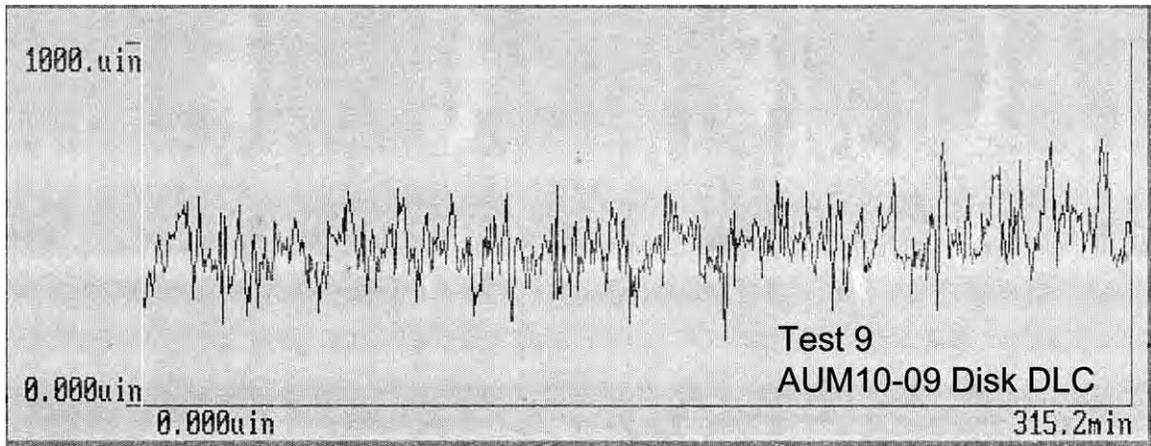
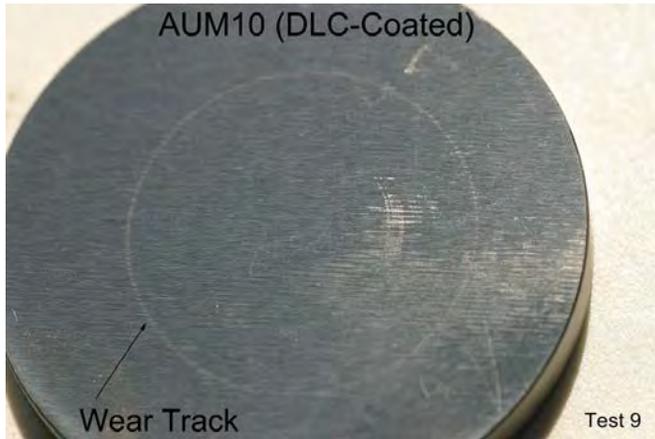
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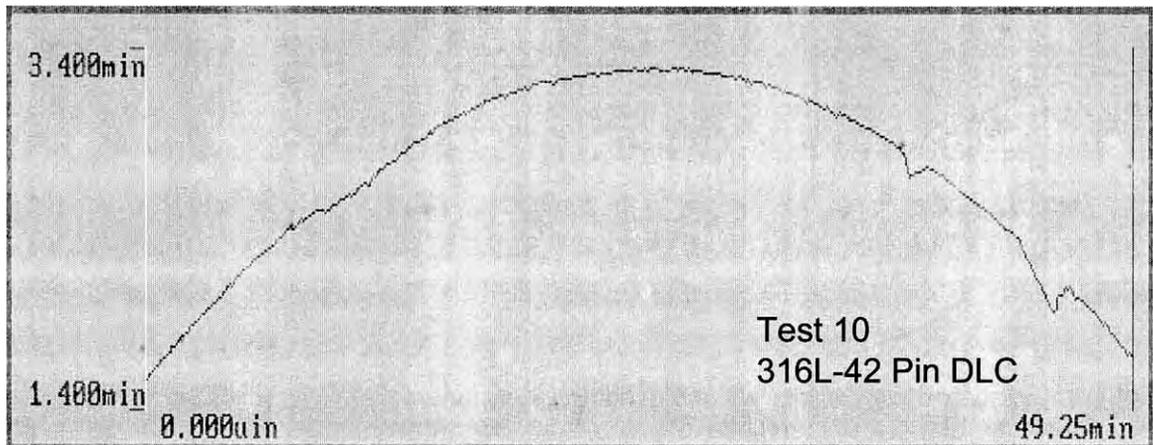
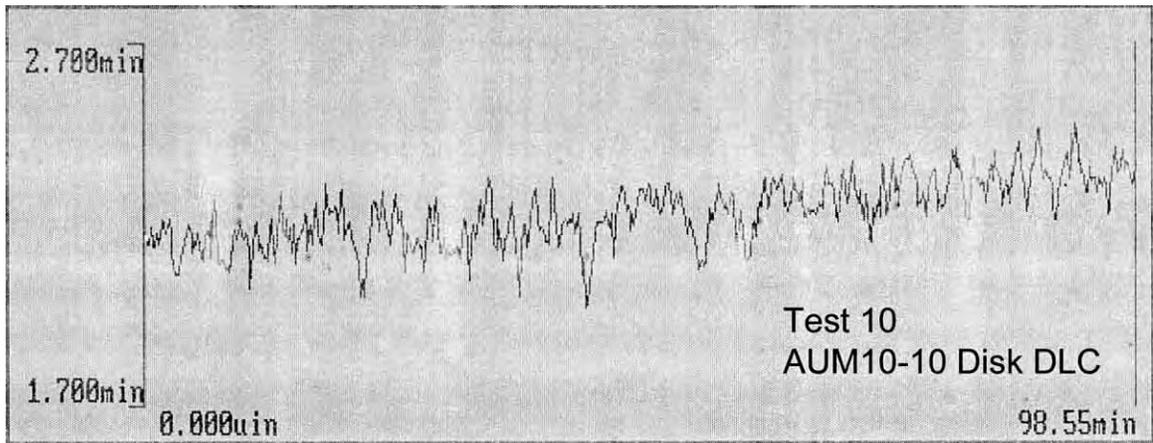
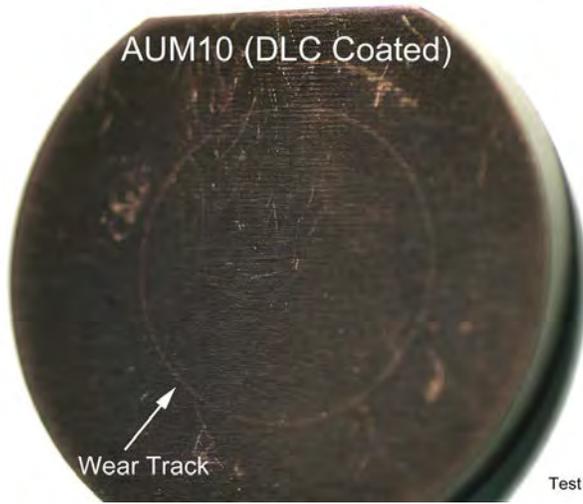
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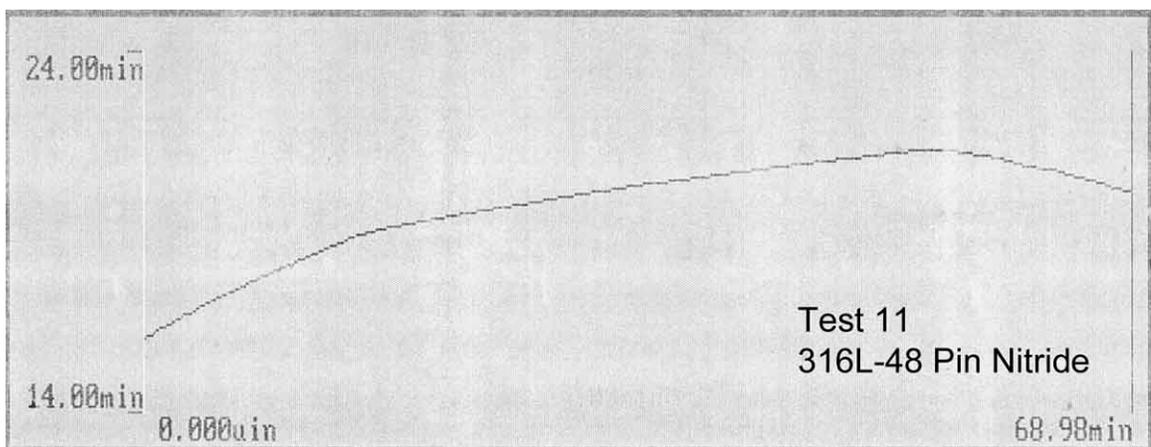
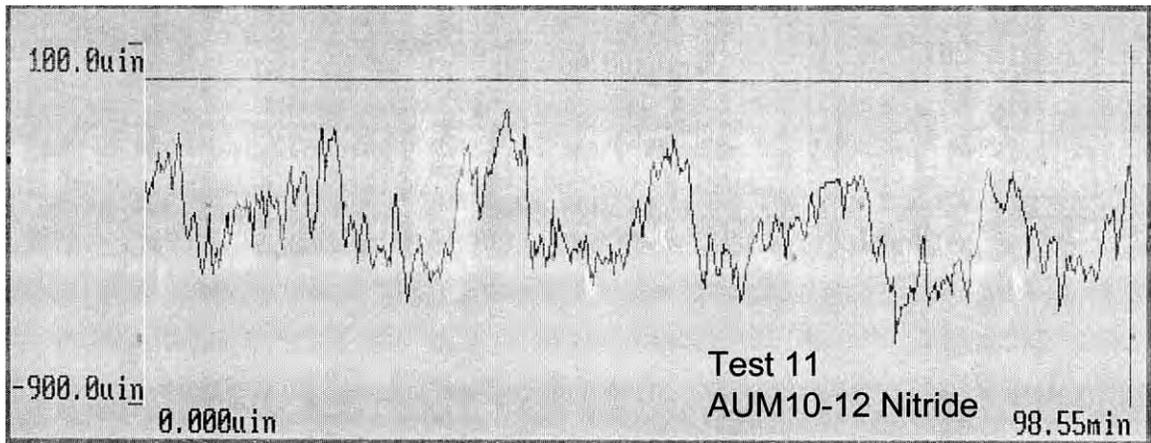
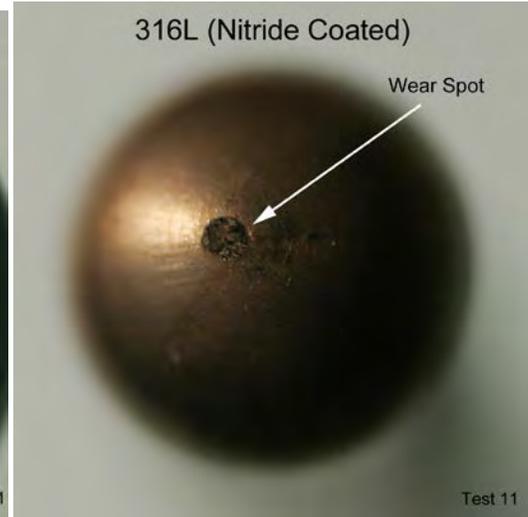
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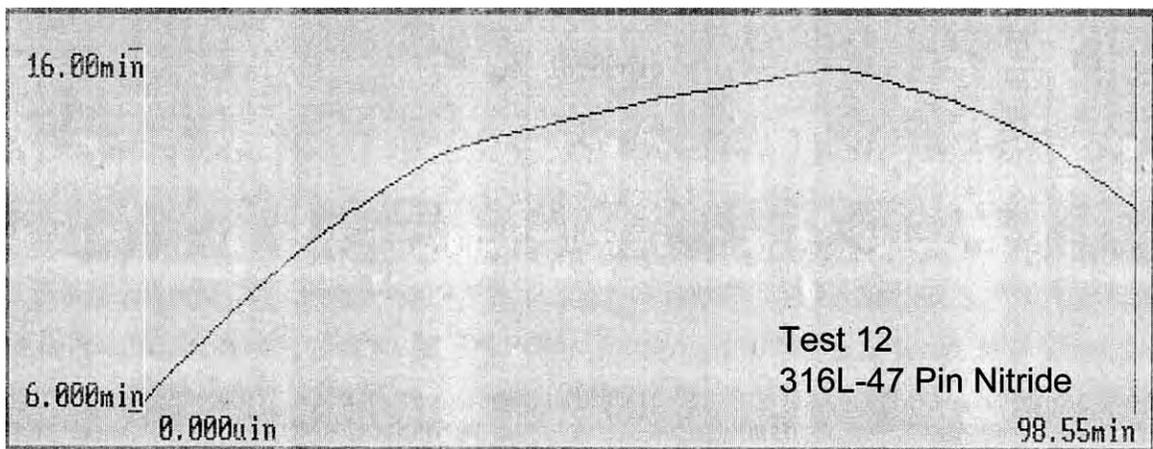
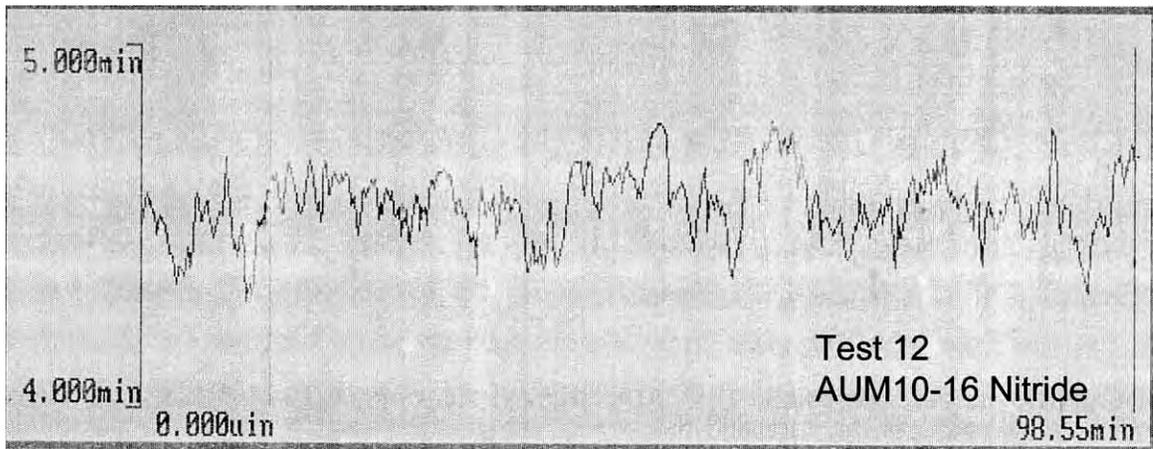
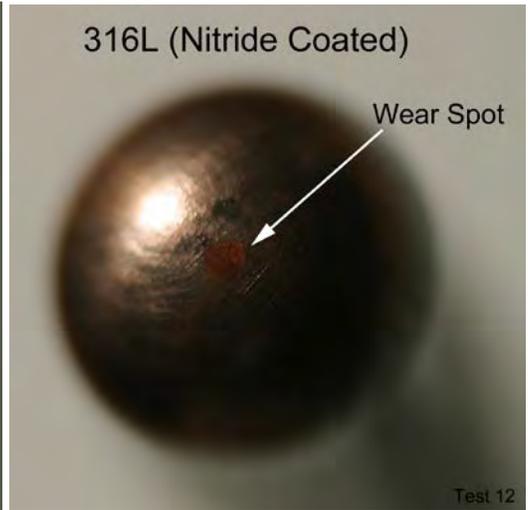
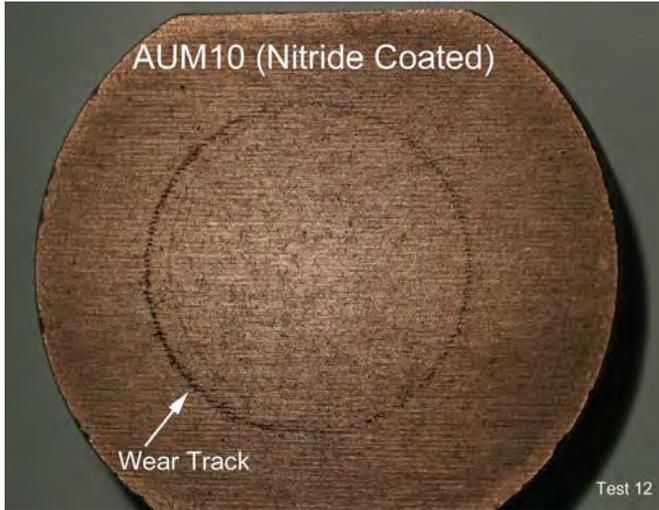
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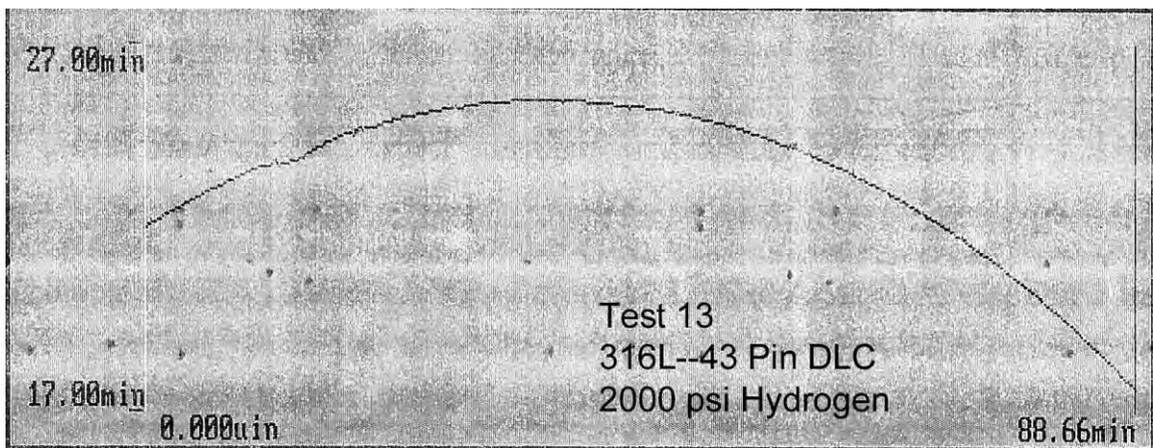
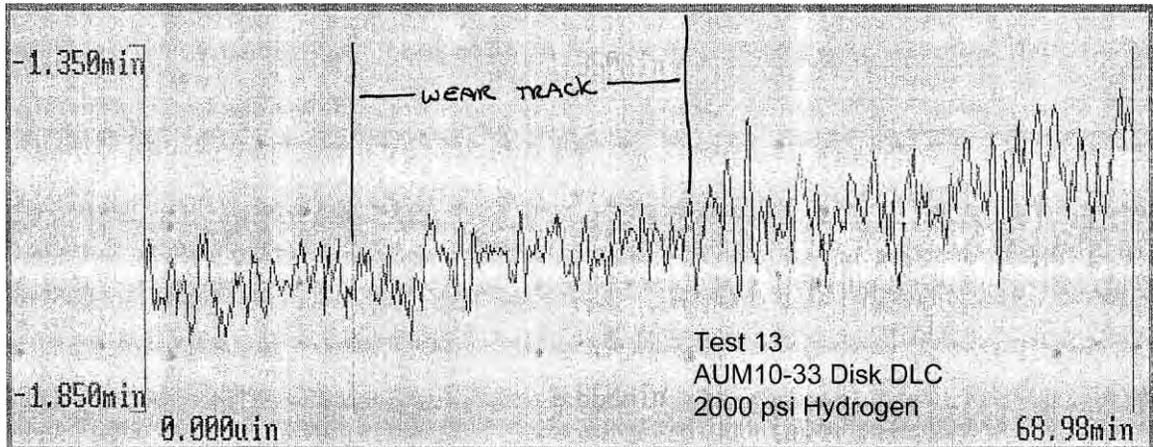
Test 11



Test 12

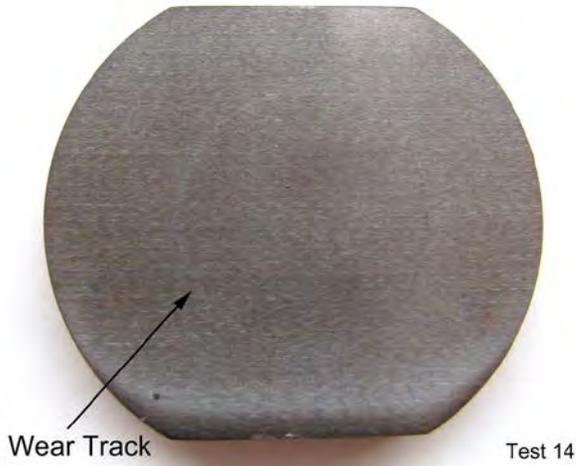


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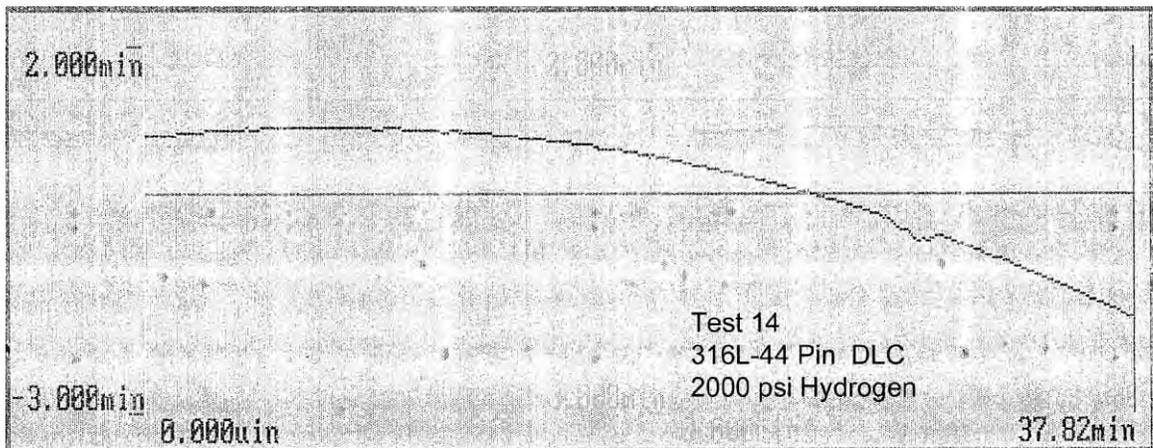
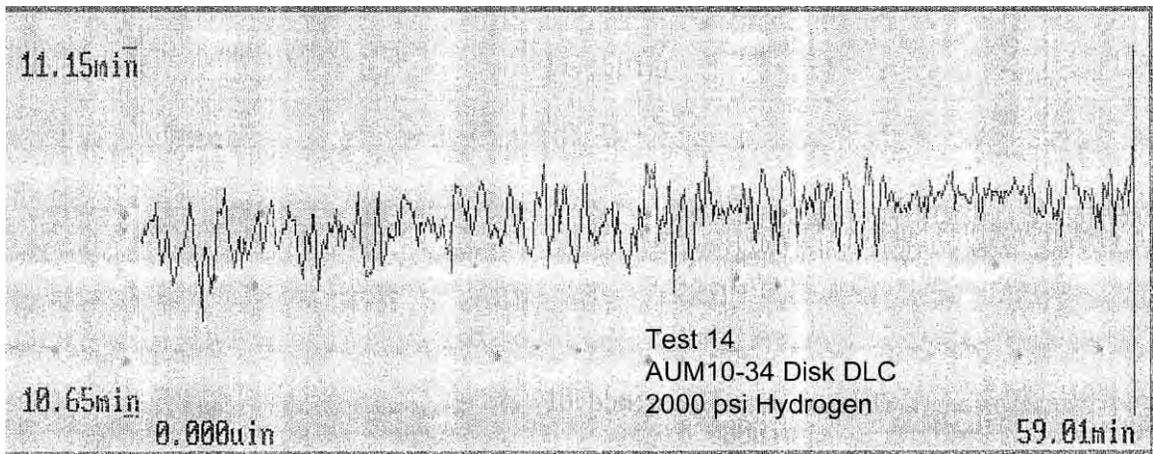


