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ROLLING CONTACT FATIGUE OF SURFACE MODIFIED 440C USING A "GE-POLYMET" TYPE DISC ROD TEST RIG

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16. ABSTRACT

Through hardened 440C martensitic stainless steel test specimens were surface modified and tested for changes in rolling contact fatigue using a disc on rod test rig. The surface modifications consisted of nitrogen, boron, titanium, chromium, tantalum, carbon, or molybdenum, ion implantation at various ion fluences and energies. Tests were also performed on specimens reactively sputtered with titanium nitride.

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INTRODUCTION

A study was undertaken to improve the durability of turbopump bearings by modifying the bearing active surfaces through ion implantation or by application of thin tribological films. It was planned to develop a process which would increase resistance to wear, and rolling contact fatigue (RCF), and also decrease the coefficient of friction of the active bearing surfaces.

Ion implantation was chosen as a process to increase wear resistance due to the effect it has on the surface of the alloy and the fact that it does not change the dimensional properties of the substrate. The ions are accelerated in a vacuum through a high potential and impinged onto the substrate surface. This creates local crystalline damage in the near surface region down to approximately 0.1 μm (4 μin.). Some of this damage can be severe enough to create an amorphous surface region. In addition to the damage to the crystalline structure, the ions implanted can impart unique characteristics that may be advantageous. For example, implantation of chromium may improve the corrosion resistance of an alloy. The atoms implanted reside in either substitutional, if they are larger atoms such as metallic atoms, or in interstitial sites, if they are smaller implanted atoms like nitrogen or carbon.

Improvements of mechanical properties with ion implanted test specimens have been mixed. An improvement in wear resistance has been observed by a group at Sandia [1] testing nitrogen ion implanted test specimens. Others have published reports of RCF improvements with ion implanted test specimens [2,3]. Using nitrogen ion implantation, White and Dearnaley [4] saw an improvement in a four ball test of EN 31 steel test specimens. Tests, done by the author, of ion implanted RCF test specimens have yielded positive but small improvements in life using two different types of fatigue test rigs [5]. These improvements, however, have not been statistically significant at a 95 percent confidence level.

Hard thin coatings are also being tried as a means to improve wear resistance. The reactively-sputtered titanium nitride used in this study, when applied stoichiometrically, is a very hard material. This material is presently being used as a coating for machine tool and drill bits to improve cutting and increase the life of tools.

Increases in the RCF life of coated test specimens is much more dramatic than with ion implanted samples. Bhat and Davis [6] showed a significant increase in RCF life with chromium ion plated samples. Large increases in RCF were shown for titanium nitride and copper coatings in References 5, 7, 8, and 9. Increases in RCF life are seen for coatings generally less than 1 μm in thickness, while coatings thicker than 2 μm crack and spall off. This data has been consistent for hard coatings over the past five years in testing performed at Marshall Space Flight Center (MSFC).

In this study test specimens were evaluated for changes in RCF life. The results presented in this report were obtained during testing which occurred during 1983 and 1984 using a disc on rod, General Electric (Polymet) type test rig.

EXPERIMENTAL PROCEDURE

A. Test Specimens

The test specimens were made from two lots of 440C martensitic stainless steel. The nominal composition of 440C is given in Table 1. These test specimens were rough machined to the approximate size and heat treated in accordance to Table 2. From 0.030 to 0.040 in. of material was left on the test specimen radius during heat treatment to prevent decarburization of the final surface layer. Finish grinding and lapping operations followed to obtain a specimen dimension of 0.9525 cm (0.375 in) diameter by 8.25 cm (3.25 in) long and a surface finish between 0.1 and 0.2 μm (4 to 8 μin.) Ra (roughness average). A minimum hardness of Rockwell C 58 was obtained for each test specimen.

Figure 1 illustrates the microstructure of sample number 20s, viewed normal to the direction of hot rolling, and is typical of the other samples as well as hardened 440C steel in general. Figure 2 shows a view of the same sample parallel to the direction of hot rolling. A minor amount of carbide banding can be noticed traveling in the direction of hot work. This carbide banding is less noticeable than that typically seen in 440C components. Carbide banding is dependent on several factors including amount of hot work, size to which material was hot worked, and location of microstructural sample within the worked material. As an example, microstructure carbide banding from a STS 27 VIM/VAR 440C failed bearing indicated much heavier banding than that seen in sample 20s. It is considered advantageous to have low amounts of carbide banding.

B. Surface Modifications

The ion implantation was performed under contract at two separate facilities. One facility was a university laboratory at Georgia Institute of Technology, the other facility was Spire Corp., a commercial surface modification house. Three of the samples implanted by Spire were held stationary during implantation, until the necessary hardware was developed to rotate the specimens during implantation. These stationary samples are noted in Table 3. The specimens modified by Georgia Tech have a "g" suffix, while those specimens modified by Spire have an "s" suffix.