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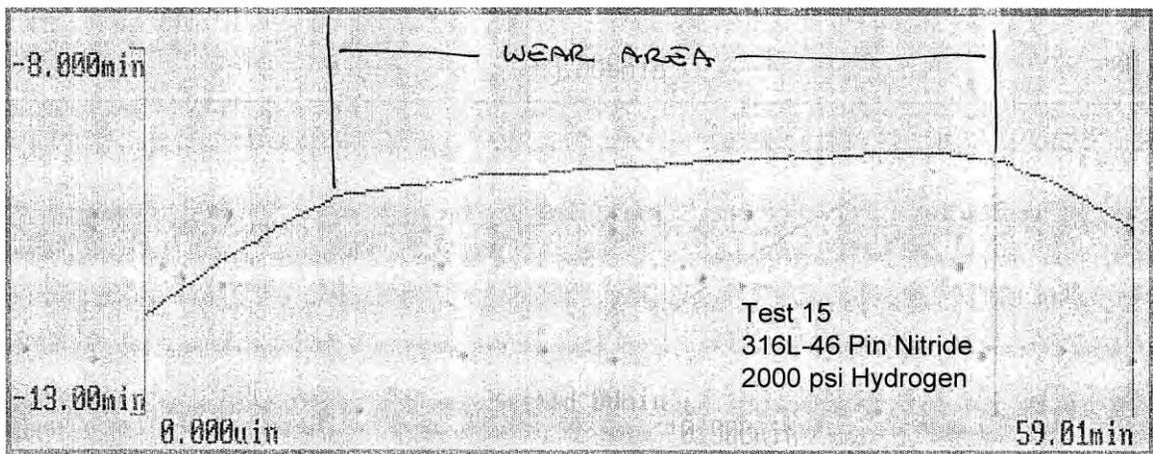
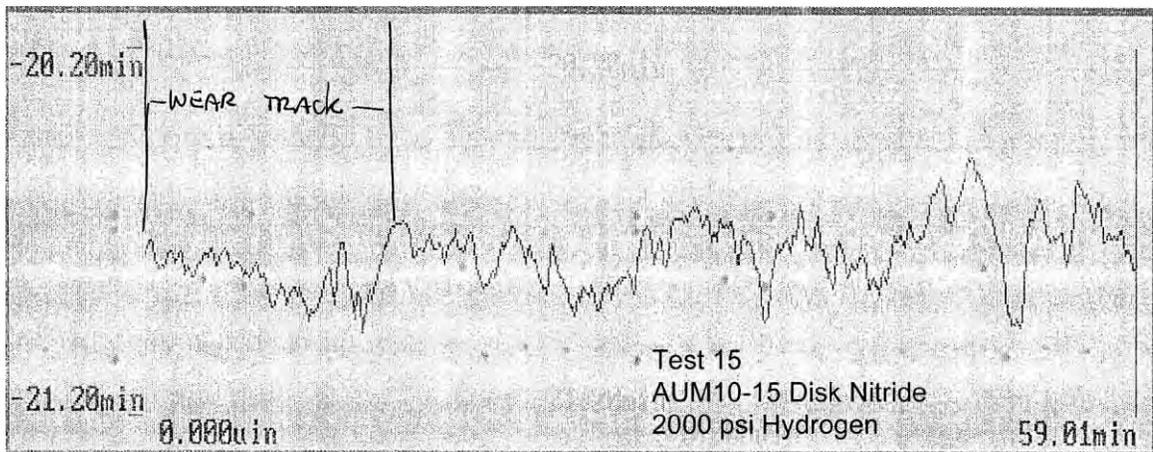
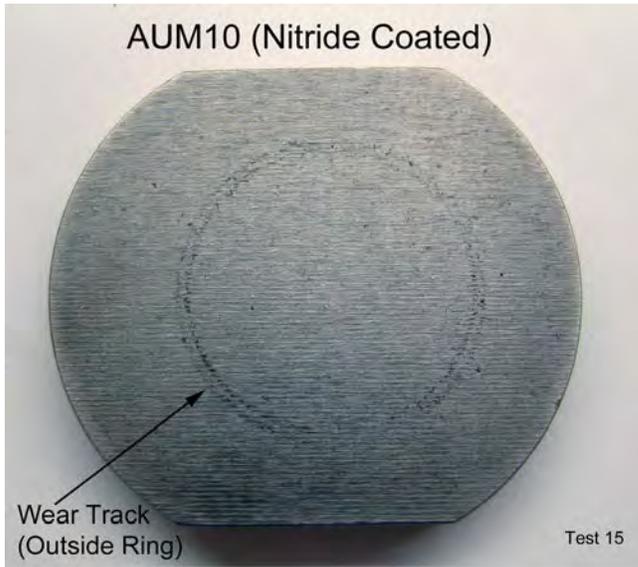
AUM10 (DLC Coated)



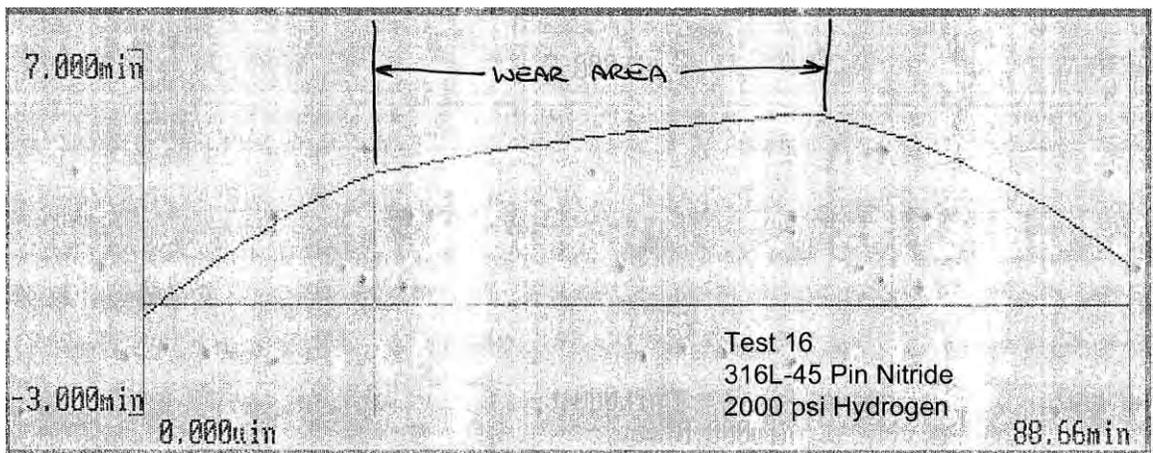
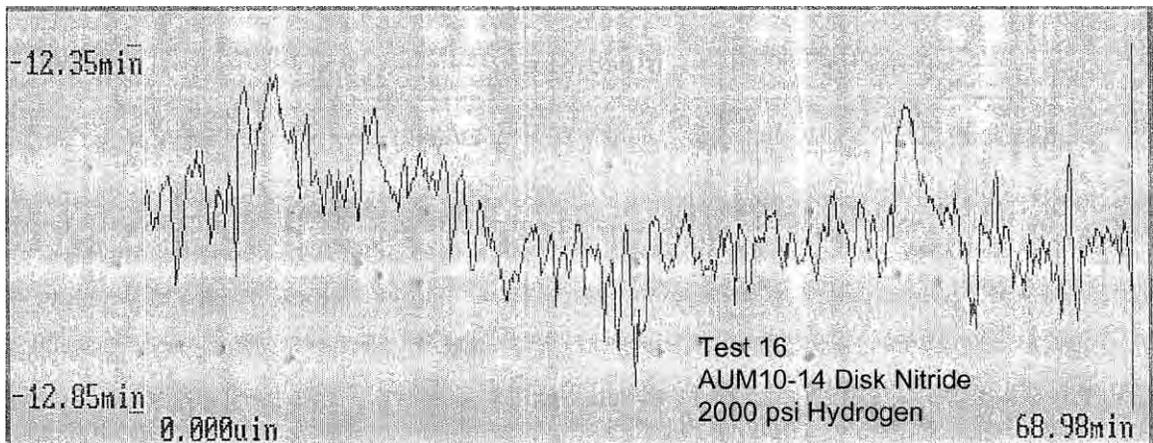
316L (DLC Coated)



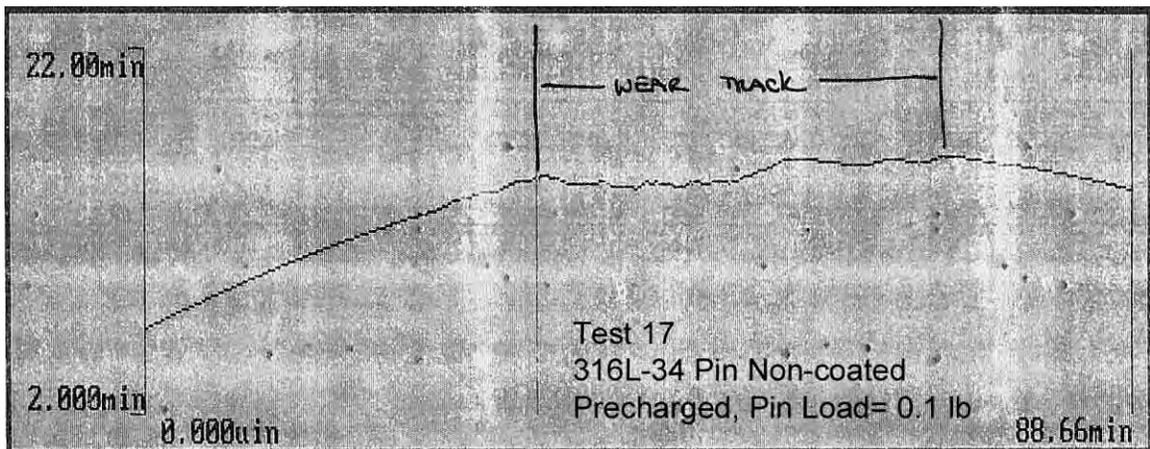
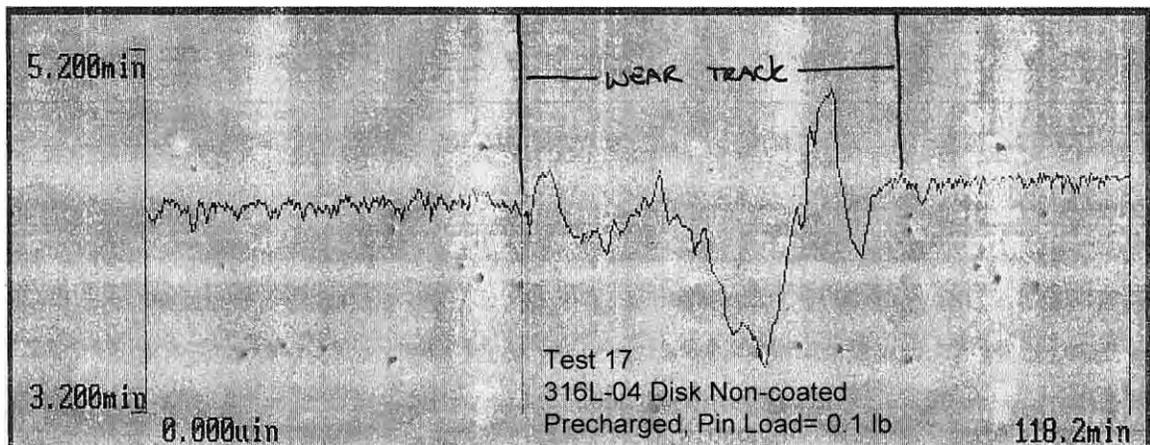
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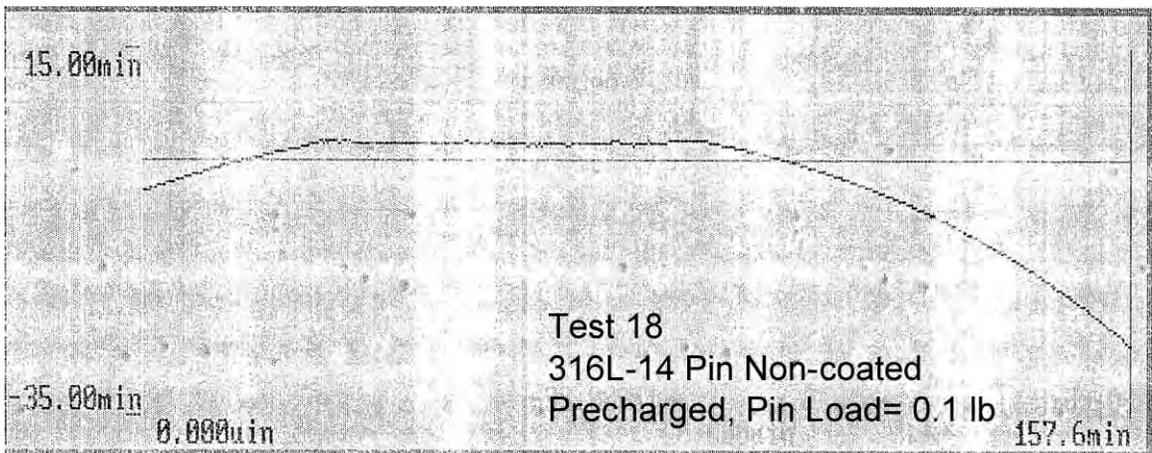
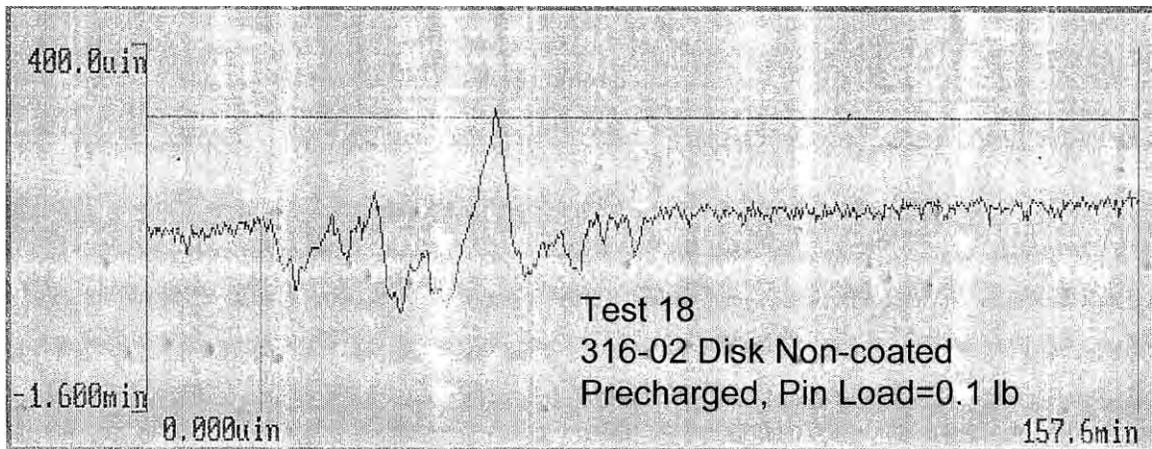
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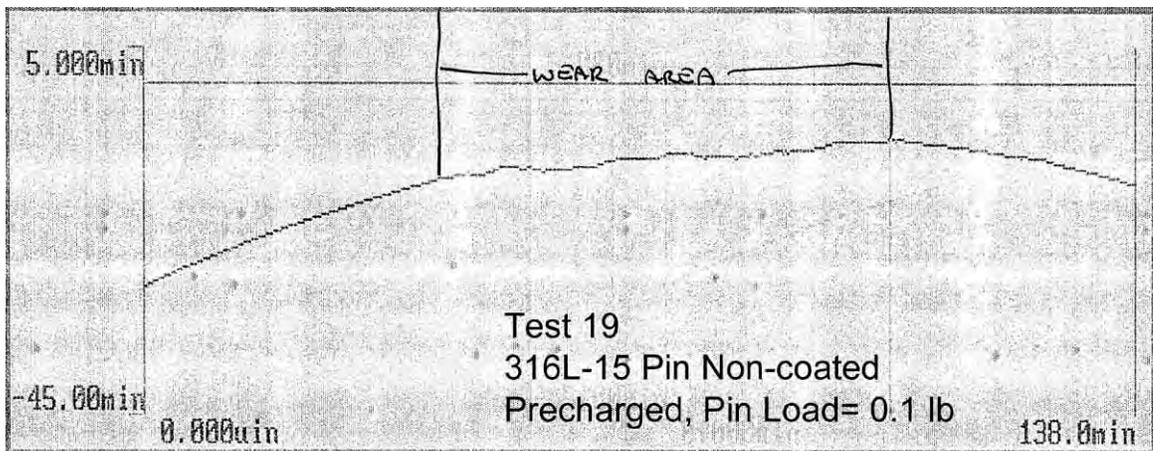
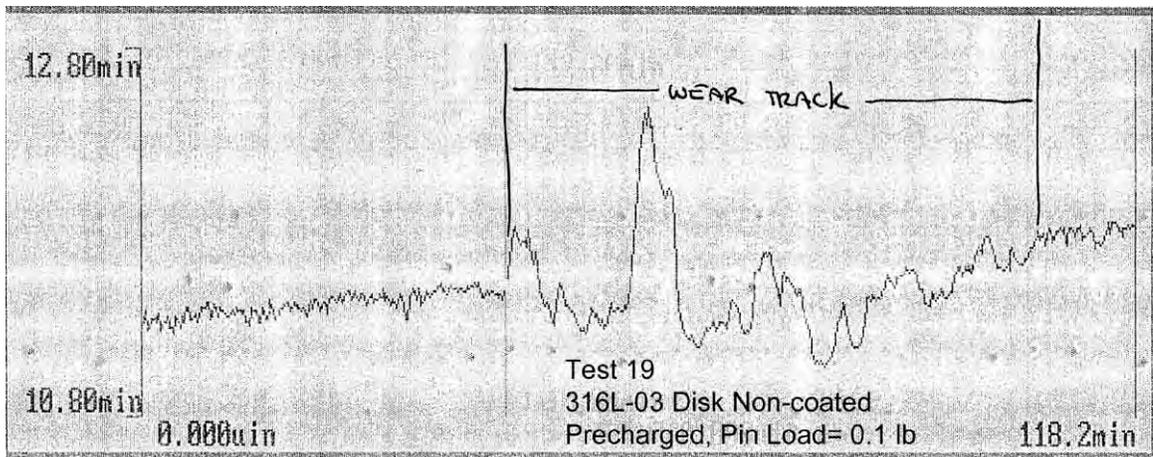
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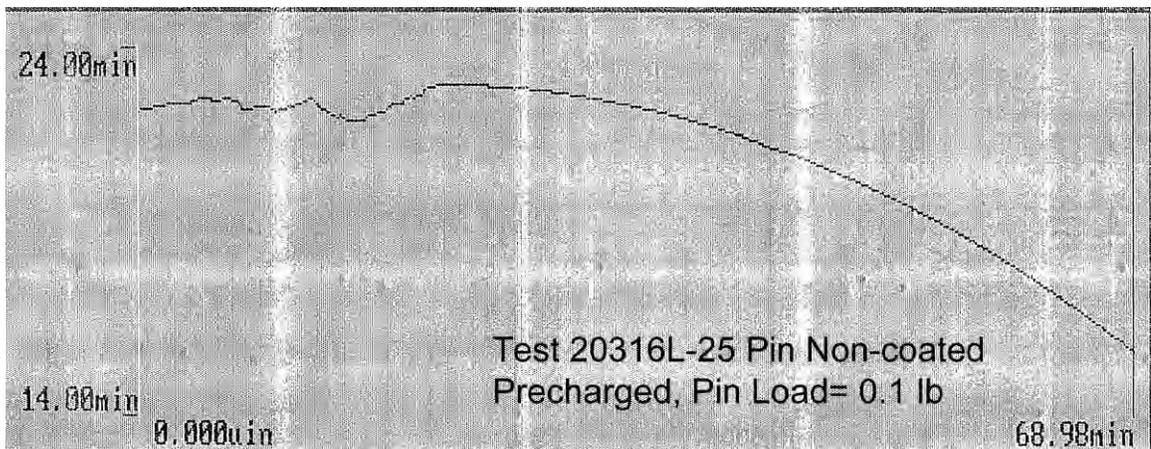
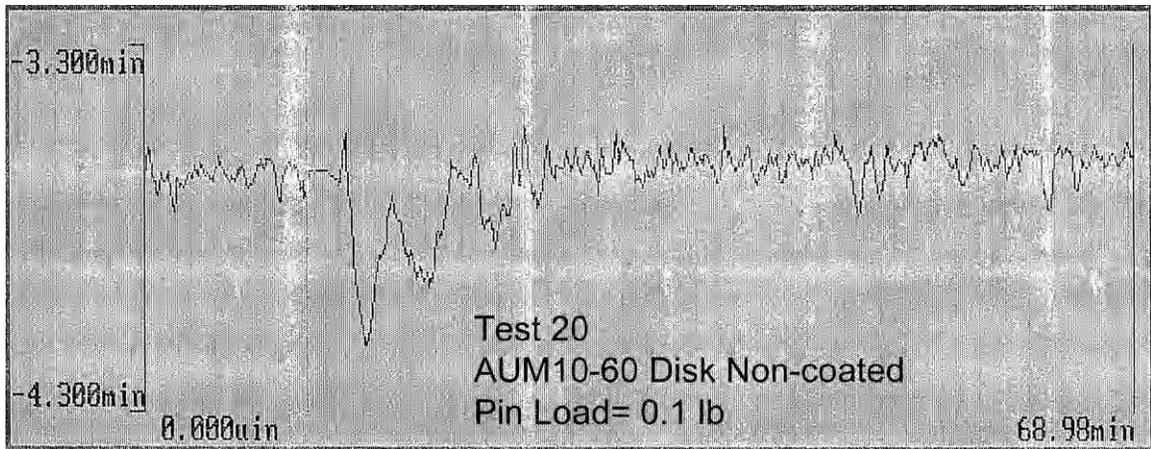
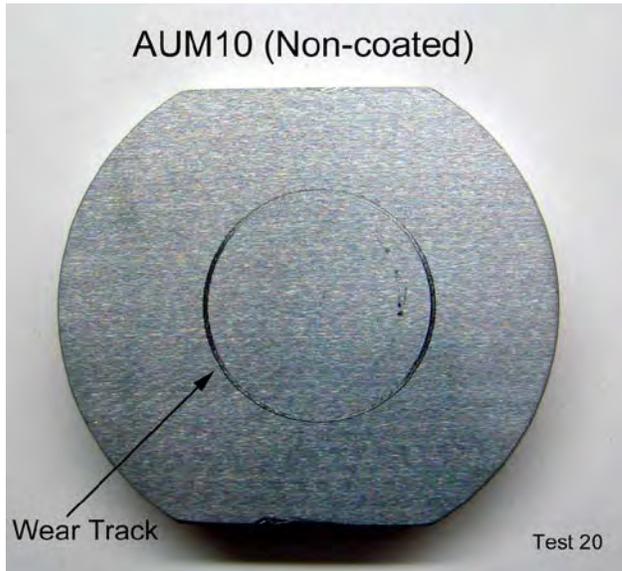
Test 18



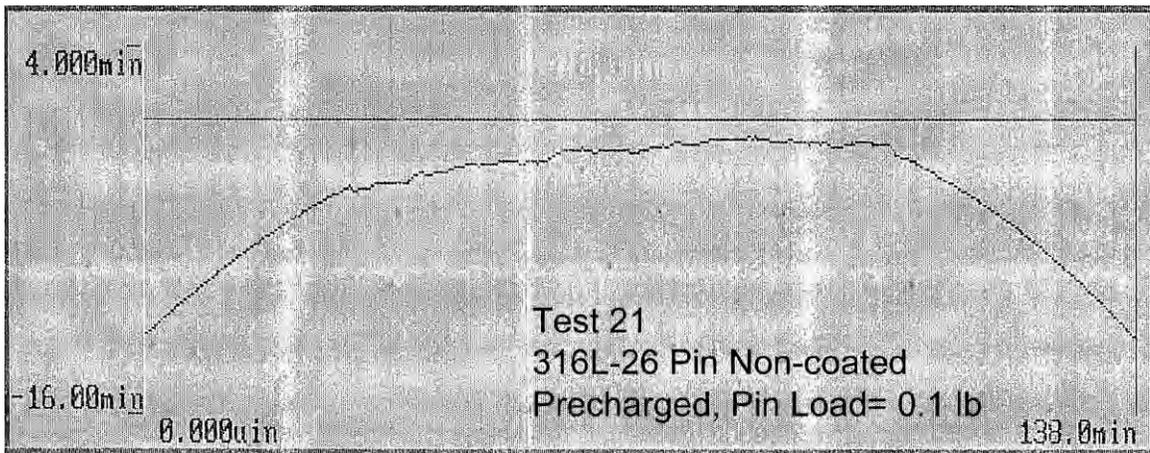
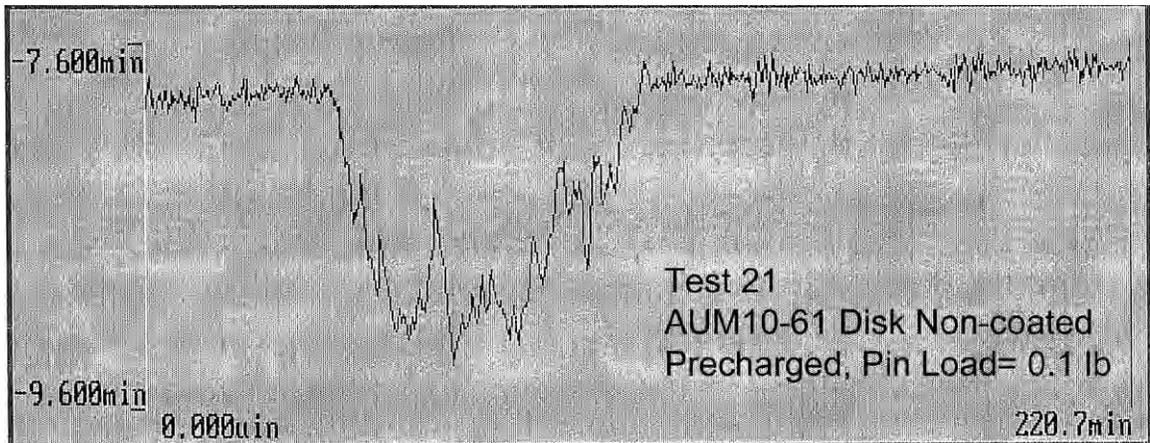
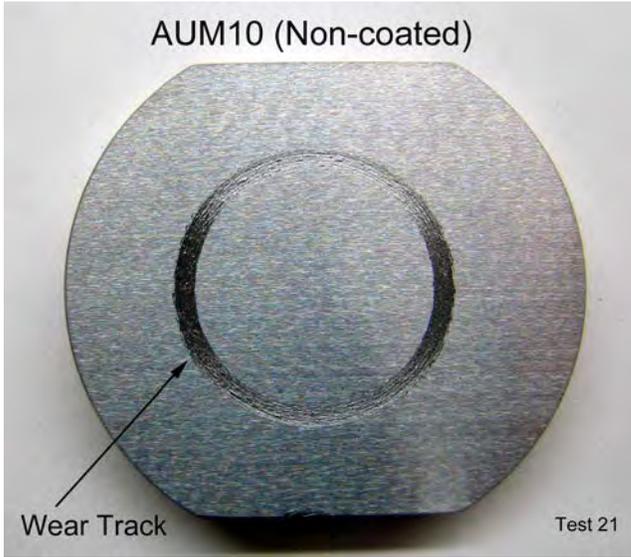
Test 19



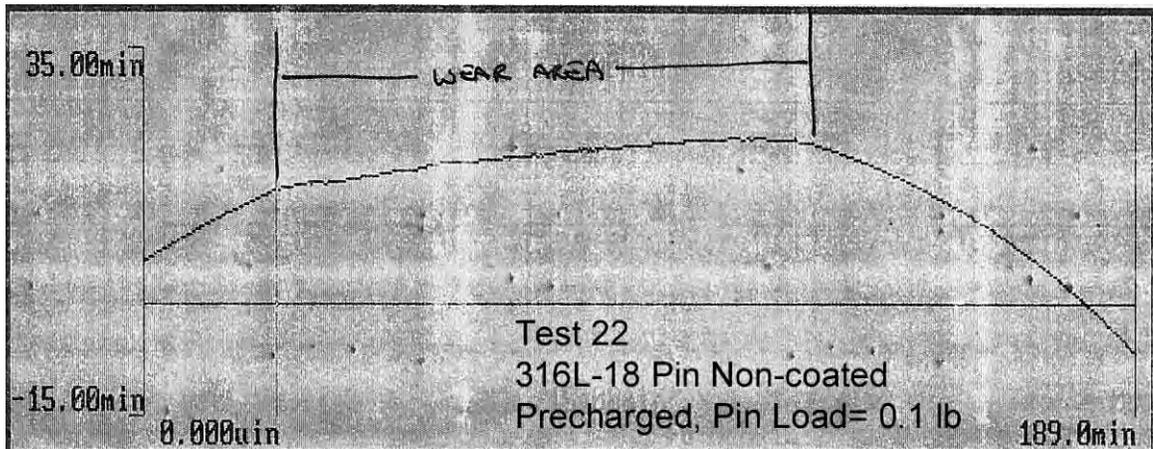
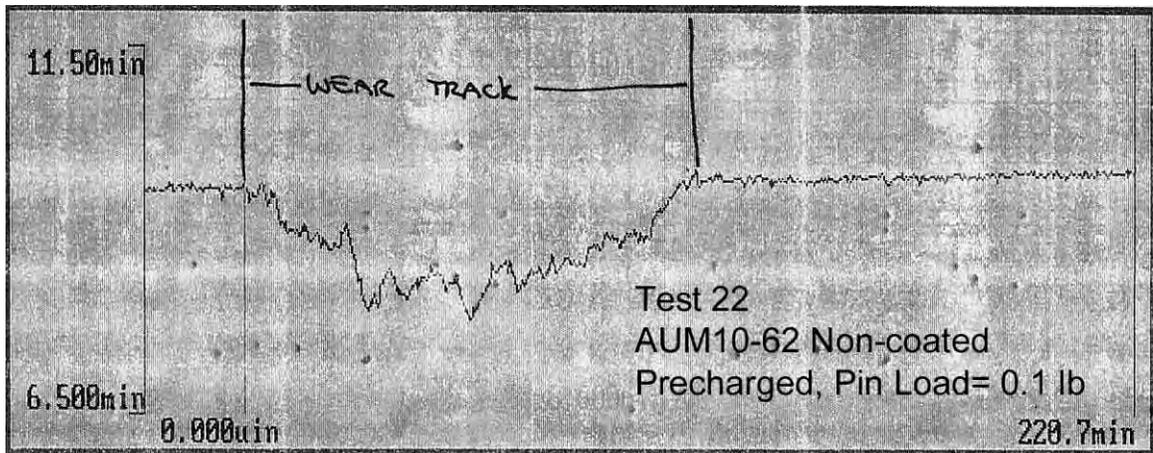
Test 20



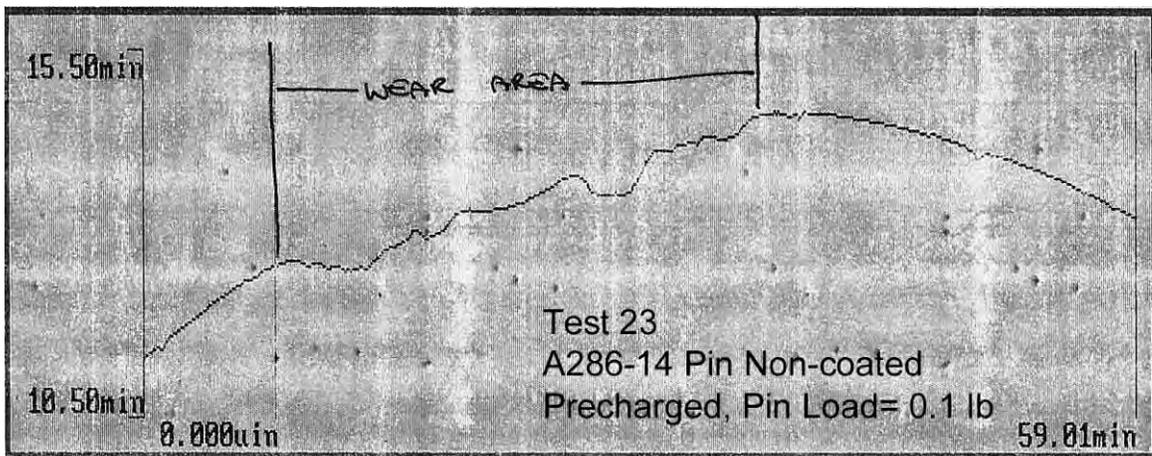
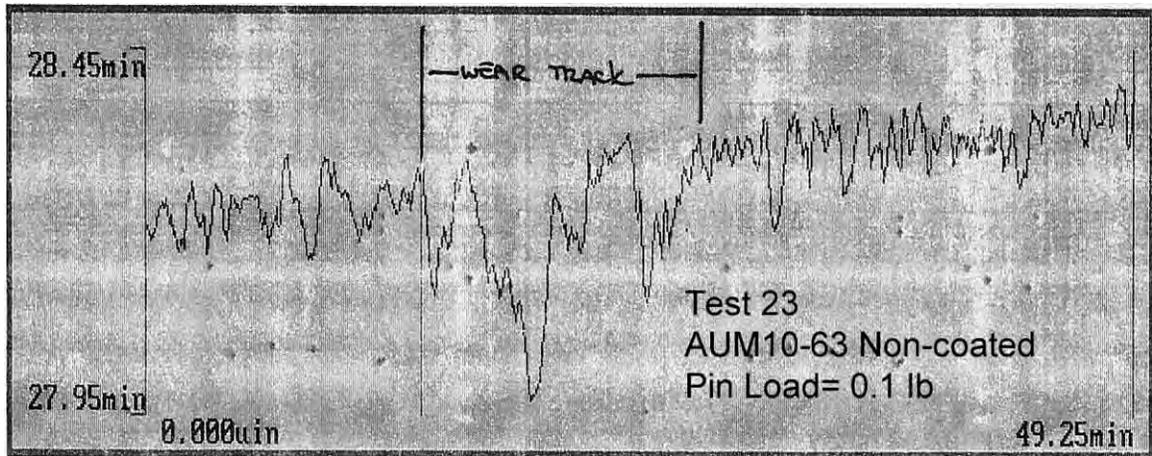
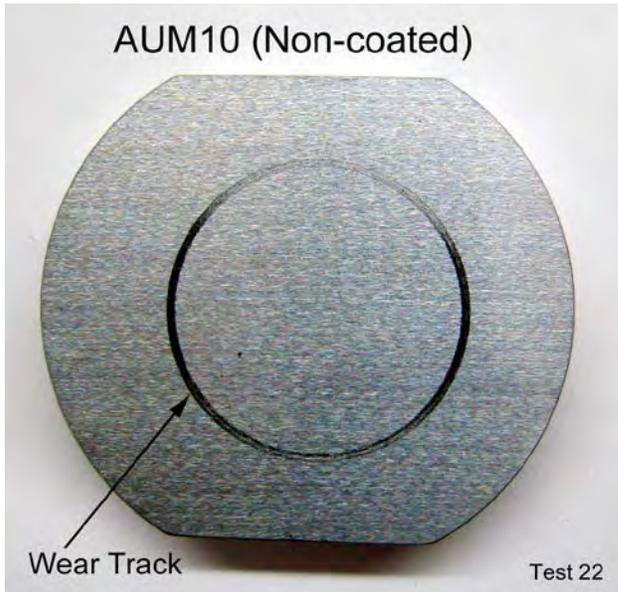
Test 21



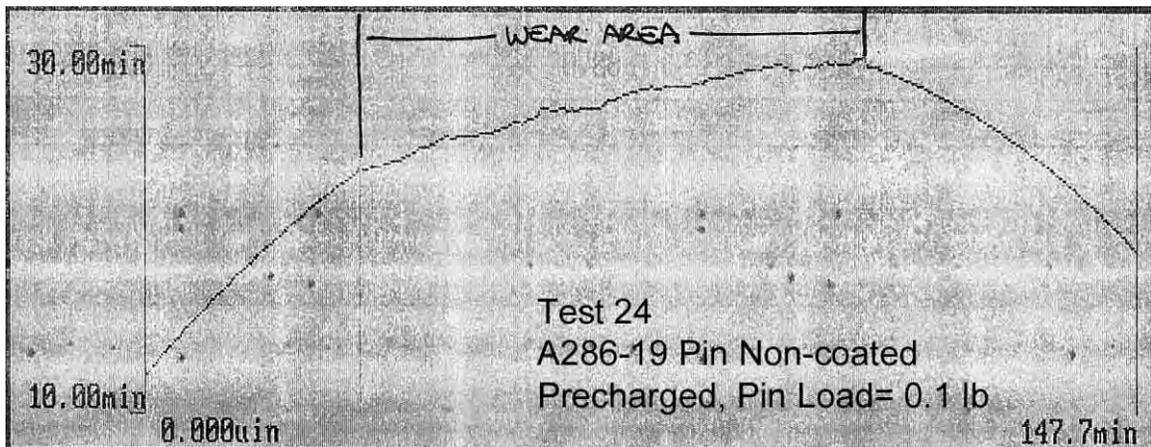
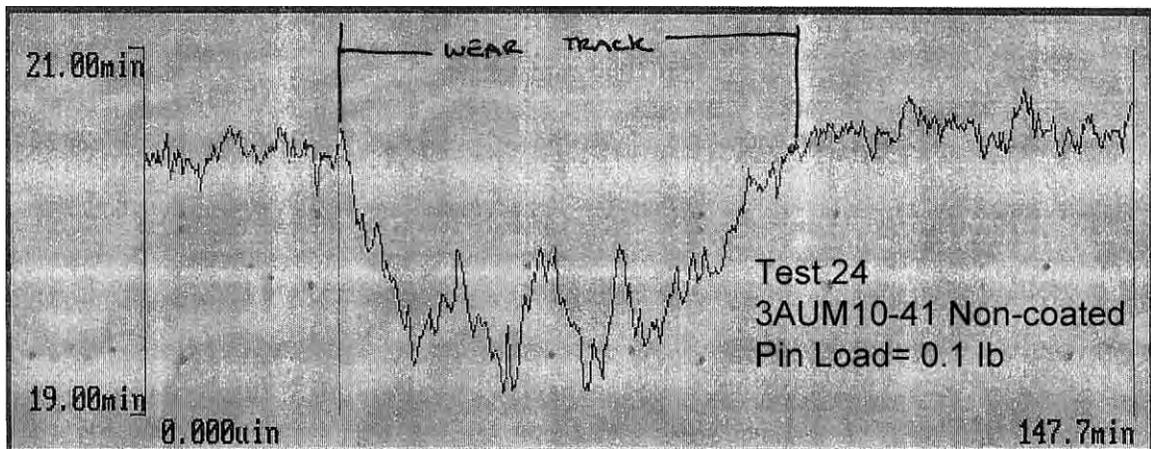
Test 22



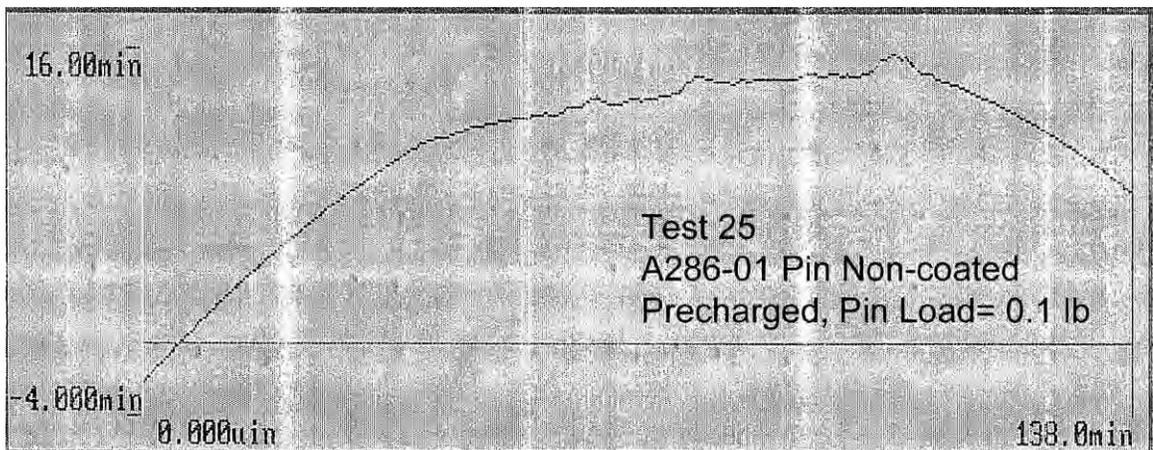
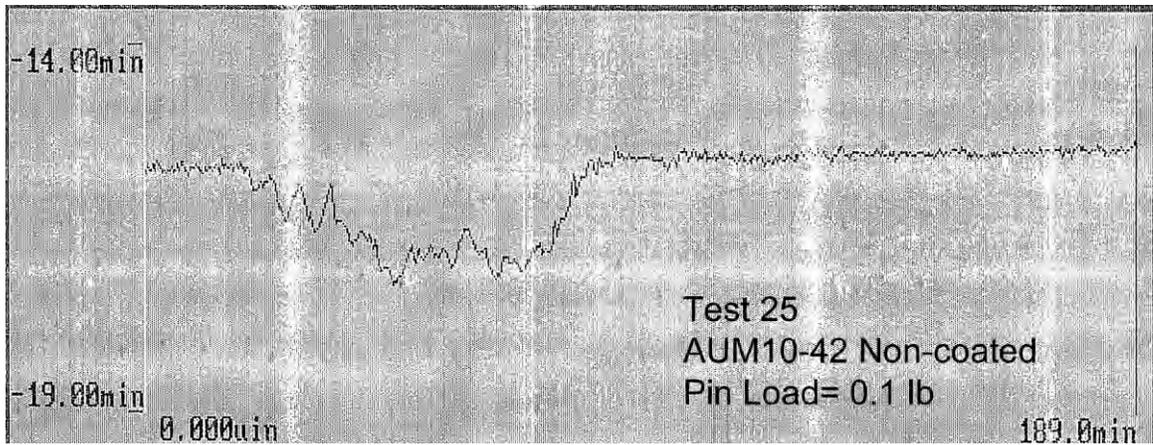
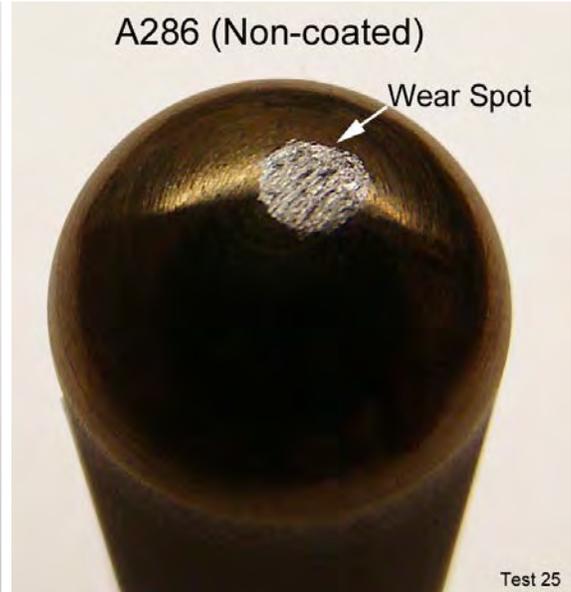
Test 23