The titanium nitride coatings were also applied under contract by Georgia Tech. They were applied using a reactive sputtering technique. This involves sputtering pure titanium from a target in a vacuum chamber with a partial pressure of both argon and nitrogen gas. The partial pressure of nitrogen being approximately one tenth that of the argon. As the titanium is coated onto the substrate it reacts with the partial pressure of nitrogen in the chamber to create titanium nitride. Total pressure in the chamber is about 10 milliTorr. The coatings were applied to thicknesses of between 0.1 and 0.8 μ. Ideally, coatings should be applied in a stoichiometrically equal nitrogen/titanium compound which is bright gold in appearance.

C. Rolling Contact Fatigue Testing

The RCF tests conducted in this investigation were performed using a disc on rod "GE" type test rig. The specific rig used in this test series was manufactured by Polymet Corp. A short description of the tester follows, and a more detailed description can be found in Reference 10.

Testing consisted of loading two opposed hardened M50 discs against the test specimen to a load of 326 lb. This load resulted in a maximum Hertzian contact stress of approximately 700,000 psi, calculated assuming elastic deformation. The test specimen was driven through a precision collet by a high speed motor at 10,000 rpm. The loading discs were through-hardened M50 steel with a hardness of RCH 63. The diameter was approximately 7 in. and the width was 0.5 in., with a 0.25-in. radius on the cross-curvature loading edge. The loading discs were to be reused until the loading surface became degraded either by spalling, wear, or pitting. At this point the discs were removed and sent out to regrind the 0.25-in. edge radius. The users manual recommended regrinding of the active surface after about 40 tests. Regrinding of the loading discs occurred after approximately every 20 tests to ensure a good and relatively equivalent disc surface for all tests.

Jet turbine engine oil lubricant (Mil-L-7808G) from the same lot was dripped into the contact region at a rate of between 4 to 8 drops per every 10 sec. The oil was reused about six times in this test series. When a spall occurred in the test sample wear track, an accelerometer would pick up the increased vibration and shut the test off. Cycles to failure were recorded from a meter which measured each revolution of the shaft. There were two stress cycles for every test specimen revolution.

The number of tests per test specimen ranged from 4 to 9. This depended on the length of the rod and the amount of the rod which had a modified surface.

RESULTS

The test data was entered compiled and stored using a Weibull computer plotting program [11]. Data for the individual test samples are given in Table 3, including sample description, hardness, B10 life, Weibull slope, and number of failures. To determine confidence levels, a technique from Reference 12 was used. Individual test specimen data were compared to the baseline data, with an increased life having a corresponding confidence in the probability that the test sample has a greater fatigue life than the baseline. This analysis was performed for both the mean and the B10 lives and is given in Table 4.

Baseline data for this report was generated from nine test specimens, all of the same lot and manufactured at the same time. A total of 66 separate tests make up the baseline which allows for a high degree of confidence in determining actual increases in RCF life. There were no suspensions in generation of the baseline for this report. The Weibull plot for the baseline with all 66 failures plotted is shown in Figure 3.

Five test tracks of test sample number 15s (N ion implanted) are shown in Figure 4. Four spalls can readily be seen on this section of the test specimen. The ion implantation is delineated by the dark to lighter region. Figures 5 and 6 show titanium nitride sample number 30g. This is a poor coating, evidenced by the lack of any coating left in the wear track. This coating appears extremely brittle with very little adhesion. Figure 6 is a closeup of this sample near a Rockwell "C" hardness indentation. Plastic deformation occurred around the indentation by material mounding up around the brale indentor. This can clearly be viewed in a scanning electron microscope. The coating has spalled and flaked off in and around this indentation where deformation of the substrate has occurred. Rockwell "C" hardness indenting is a good, inexpensive, and fast test to determine coating adhesion.

In contrast to sample number 30g in figures 5 and 6, the titanium nitride coated sample number 27g is shown in Figures 7 and 8. Although the coatings are comparable in thickness and color, the adhesion of the two coatings is vastly different. There were no places on this test specimen where the coating had flaked or worn off leaving bare 440C substrate. Even the Rockwell "C" hardness indentations and surrounding material mounding had a continuous defect-free coating when viewed under optical microscopes. The only places where bare substrate is visible, is where spalls had occurred, the coating going off with the small piece of spalled substrate.