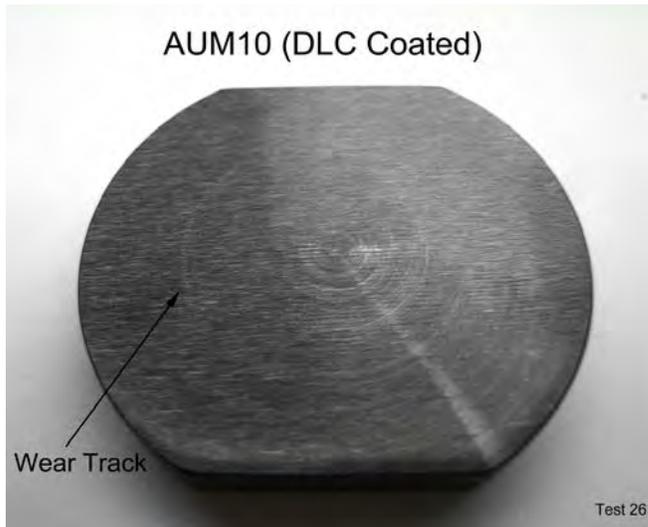
Test 24



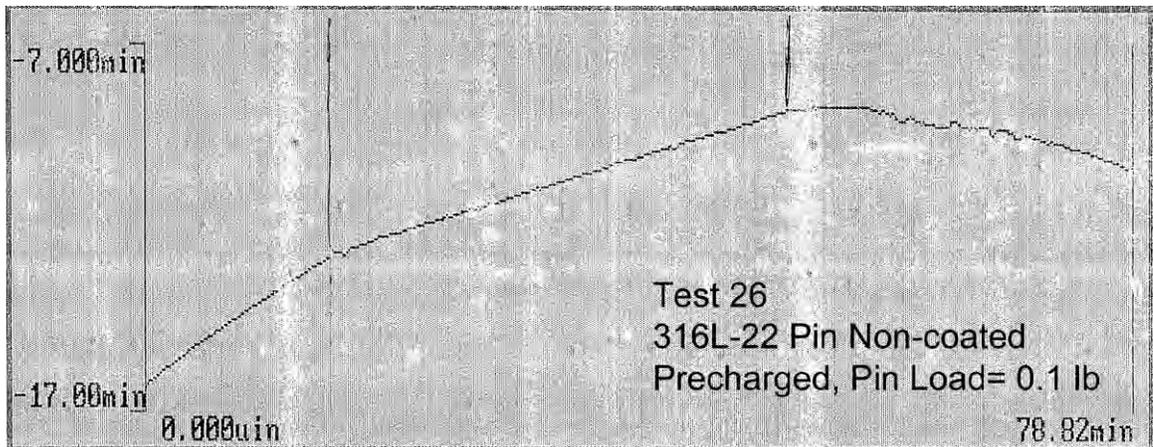
Test 25



Test 26



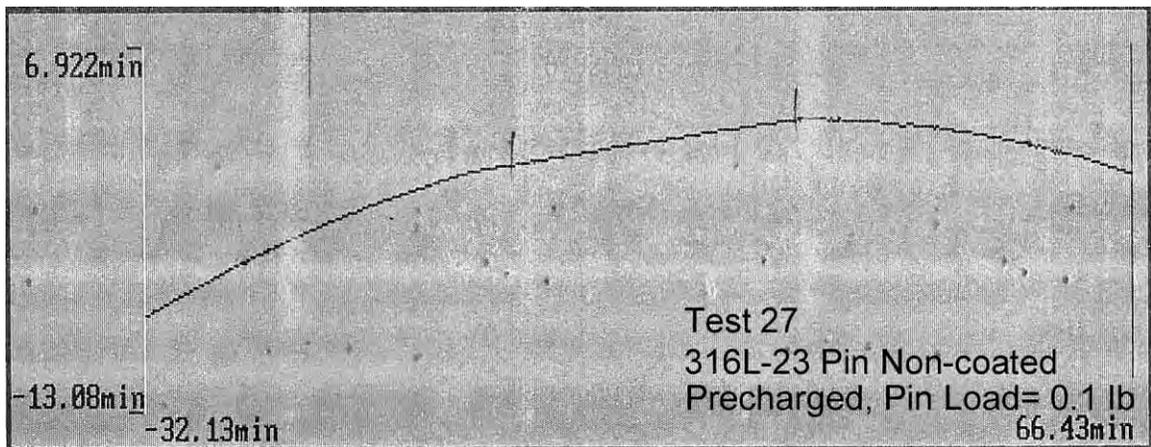
(Wear track on disk was not measurable.)



Test 27

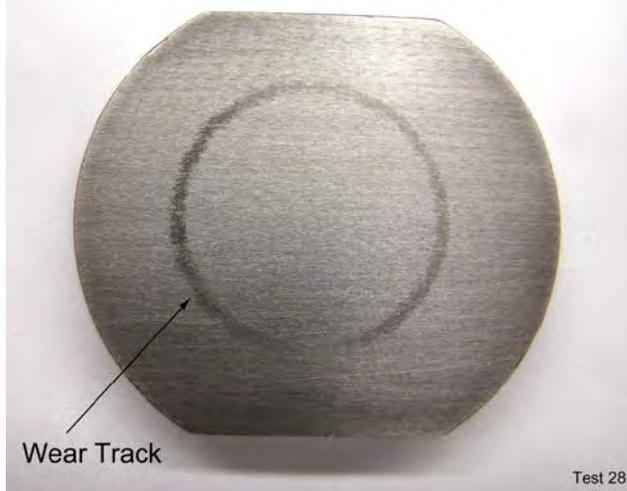


(Wear track on disk was not measurable.)

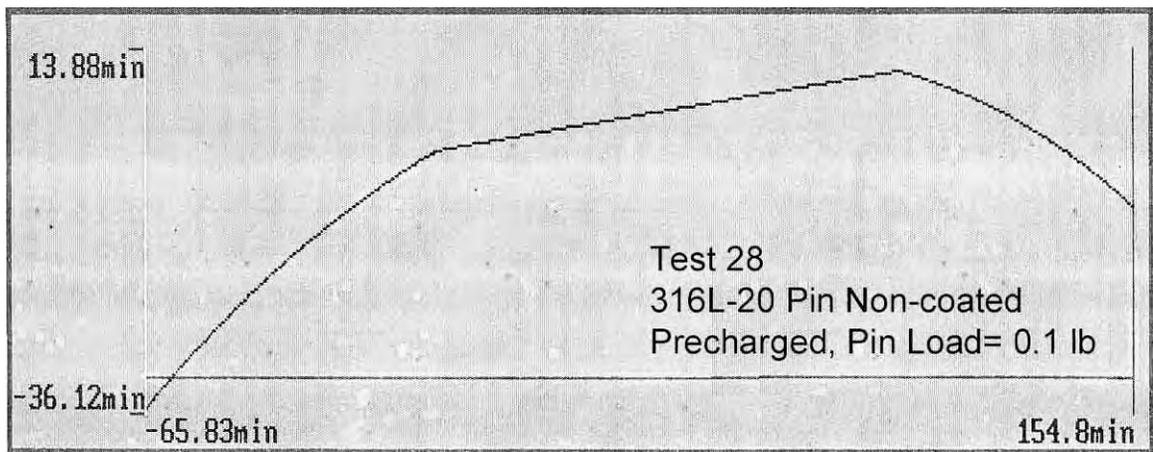
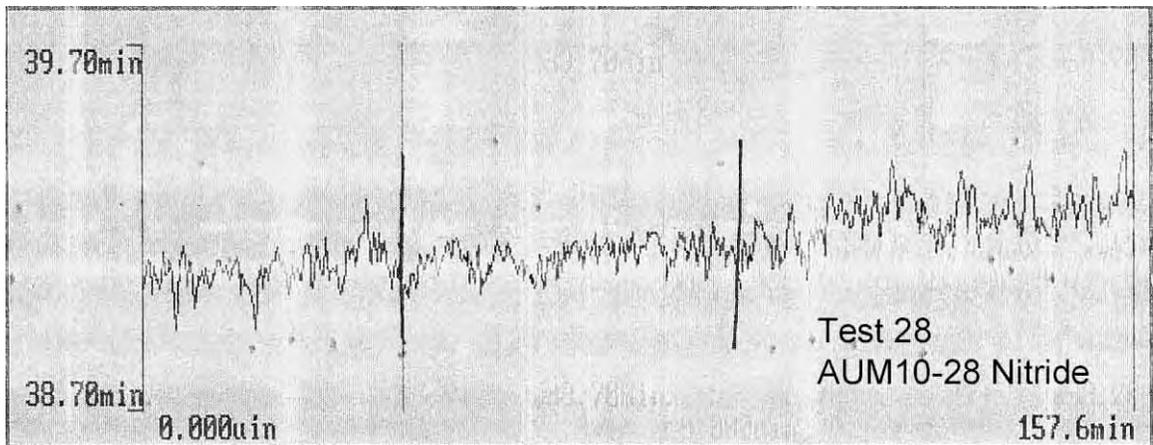


Test 28

AUM10 (Nitride Coated)



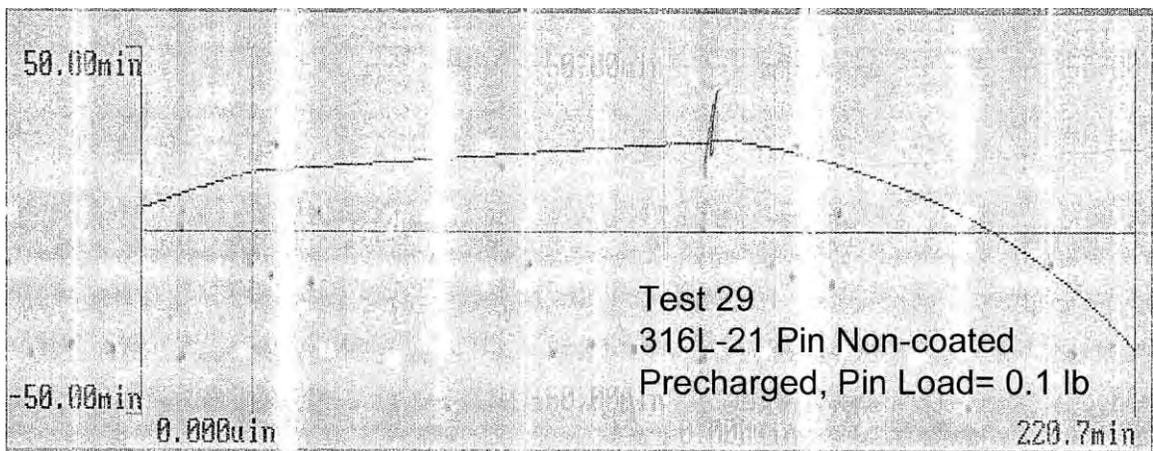
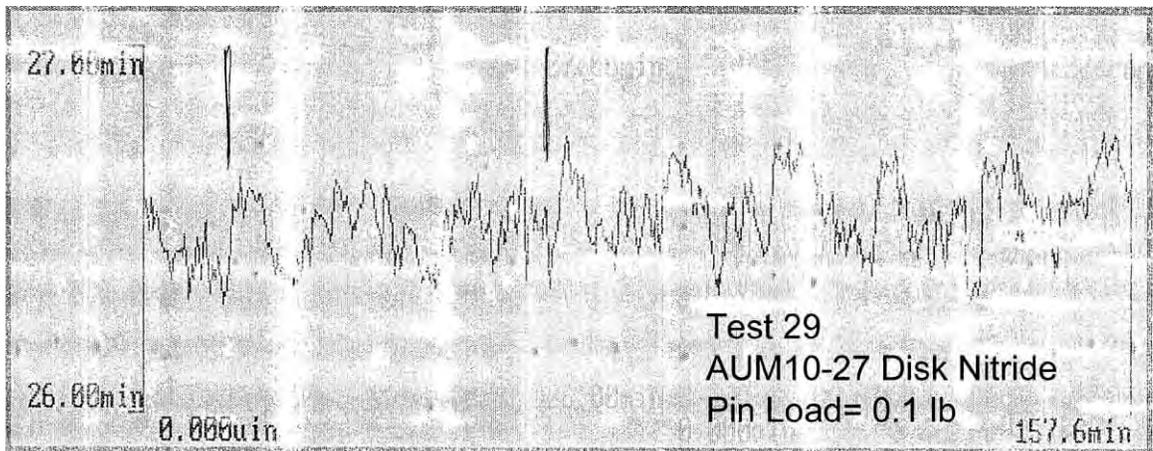
316L (Non-coated, Precharged)



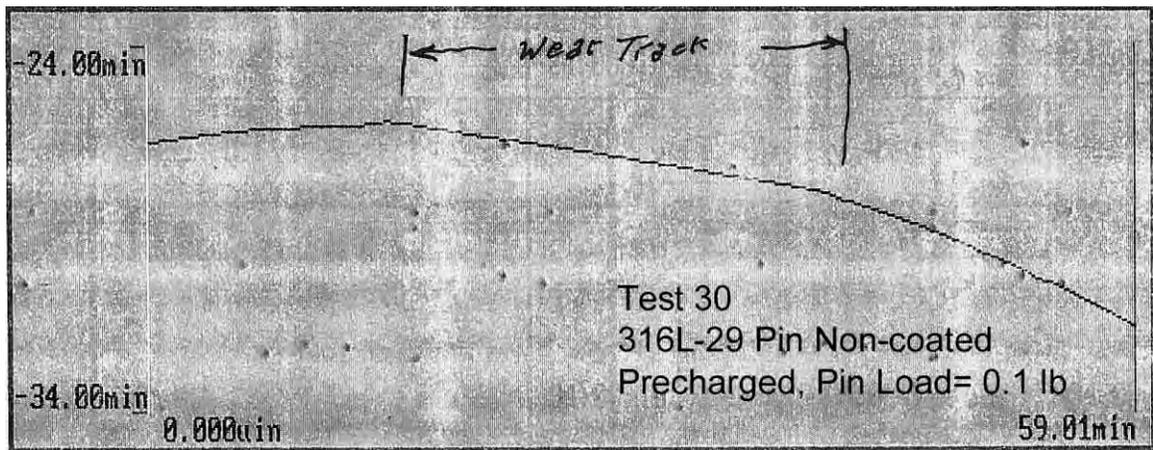
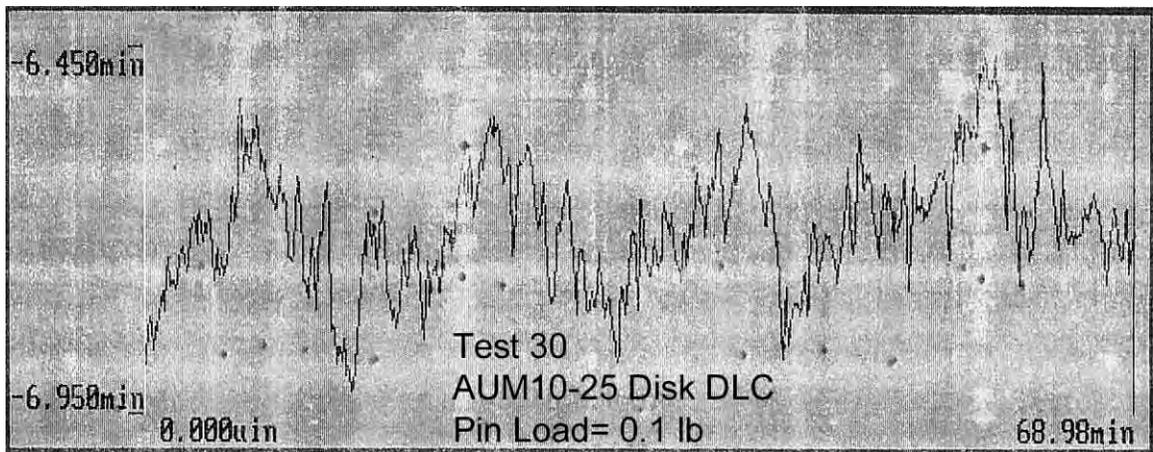
Test 29



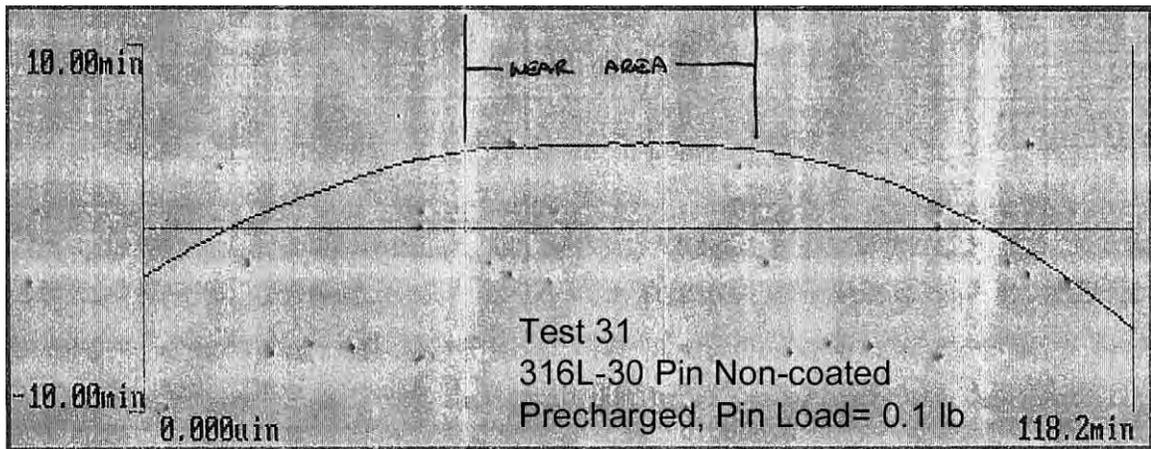
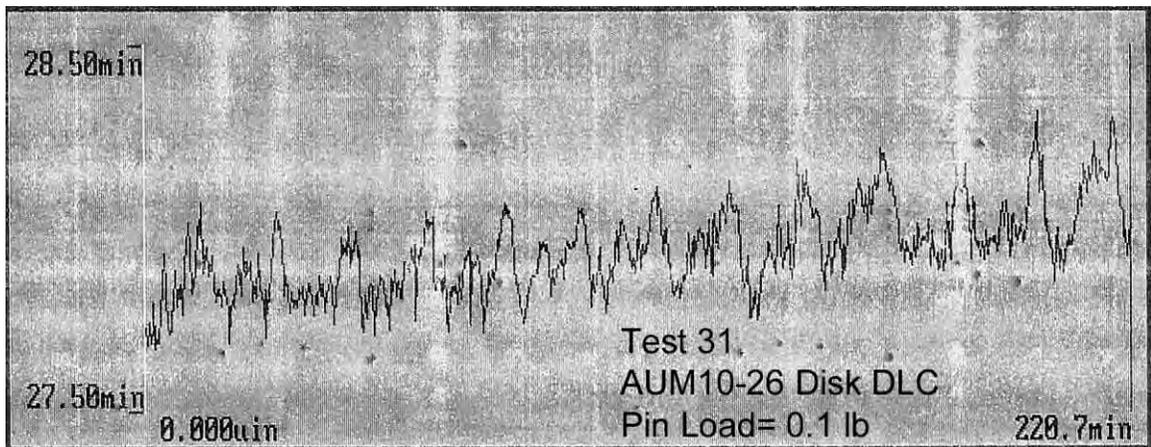
316L (Non-coated, Precharged)



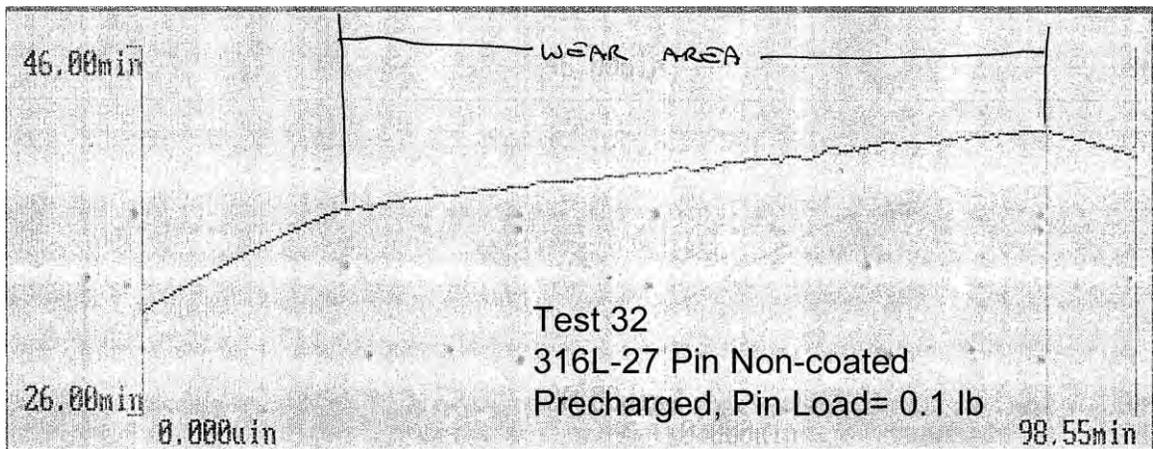
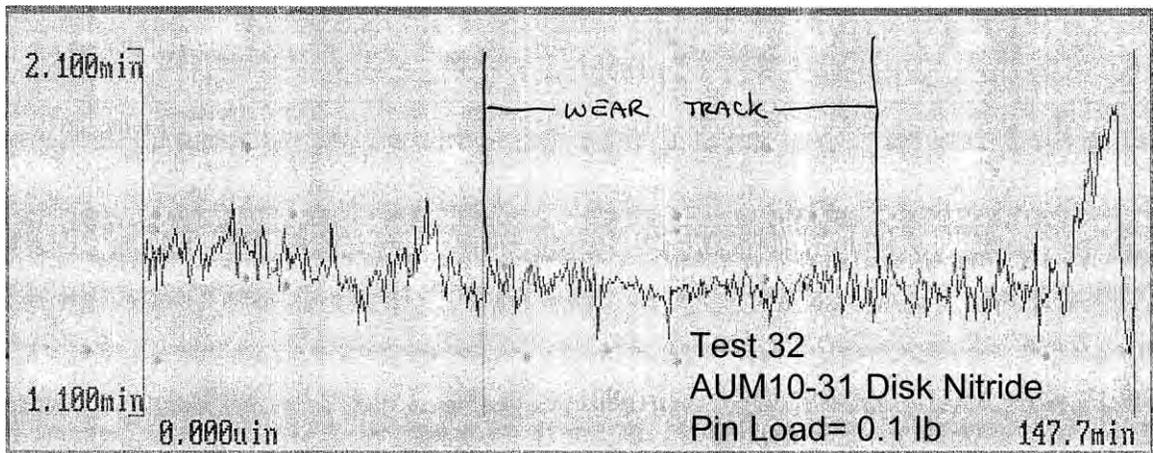
Test 30