Five test tracks and three spalls are shown in the view of baseline sample number 47 in Figure 9. Figure 10 is a profilometer trace of the five test tracks exhibited on the baseline sample in Figure 9. This trace was taken using a contact stylus profilometer tracing down the length of the test specimen in the axial direction. The test track closest to the rod end is shown falling in elevation compared to the rest of the test tracks. This is due to excessive grinding and/or honing of the diameter near the test specimen end. Plastic deformation of the 440C substrate is evident in the trace by the raised areas on either side of the test tracks. The test tracks appear to have a higher amount of slightly raised deformation symmetrical on either side of the track center. The test tracks exhibit a mixture of wear and plastic deformation resulting in the depressed regions.

An Auger analysis was performed on a RCF test specimen nitrogen ion implanted as shown in Figure 11 [13]. The classic concentration distribution curve of an implanted species is shown by following the nitrogen curve. Carbon and chromium appear displaced by the nitrogen atoms in the 200 Å to 1200 Å region. This is shown by a decrease in carbon and chromium in the same region where nitrogen has the greatest concentration. The peak nitrogen concentration is at approximately 700 Å depth into the substrate. This indicates that ion implantation is a very shallow modification to a substrate surface.

DISCUSSION OF RESULTS

Ion implantation as a modification to the surface was expected to create a compressive stress in the near surface region. The surface compressive stress is caused by several factors, including damage to the metallurgical lattice making it less compact, and the forcing of atoms into interstitial and substitutional lattice sites depending on the implanting species causing additional strain to the lattice. This compressive stress would delay the initiation and subsequent crack growth to spallation of RCF [14].

The data as presented in Table 4 shows that none of the ion implanted test specimens had a statistically significant increase over the baseline. Statistical significance is set at 95 percent for this discussion.

Out of 14 ion-implanted test specimens, three gave an increase in mean life over the baseline, the highest confidence being 87 percent which is approaching statistical significance. While, nine of the 14 gave an increase over the baseline B10 life, although the highest confidence was only 67 percent. It is interesting to note that fewer samples showed a positive trend at the mean life than at the B10 life, even when it is less stringent to achieve significance at the mean. It appears that with additional testing (larger DOF), a significant improvement of the mean life might be realized from samples 09s and 06g, which were ion implanted with nitrogen and boron respectively. This is assuming the same magnitude of improvement with many more tests for these two samples.

It is the authors considered opinion, gained over several years experience of RCF testing ion-implanted modified test specimens exposed to various elements, fluences and energies, that the potential for significant RCF improvements of ion-implanted test specimens is slight. This is mainly due to the very thin layer which is modified in the ion implantation process, only 0.1 μm (4 μin.) deep. This thin layer is unable to support large changes in compressive stress to meaningful depths seen in RCF. Most of the Weibull curves of tested ion implanted specimens have been within a band around the baseline curves, not exceeding a confidence of 95 percent.

Of the thin film coated test specimens, two of them showed promise, with specimen 07g being statistically significant over the baseline mean RCF life. The B10 life was not as promising partly due to two suspensions which did not have the true mathematical effect on the Weibull line as failures would. Thin hard films have great potential for improving RCF life. The major drawback is consistency in coatings from one source to another and even from the same source. Additional work needs to be done to properly identify the process parameters, to what degree they affect the coating mechanical properties, and to control them during the coating process.

Some of the test specimens were overheated during processing as shown by a low hardness reading. Test specimens with a Rockwell "C" hardness of 56 or lower showed poor performance and had no chance of providing improvements. However, the only test specimen in the group to show statistical significance was softened from pre-processing hardness of over RCH 58 to RCH 56.8; evidently some softening is not harmful and may even be beneficial by increasing RCF life as well as other properties such as fracture toughness.

CONCLUSIONS

The ion implantation process using GE type RCF testers provided no statistically significant improvement in rolling contact fatigue over the baseline.

A 0.2-μ titanium nitride coated test specimen exhibited a statistically significant improvement in rolling contact life over the baseline. Hard thin films show potential for showing large increases in RCF life with the correct processing.

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TABLE 1. NOMINAL COMPOSITION OF 440C (W/o)

Fe	C	Cr	Mo	Mn	Si	P	S
Bal.	0.95-1.20	16-18	0.75	<1	<1	0.04	0.03

TABLE 2. 440C HEAT TREATMENT

A. Austenitize at 1930°F ± 30°F for 1 hr at temperature

B. Quench in 120°F to 150°F oil

C. Temper 1 hr minimum at 325°F +25°/–0°F

D. Cool in air to approximately 70°F

E. Cold soak in liquid nitrogen for 30 min

F. Temper 1 hr minimum at 325°F +25°/–0°F

TABLE 3. HARDNESS AND FATIGUE LIFE DATA FOR TESTED SAMPLES (EM IS A SUBSTITUTE FOR x10^{17} IONS/cm^2)