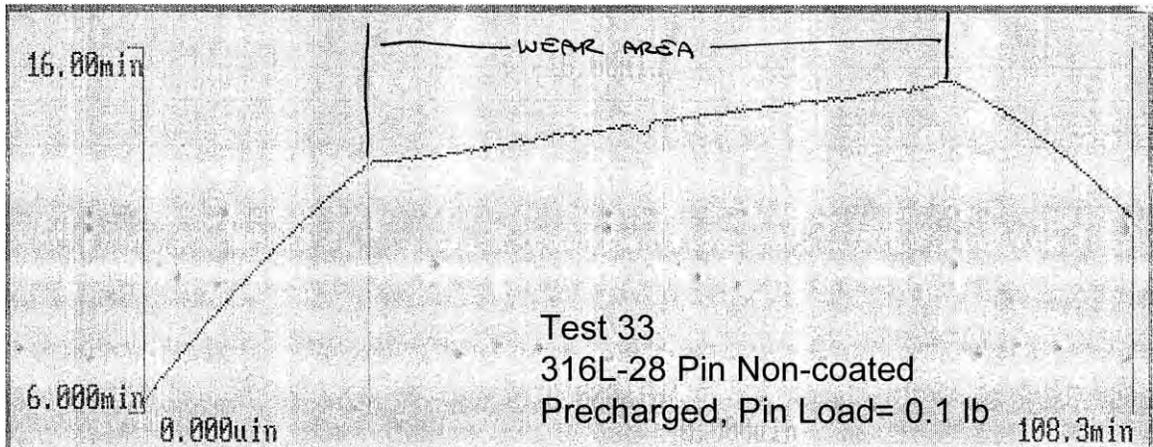
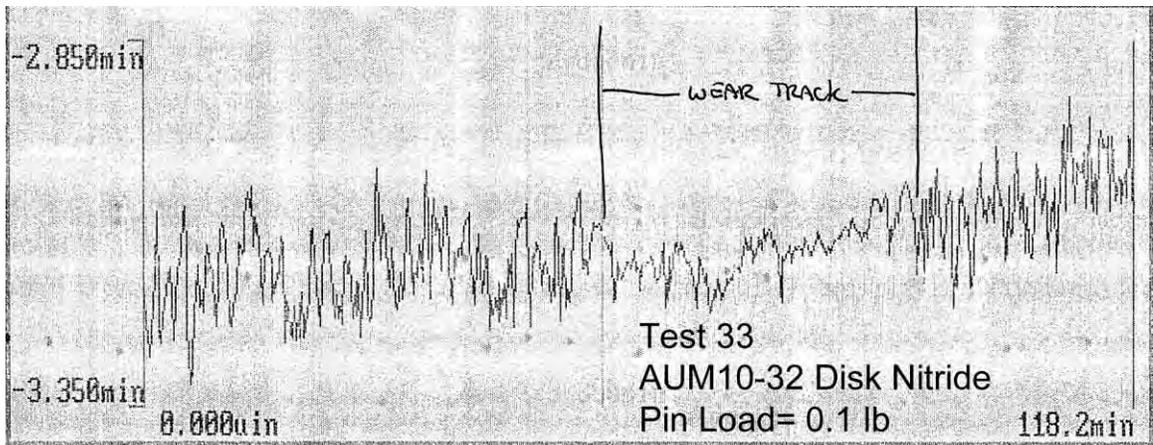
Test 31



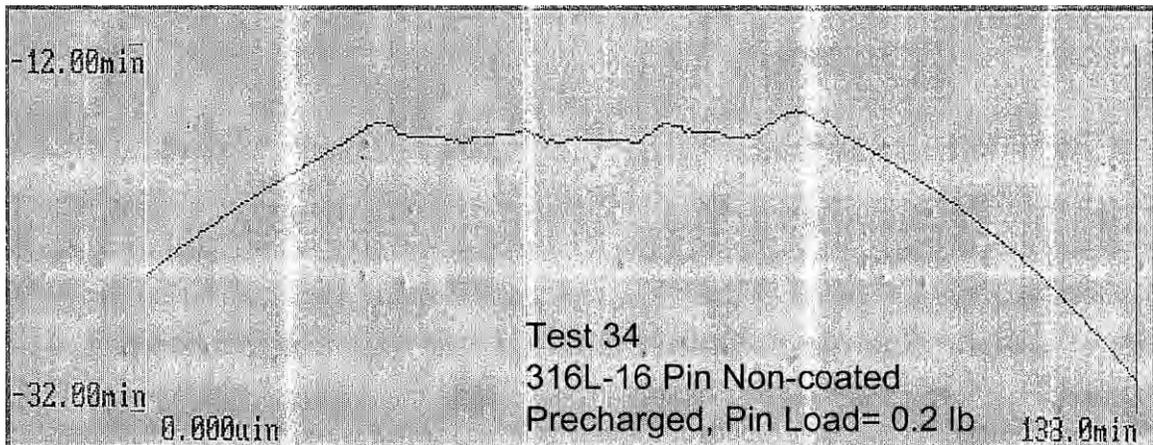
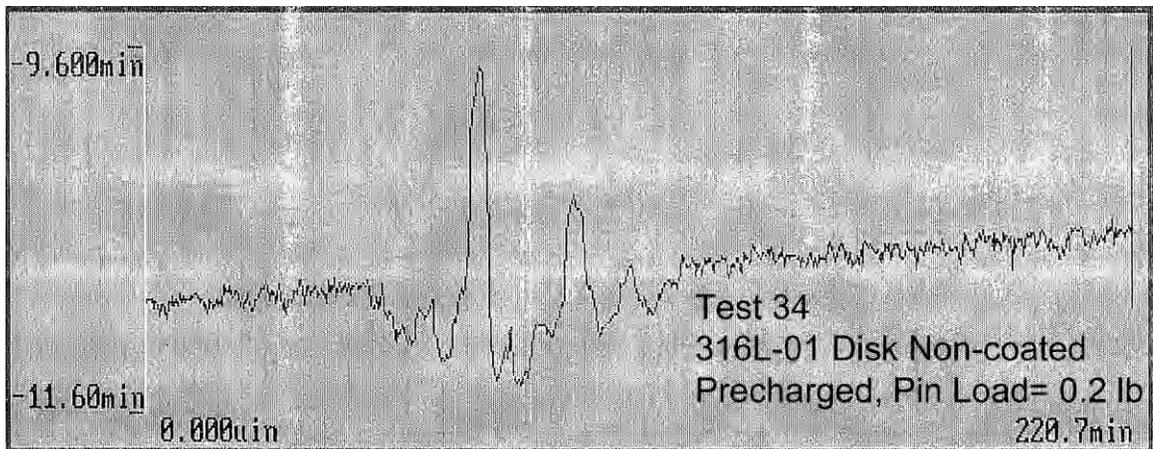
Test 32



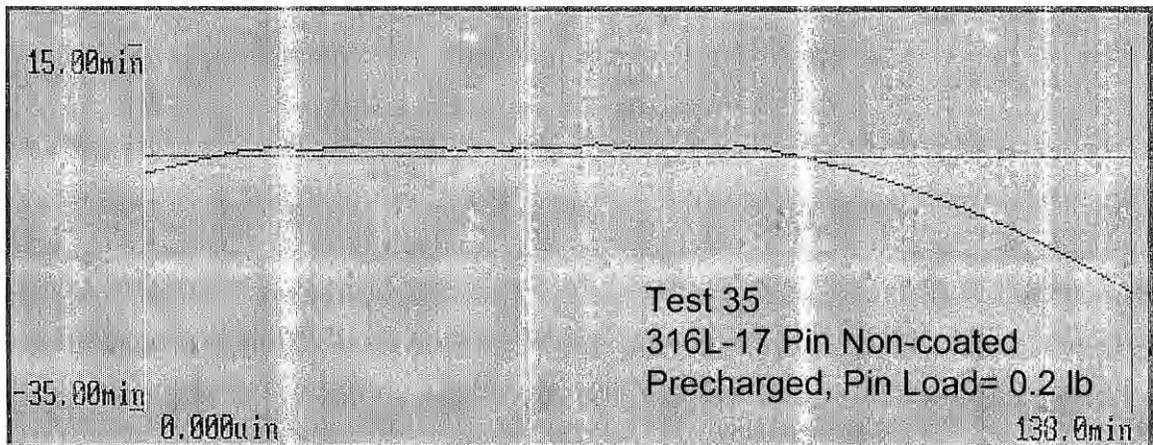
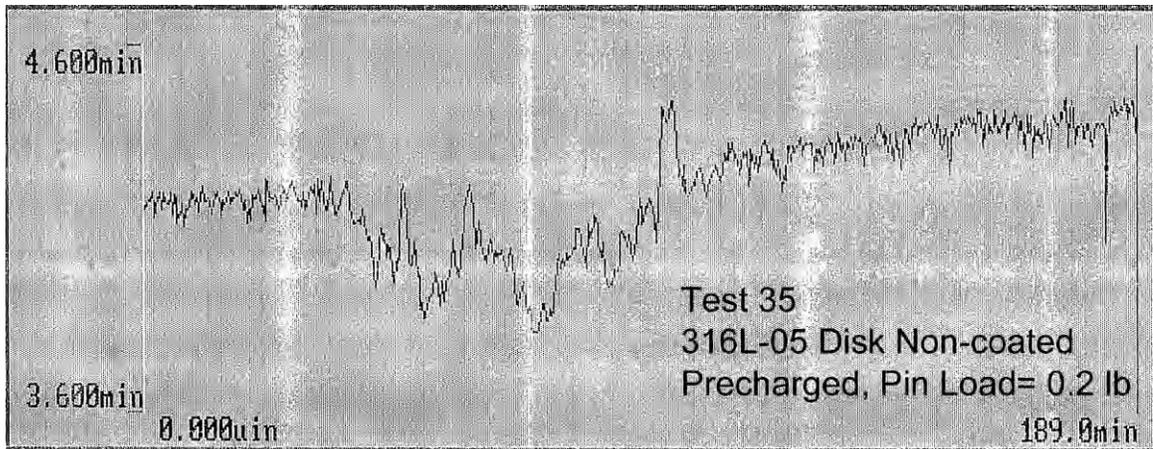
Test 33



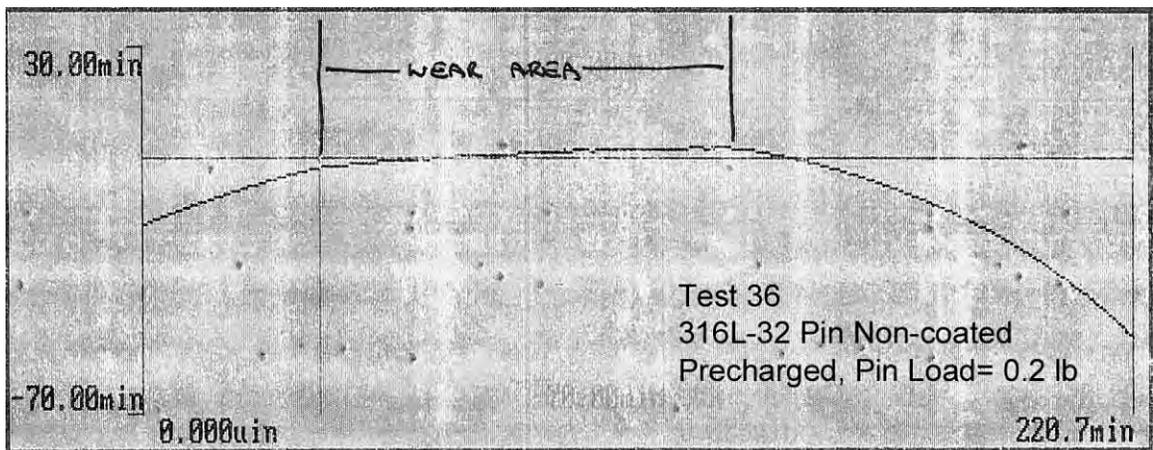
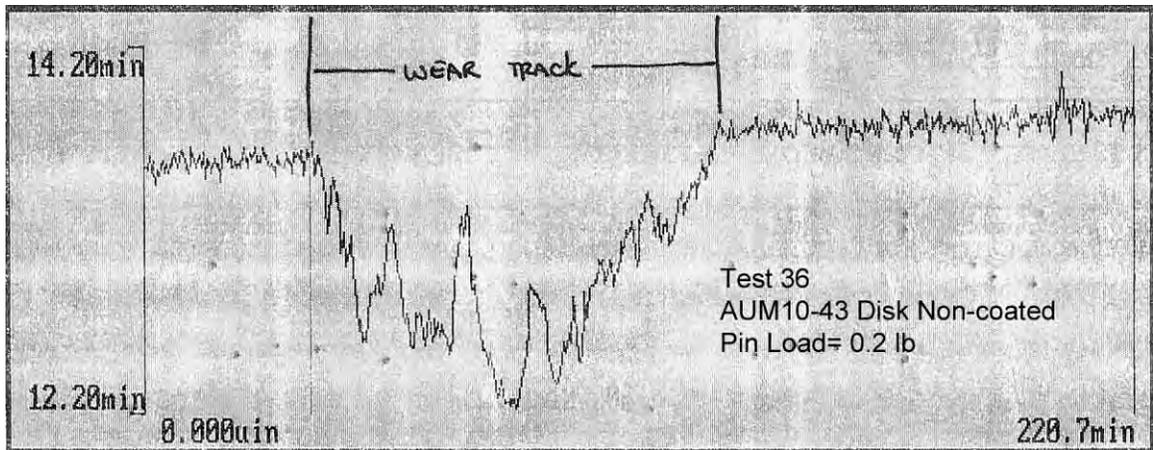
Test 34



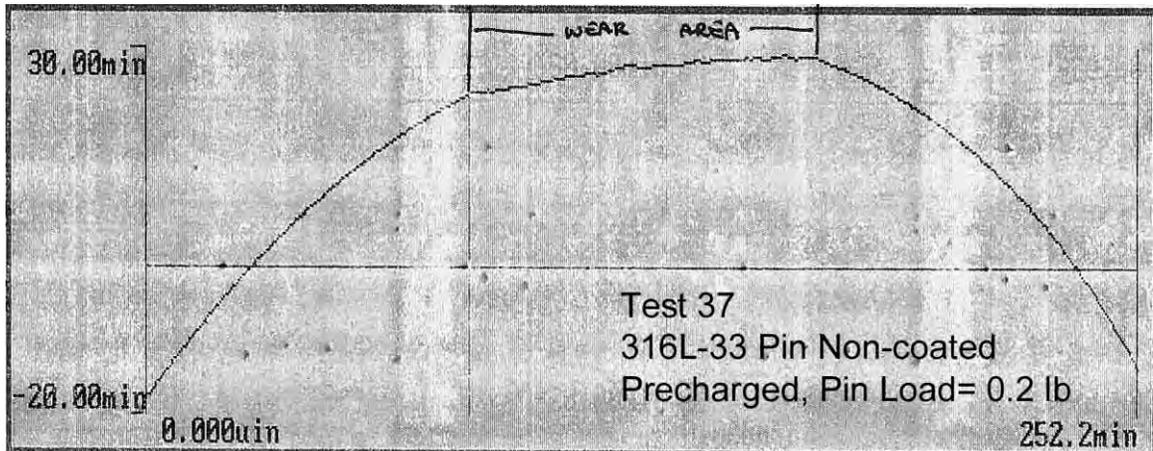
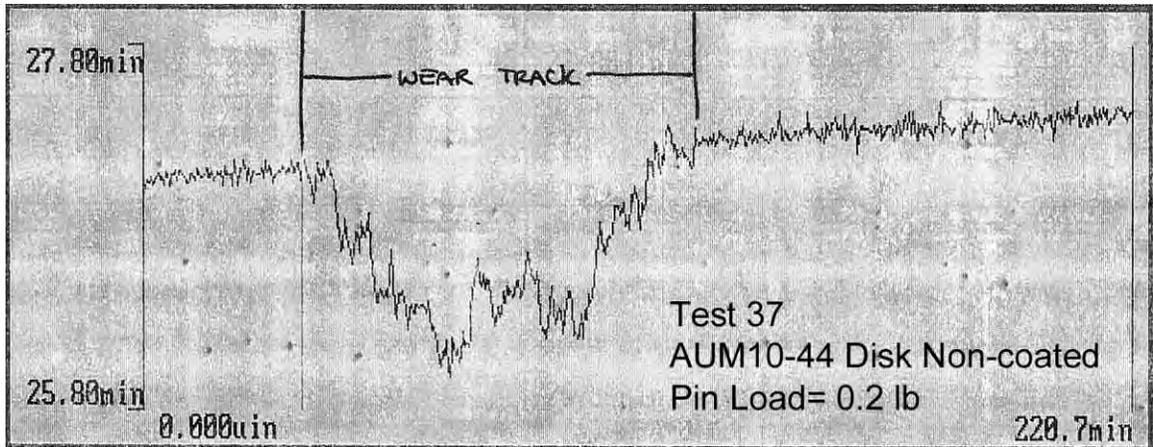
Test 35



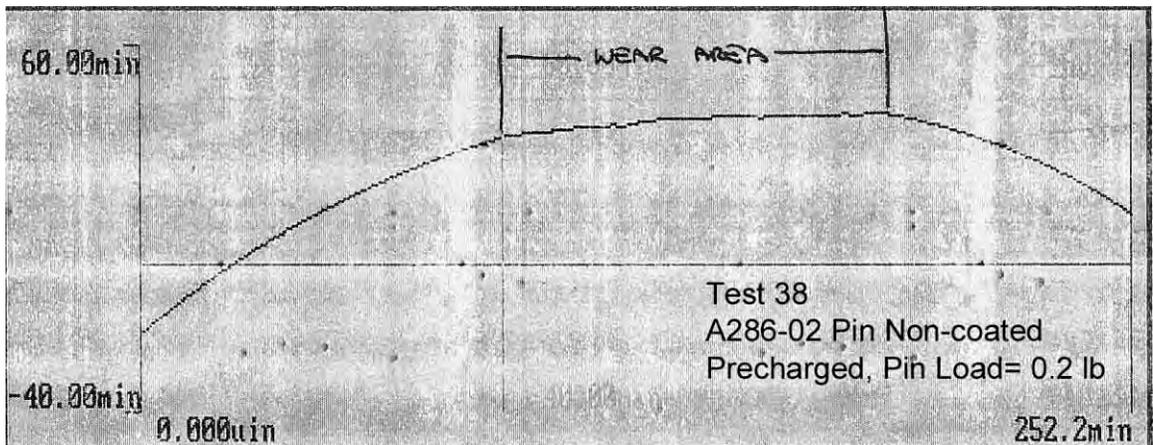
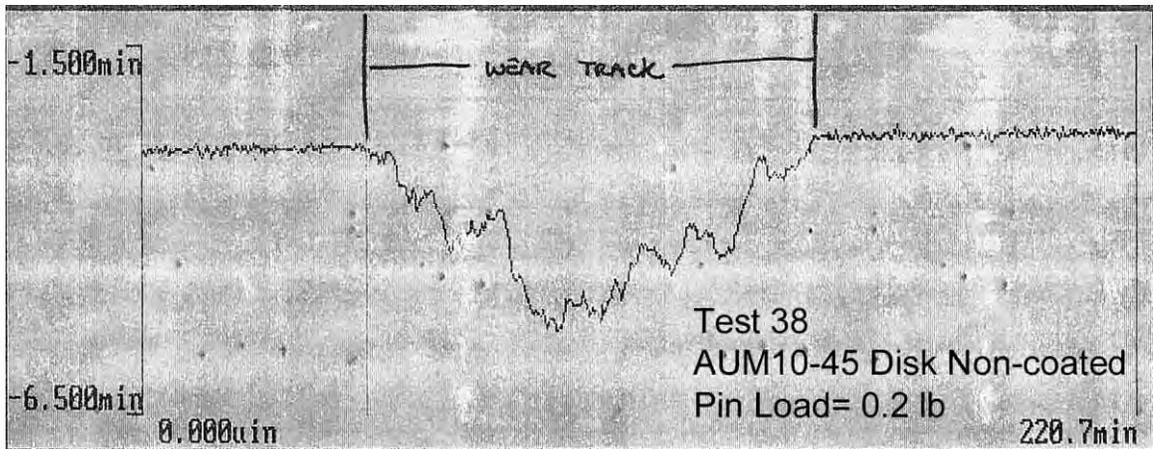
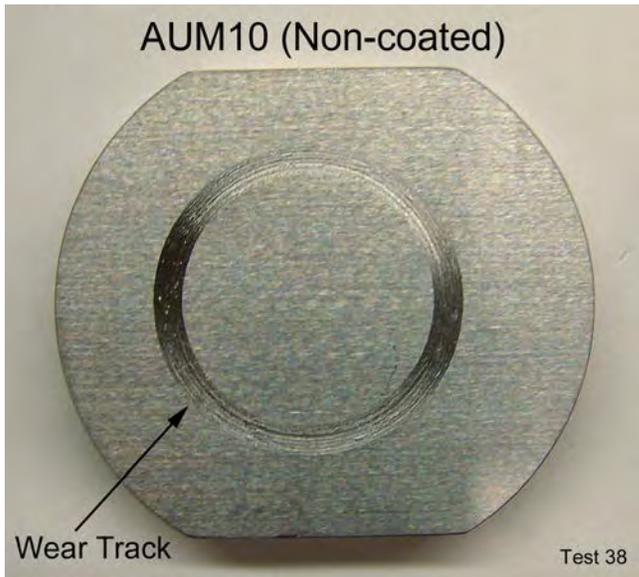
Test 36



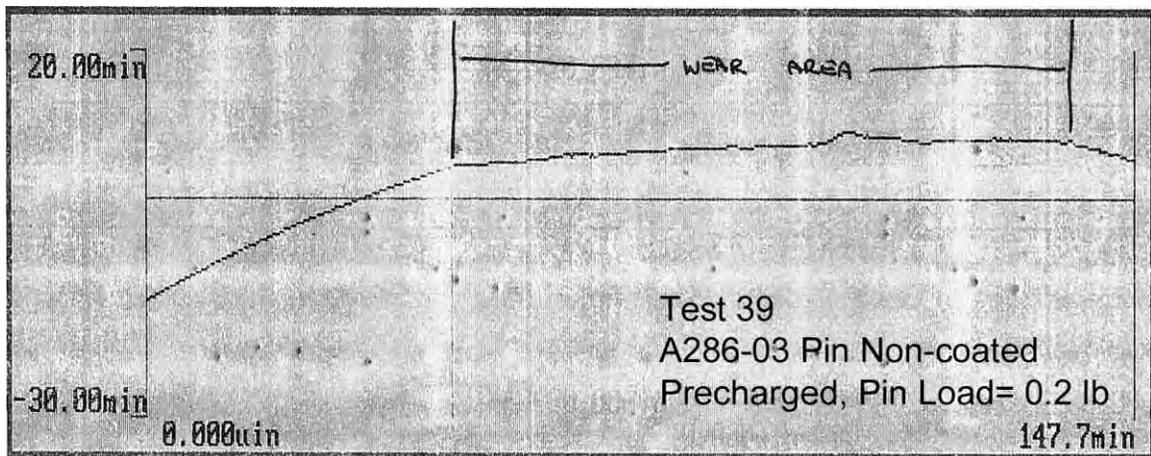
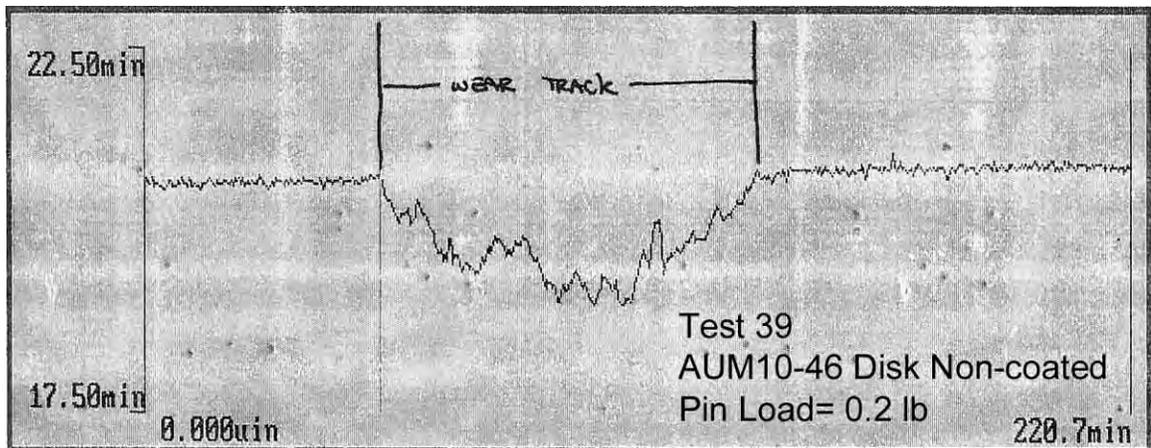
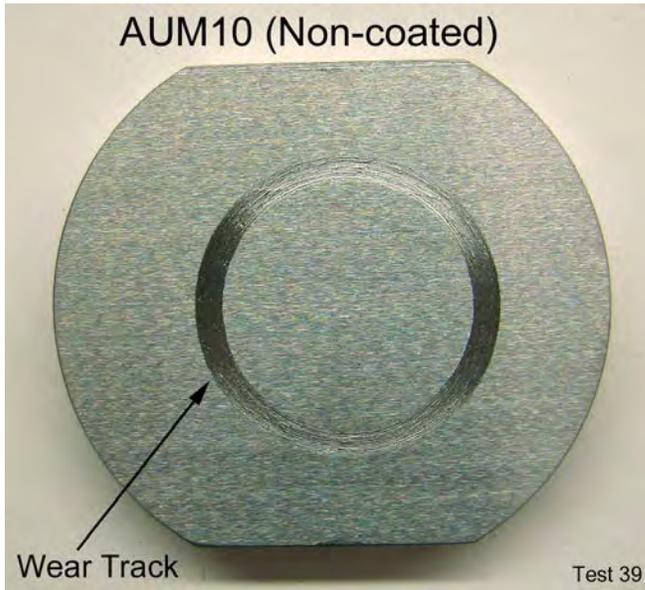
Test 37



Test 38



Test 39



Test 40

AUM10 (DLC Coated)



Wear Track

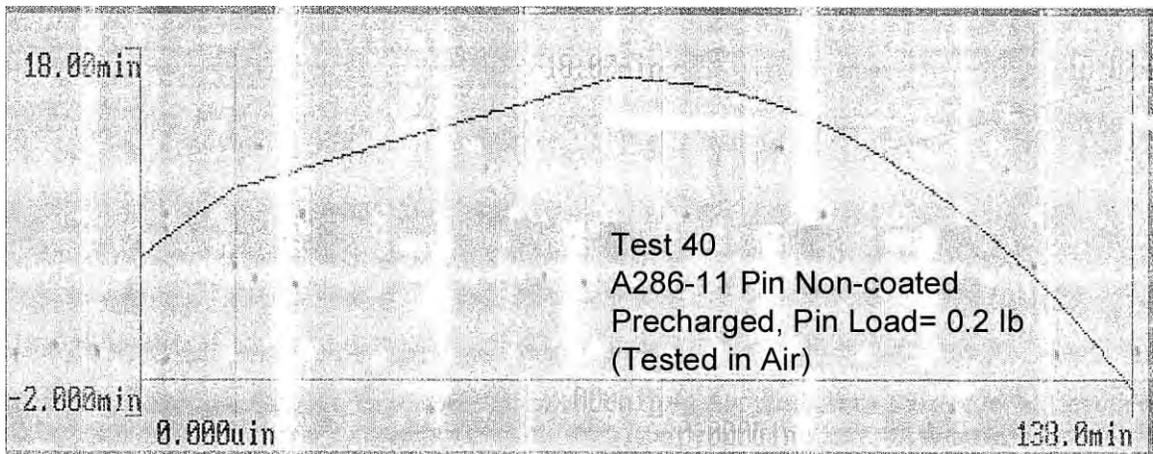
Test 40

A286 (Non-coated, Precharged)
Pin Load = 0.2 lb

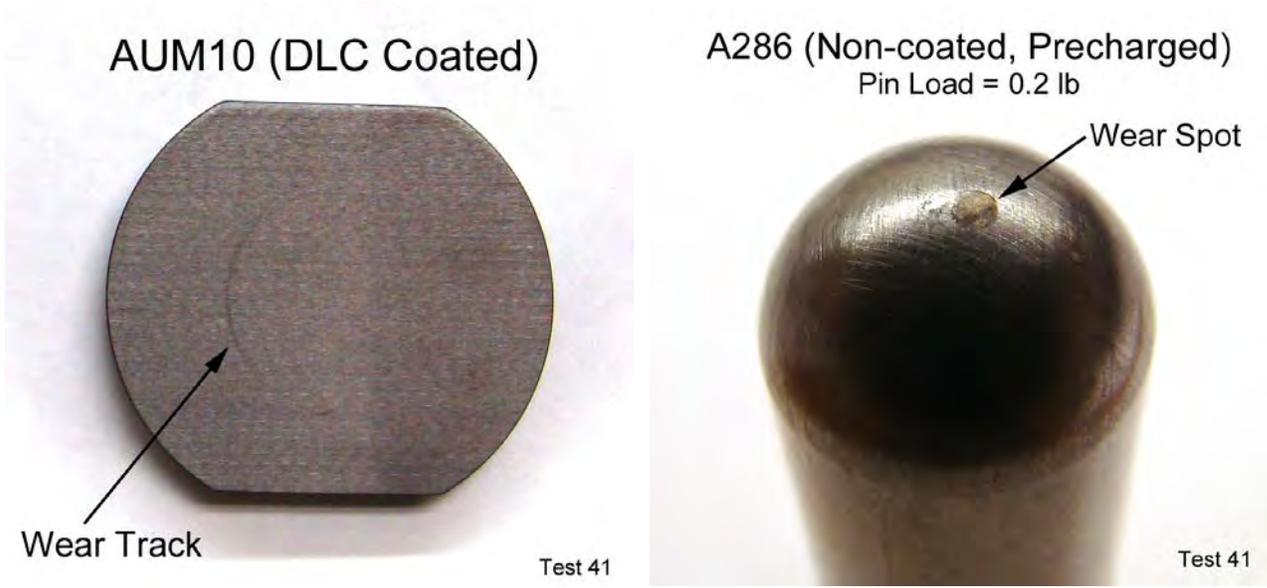


Test 40

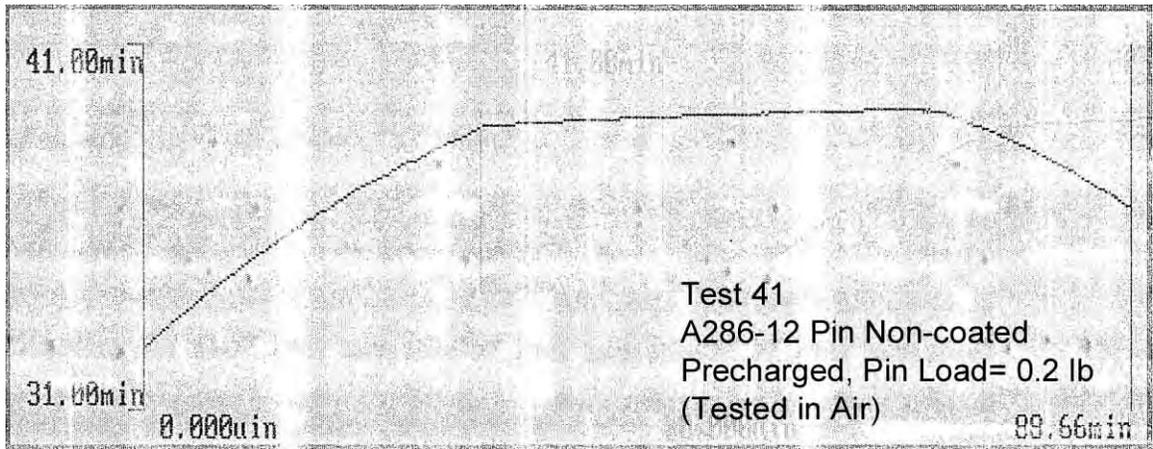
(Wear track on disk was not measurable.)



Test 41



(Wear track on disk was not measurable.)



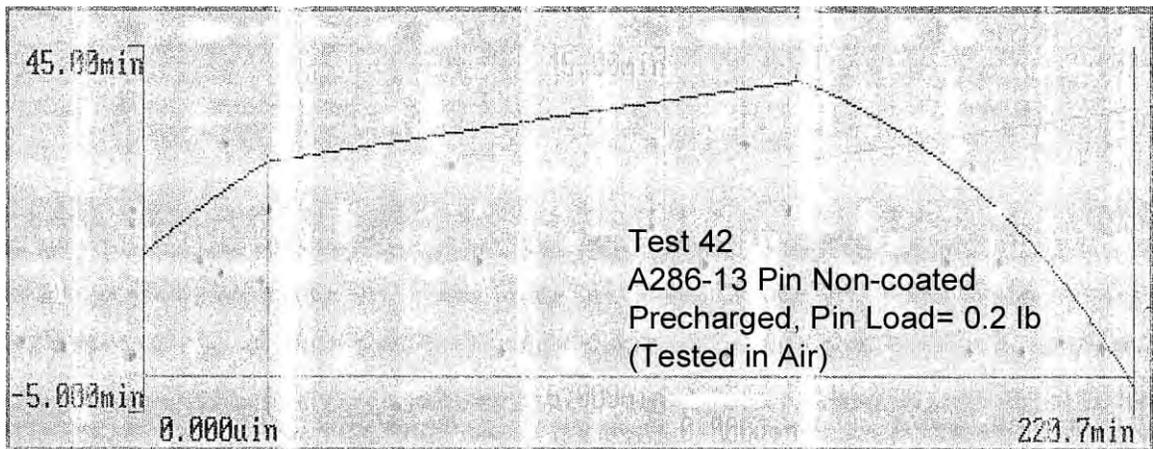
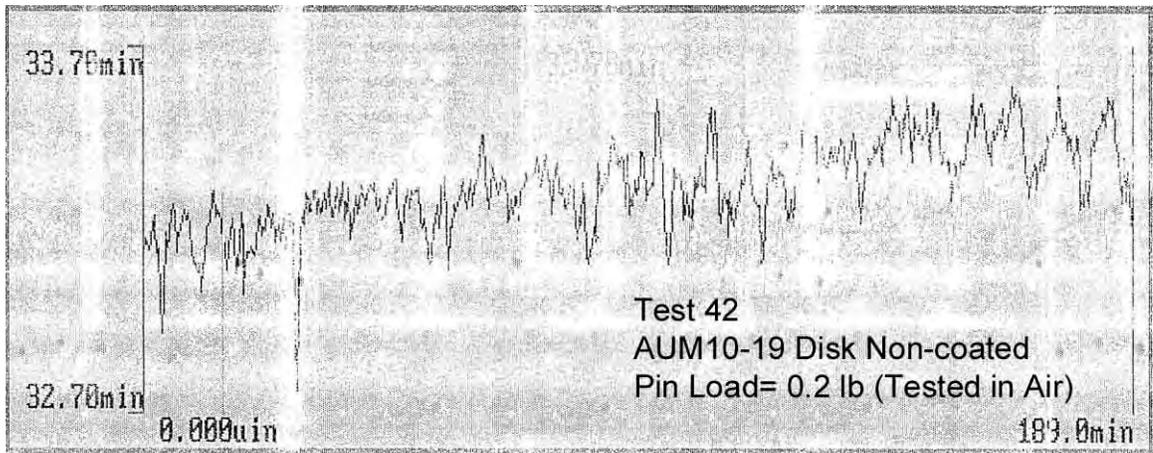
Test 42

AUM10 (Nitride Coated)

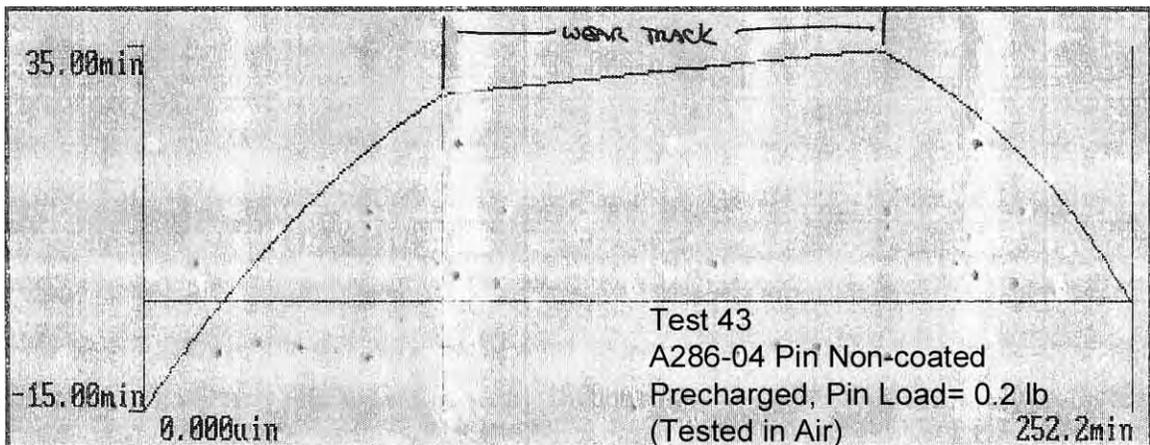
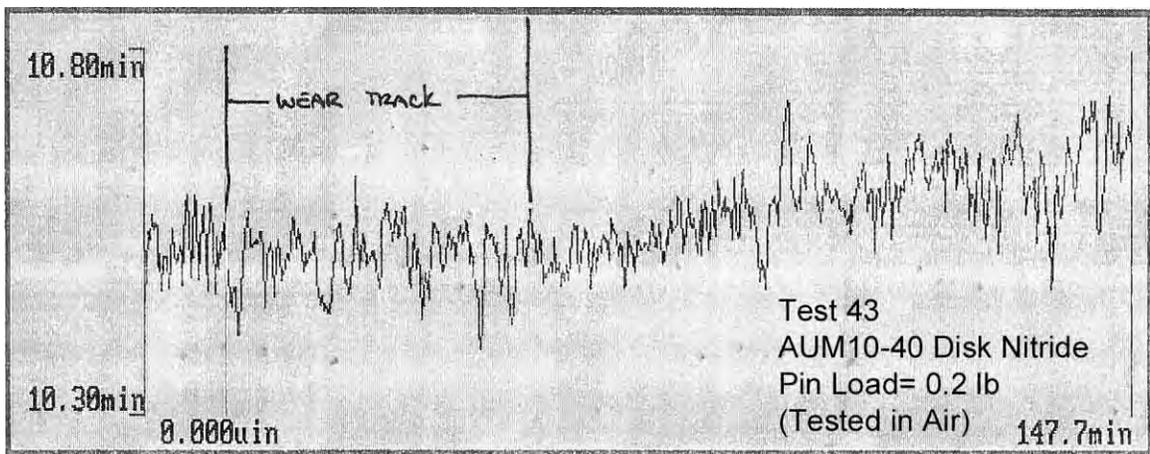
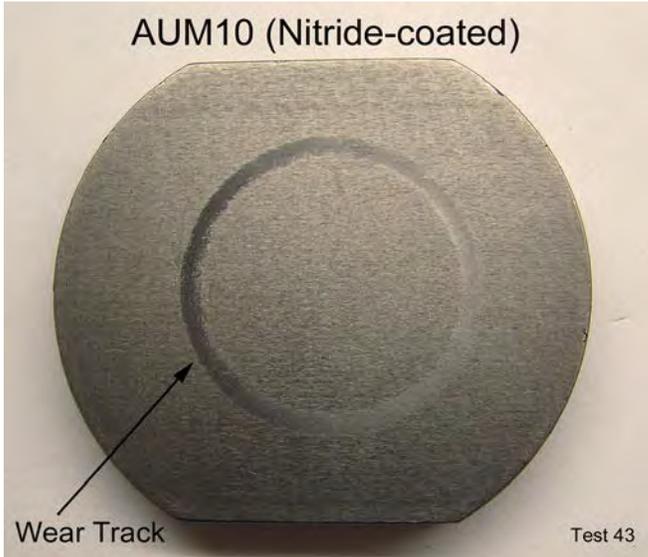


A286 (Non-coated, Precharged)

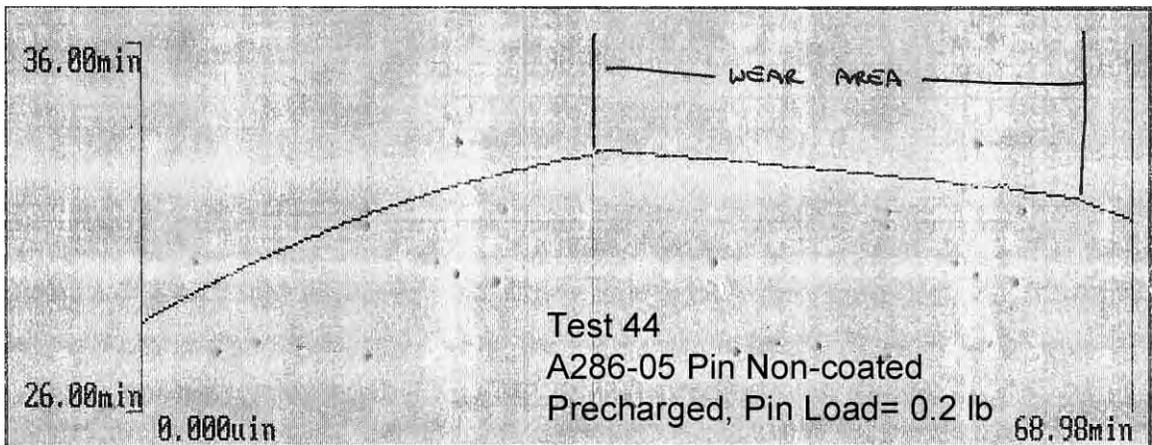
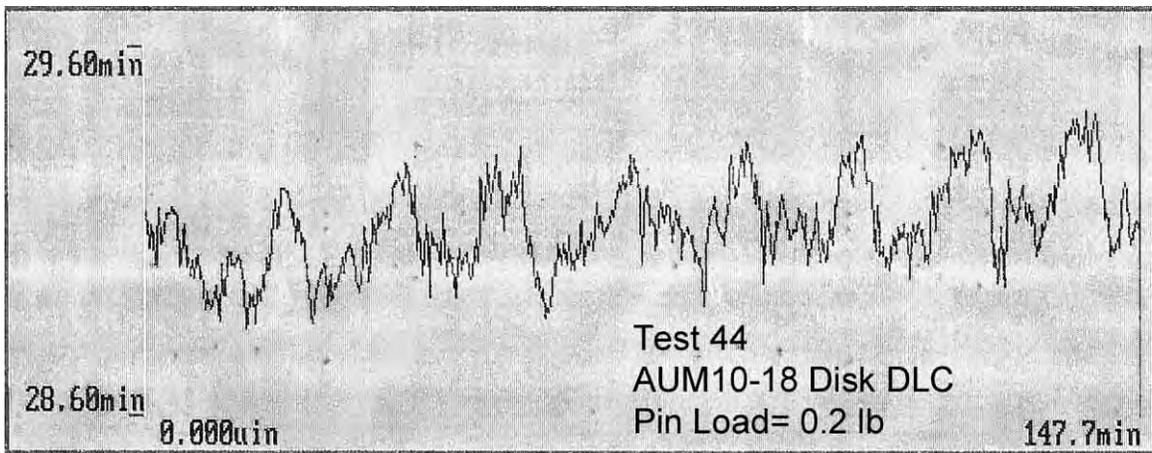
Pin Load = 0.2 lb



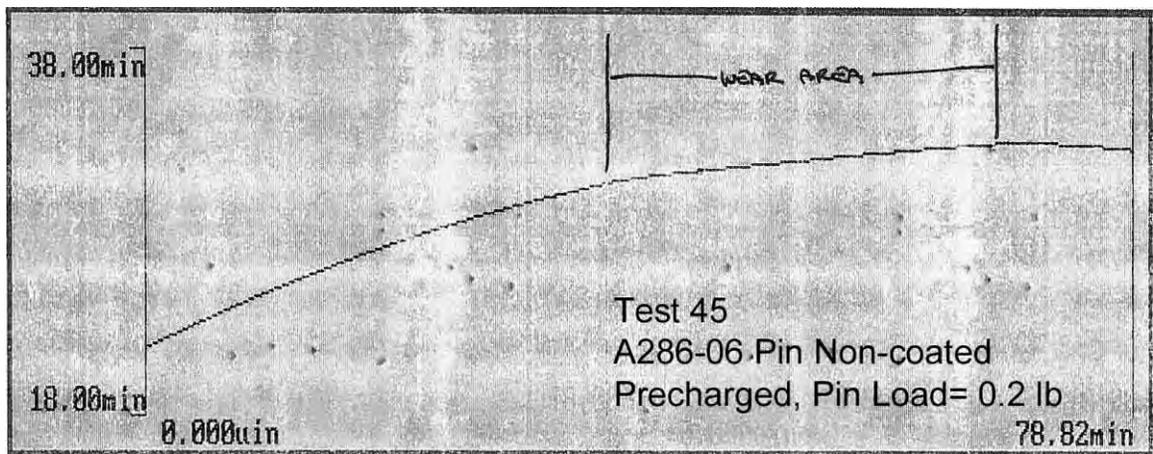
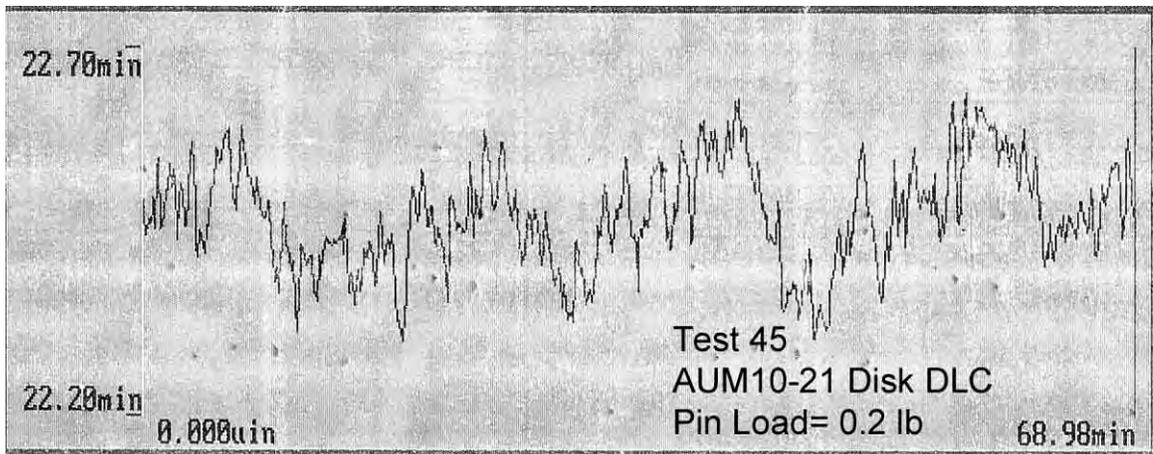
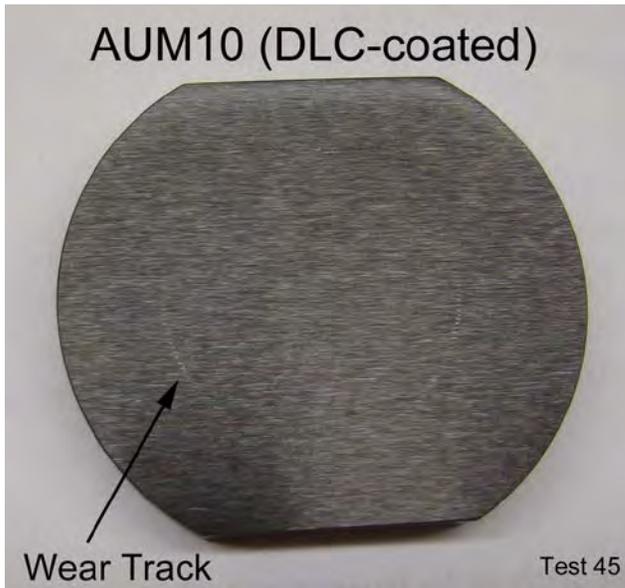
Test 43



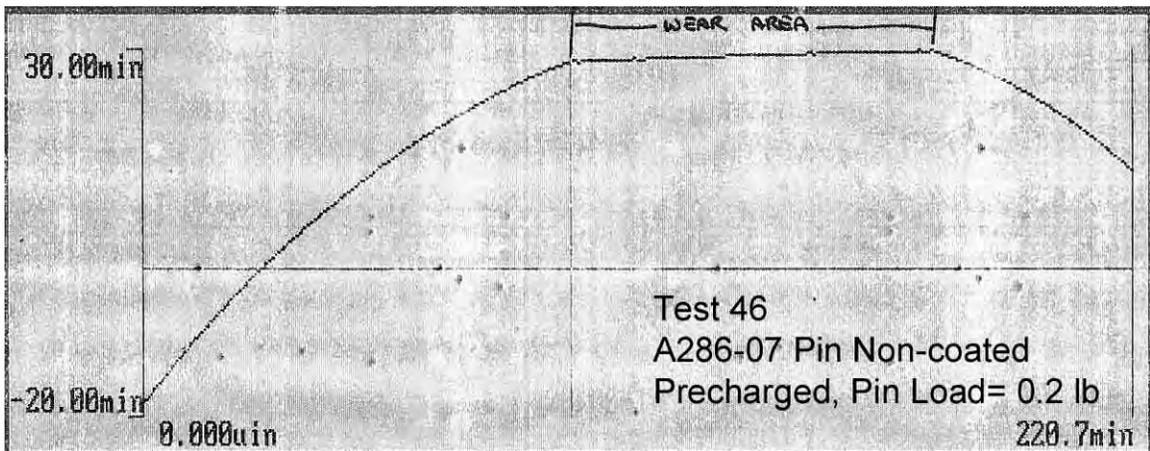
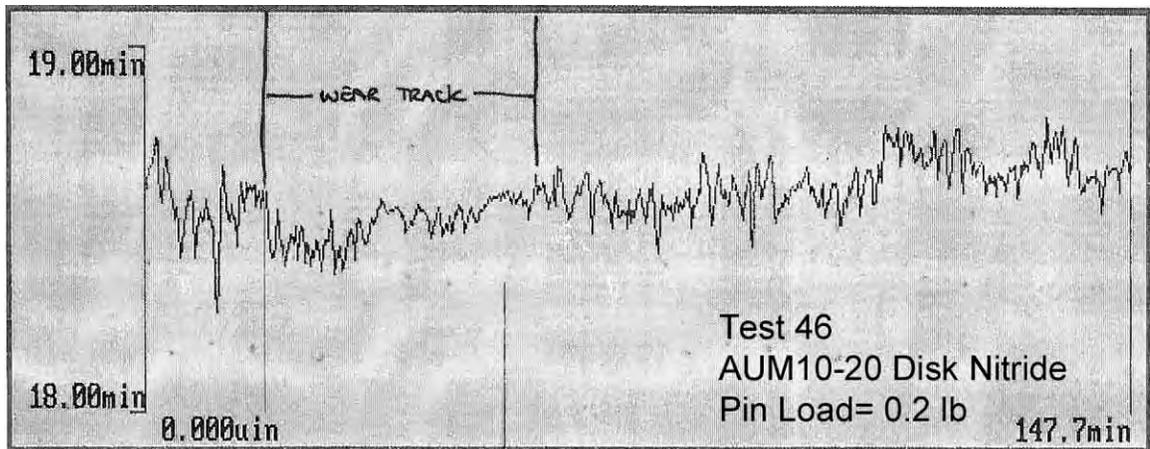
Test 44



Test 45



Test 46



Test 47

AUM10 (Nitride-coated)

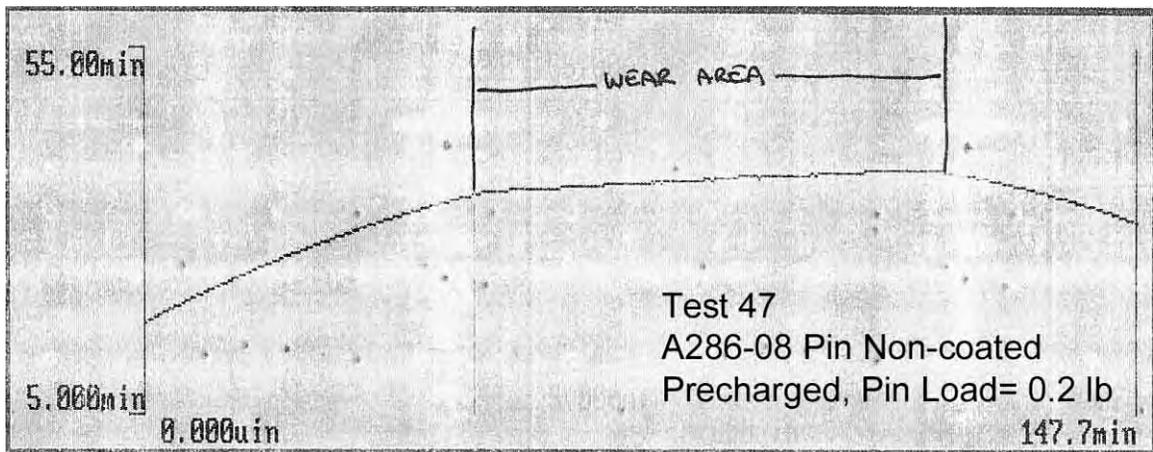
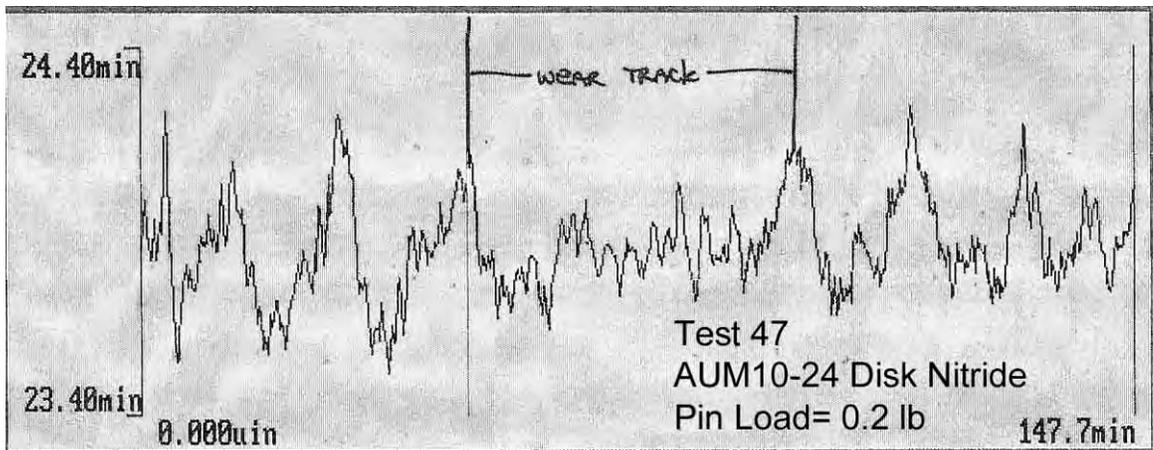


Test 47

A286 (Non-coated, Precharged)



Test 47



Test 48

AUM10 (DLC Coated)



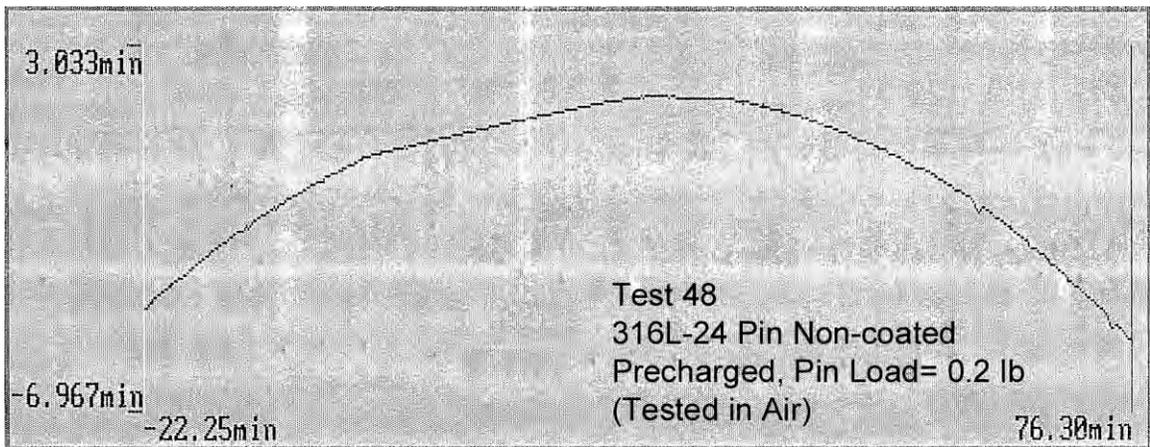
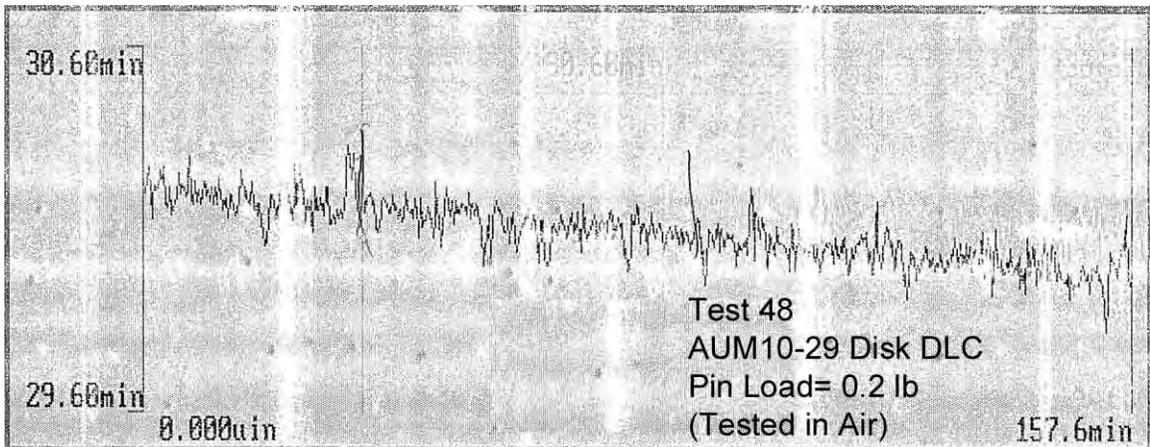
Test 48

316L (Non-coated, Precharged)

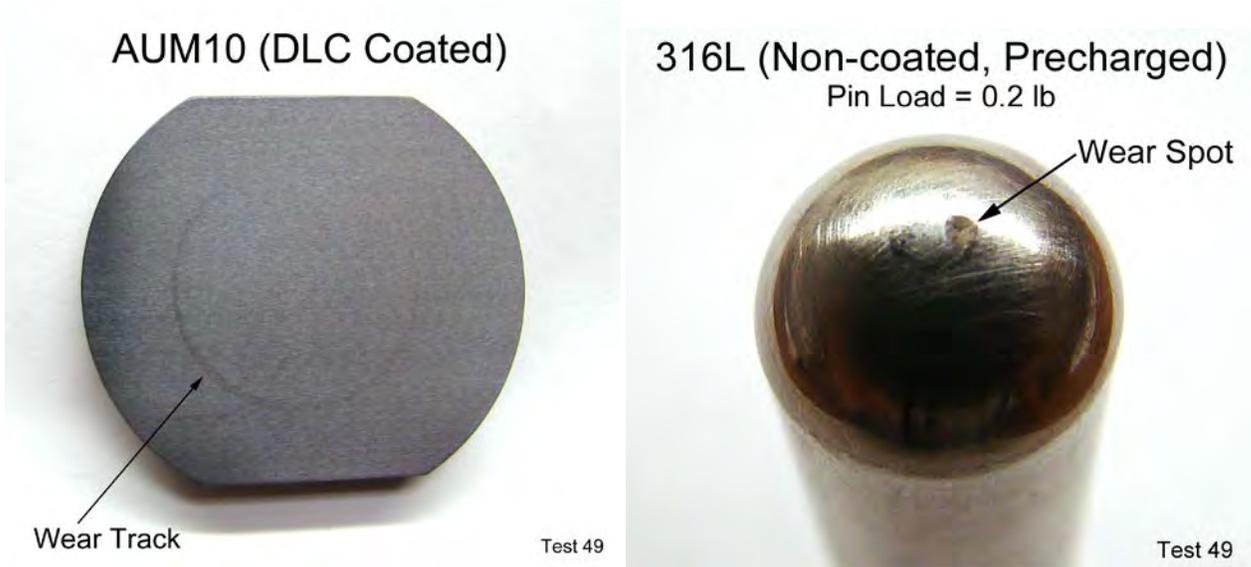
Pin Load = 0.2 lb



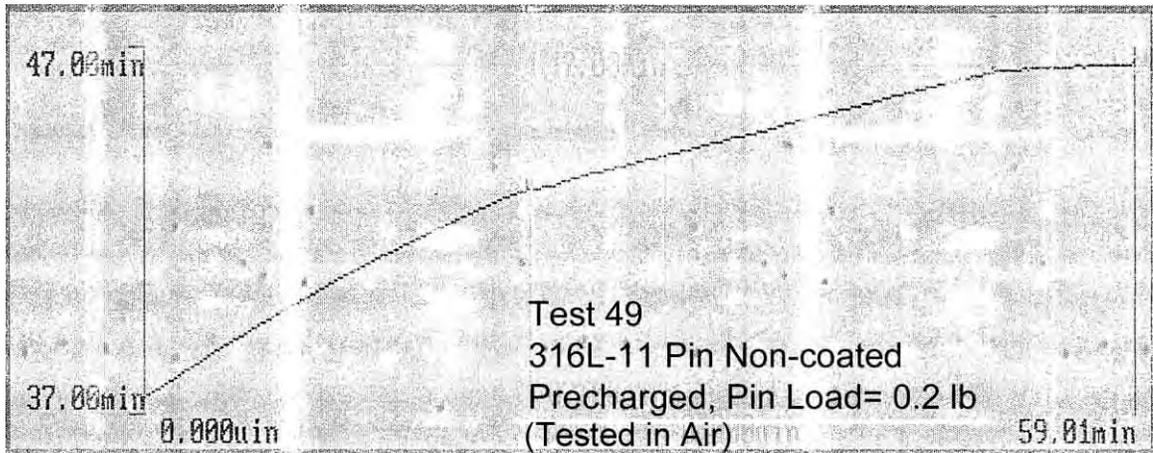
Test 48



Test 49

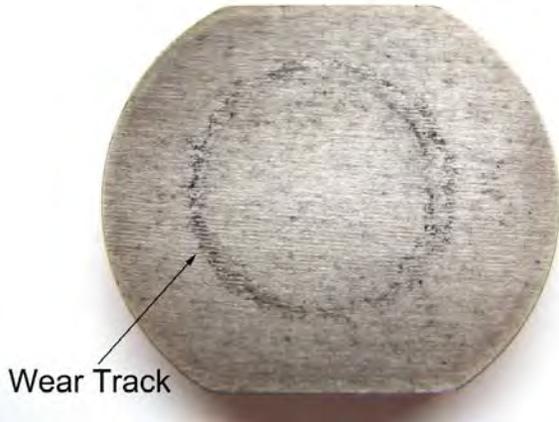


(Wear track on disk was not measurable.)



Test 50

AUM10 (Nitride Coated)

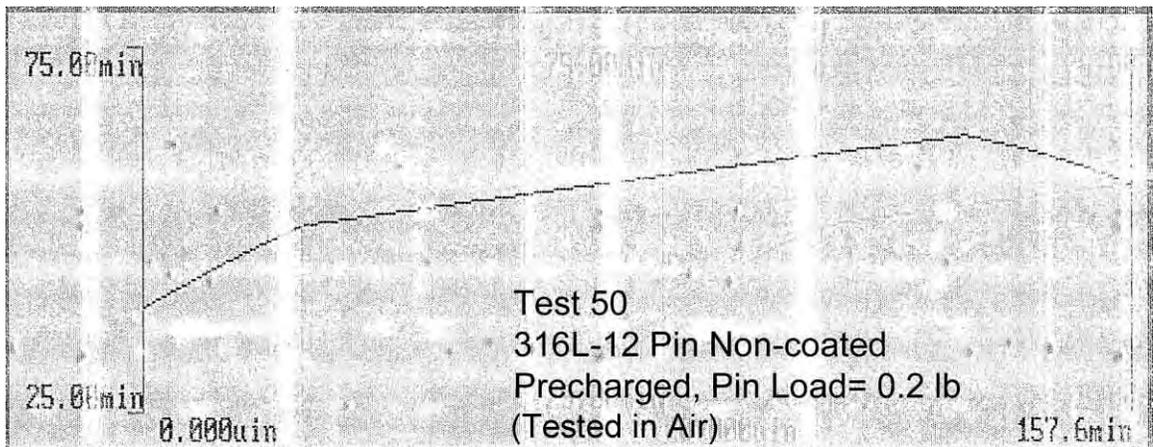
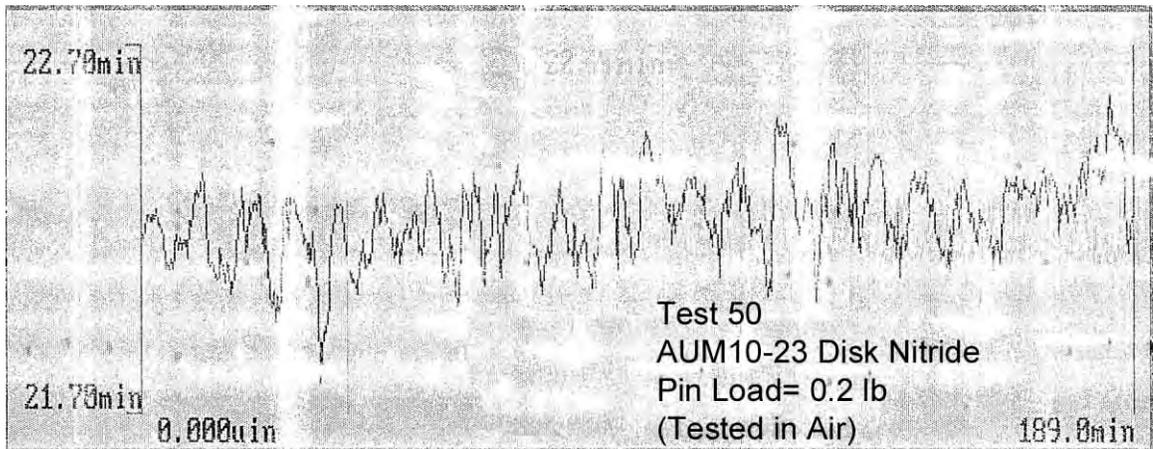


Test 50

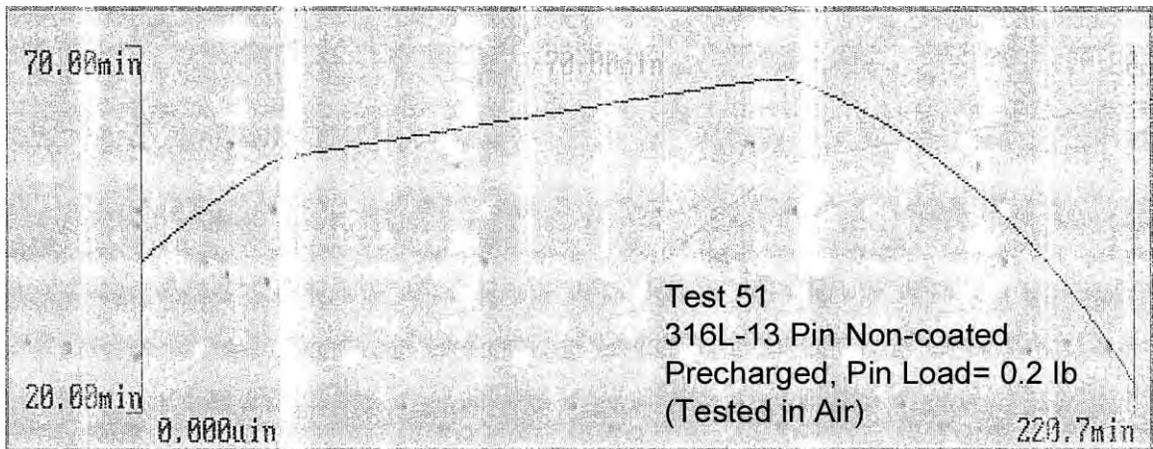
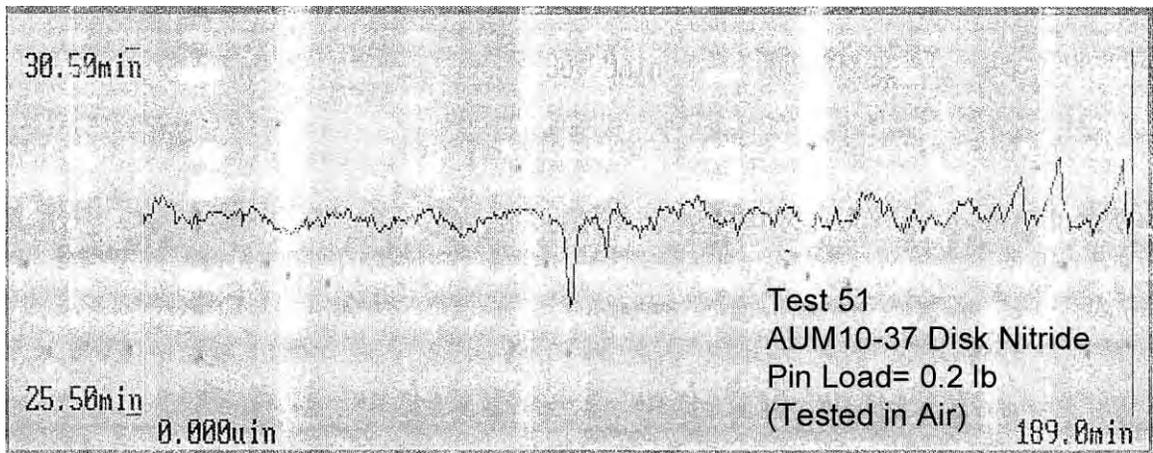
316L (Non-coated, Precharged)
Pin Load = 0.2 lb



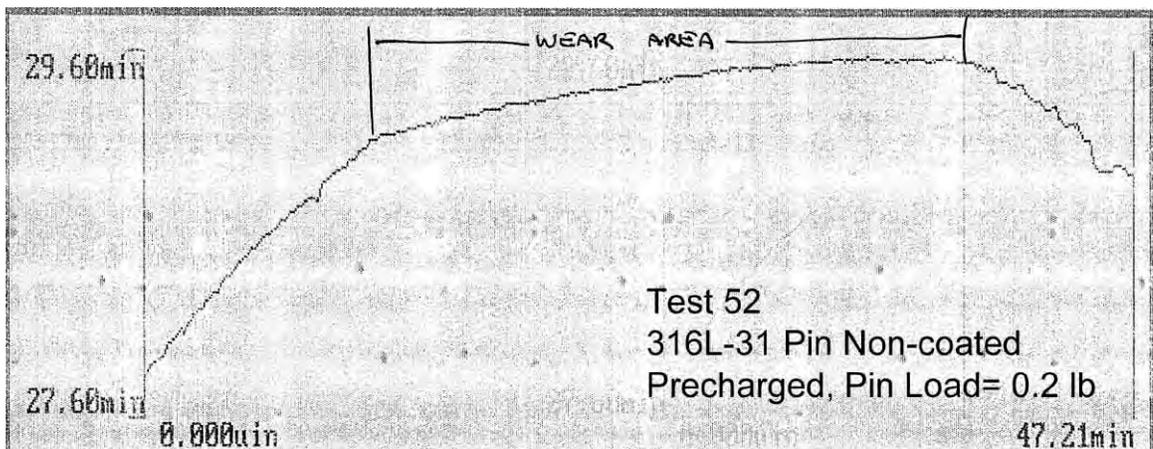
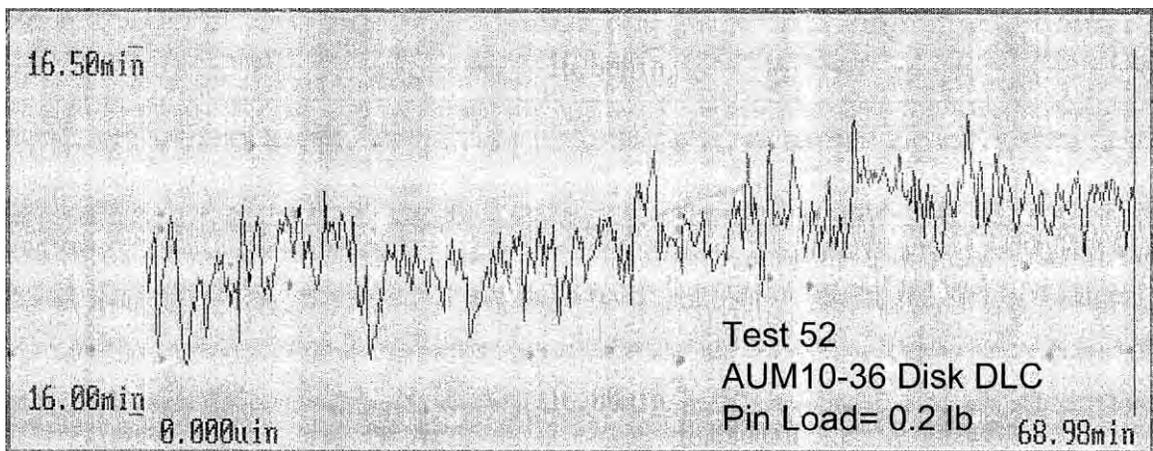
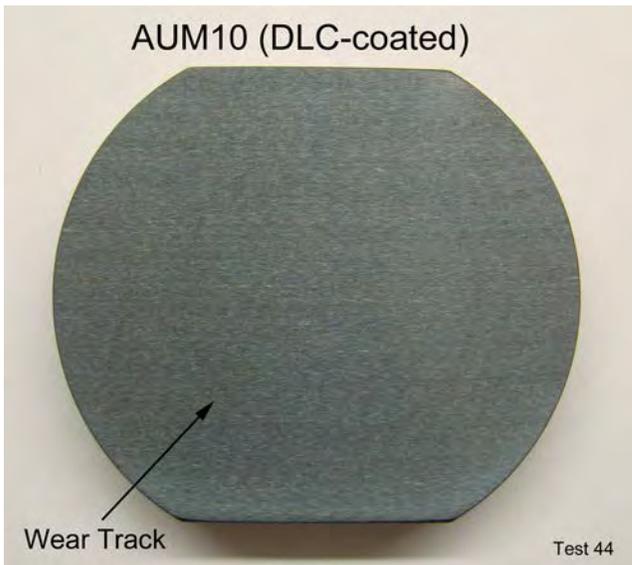
Test 50



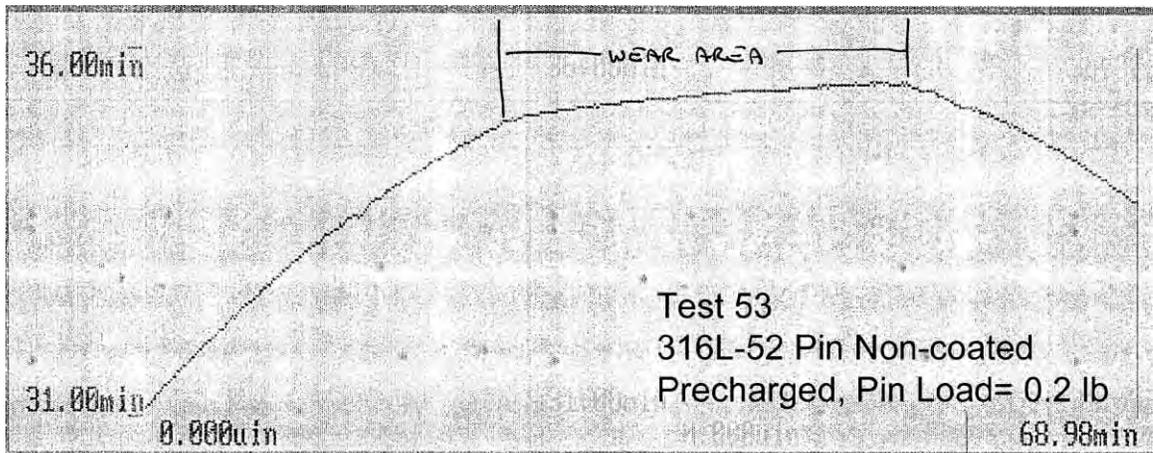
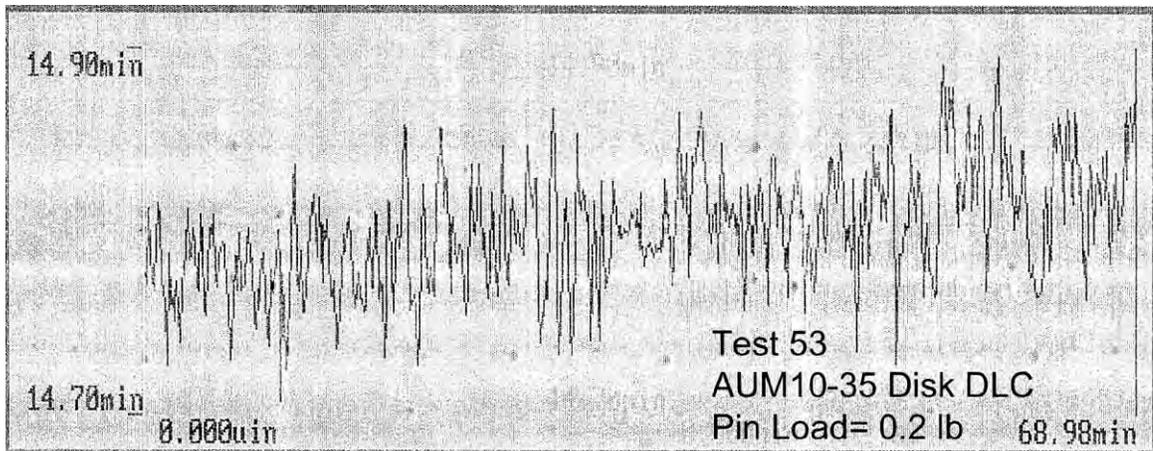
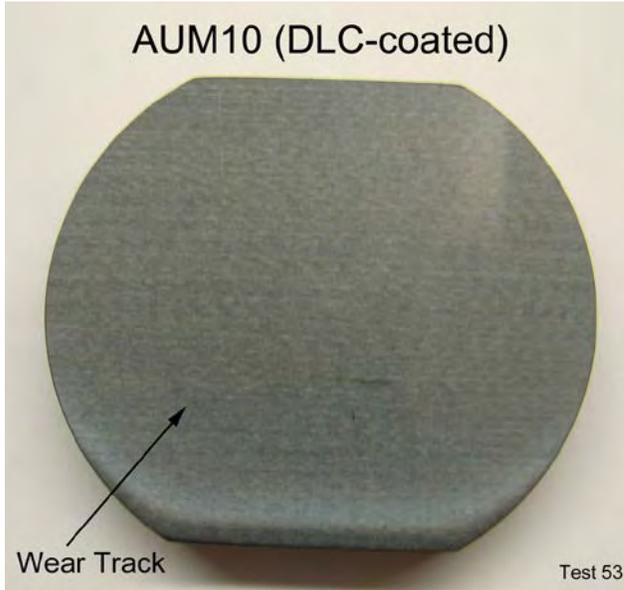
Test 51



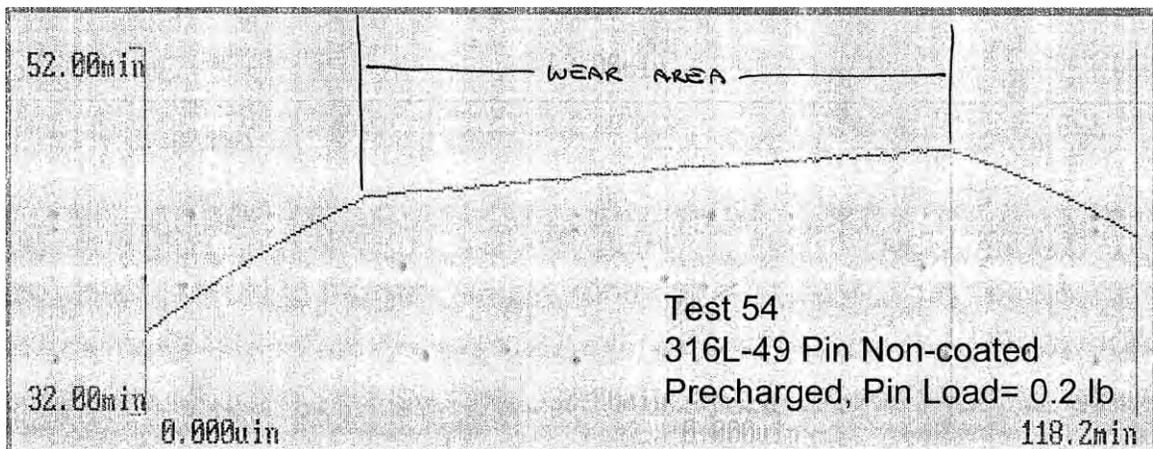
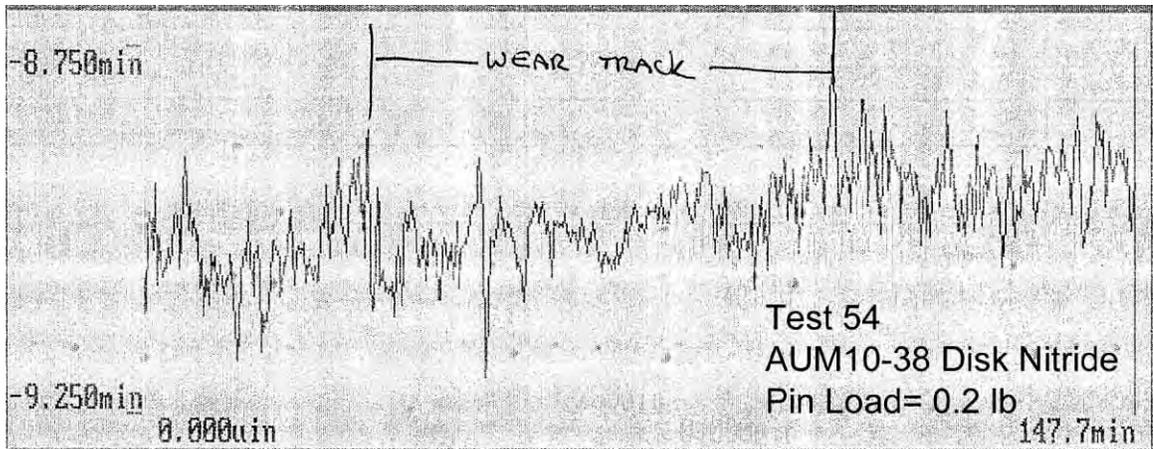
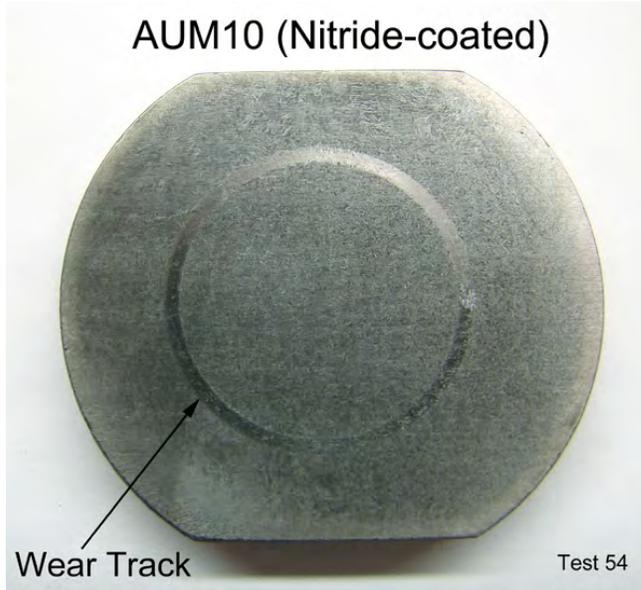
Test 52



Test 53



Test 54

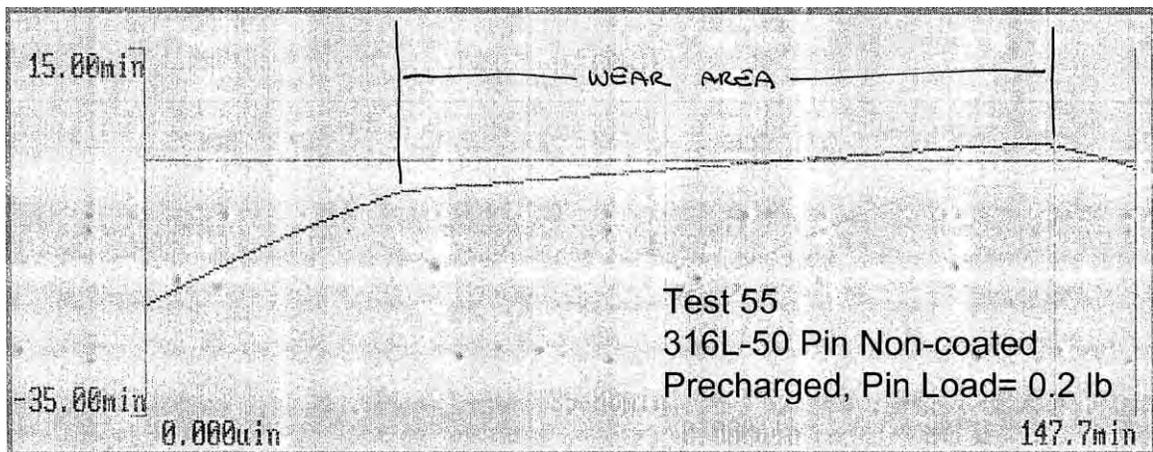
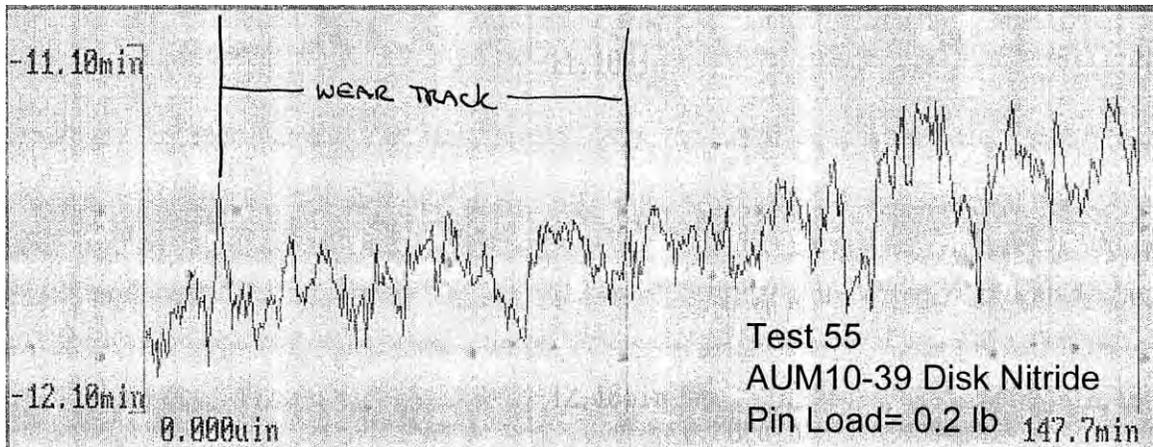


Test 55

AUM10 (Nitride-coated)



316L (Non-coated, Precharged)



SRNL-STI-2010-00013

DISTRIBUTION

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EXTERNAL

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