No.	Sample Description	RCH	B10 Life	Slope	No. of Failures
01	Baseline	58.5	2.17	2.0	7
05	Baseline	58.8	1.70	2.4	6
23	Baseline	58.6	1.93	4.0	6
24	Baseline	?	2.70	7.0	4
25	Baseline	?	1.11	2.7	5
26	Baseline	58.6	2.05	4.0	8
31	Baseline	?	1.49	2.6	9
46	Baseline	58.3	1.81	3.9	8
47	Baseline	58.4	2.31	5.3	8
02g	N implant, 2EM, 0.5 W/cm^2	57.0	2.20	5.8	6
03g	N implant, 3EM, 0.5 W/cm^2	57.5	2.18	3.7	7
04g	N implant, 2EM, 0.5 W/cm^2	56.0	1.73	5.9	6
06g	B implant, 0.4EM, 0.1 W/cm^2	?	2.09	2.8	5
32g	Ti implant, 2EM, 0.2 W/cm^2	?	1.08	3.1	5
09s	N implant, 2EM, 40 keV	58.2	1.35	1.9	7
11s	Cr implant, 2EM, 80 keV	56.0	1.55	3.5	6
13s	Ti implant, 2EM, 40 keV	57.9	2.26	9.0	5
15s	N implant, 1EM, 40 keV	58.2	1.97	6.6	6
17s	Ta implant, 1.6EM, 150 keV	58.1	2.11	4.9	8
18s	Cr implant, 2EM, 150keV	57.9	2.05	5.7	8
20s	Cr implant, 2EM, 80 keV. 0.5μ Mo	58.9	1.96	4.0	5
21s	Ar implant, 0.5EM, 0.05μ Mo	58.3	2.03	4.4	6
39s	C implant, 2EM, 50 keV	?	0.83	1.8	5
07g	TiN ion plate 0.2 μ	56.8	1.82	2.7	6 2SUSP
08g	TiN ion plate 2.0 μ	?	0.52	1.5	4
27g	TiN ion plate 0.8 μ	55.9	0.91	2.2	8
28g	TiN ion plate 0.15 μ	58.6	0.82	3.9	8
29g	TiN ion plate 0.1 μ	56.2	2.51	4.1	6
30g	TiN ion plate 0.8 μ	55.4	0.57	1.7	5
	Master Baseline, N=66	NA	1.83	3.1	66

Note: B10 life is in millions of stress cycles at 700,000 ksi. Slope is of the Weibull plotted least squares fit regression line. RCH is the Rockwell "C" hardness taken at three spots around the circumference. Sample 07g had 2 suspensions due to long run time. Samples 09s, 11s, and 13s were stationary during implantation and rotated approximately 60° between implantation sessions.

TABLE 4. COMPILATION OF RCF CONFIDENCE DATA AT THE MEAN AND B10 LIVES

Sample No.	Total DOF	Mean Life	Mean Ratio	Mean Confidence (%)	B10 Ratio	B10 Confidence (%)
02g	325	3.02	<1	<50	1.20	67
03g	390	3.69	1.08	70	1.19	66
04g	325	2.36	<1	<50	<1	<50
06g	260	4.10	1.20	85	1.14	64
32g	260	2.00	<1	<50	<1	<50
09s	390	4.04	1.2	87	<1	<50
11s	325	2.70	<1	<50	<1	<50
13s	260	2.76	<1	<50	1.23	67
15s	325	2.61	<1	<50	1.08	60
17s	455	3.08	<1	<50	1.15	67
18s	455	2.85	<1	<50	1.12	66
20s	260	3.19	<1	<50	1.07	57
21s	325	3.10	<1	<50	1.11	60
39s	260	2.64	<1	<50	<1	<50
07g	455	10.08	2.96	99	1.00	50
08g	195	1.91	<1	<50	<1	<50
27g	455	2.48	<1	<50	<1	<50
28g	455	1.33	<1	<50	<1	<50
29g	325	3.98	1.17	88	1.37	76
30g	260	1.89	<1	<50	<1	<50
baseline	65	3.41				

Note: The total degrees of freedom (DOF) is the DOF of the baseline multiplied by the DOF of the individual sample. Mean life is the arithmetic average of the failure and suspension times and is in millions of stress cycles. Mean ratio is the ratio of the baseline mean to the individual test sample mean. The B10 ratio is achieved in a similar manner. Mean confidence (or B10 confidence) gives the confidence that the individual sample has a greater life than the baseline at the mean level (or B10 level). A confidence level of 95 percent is considered statistically significant.

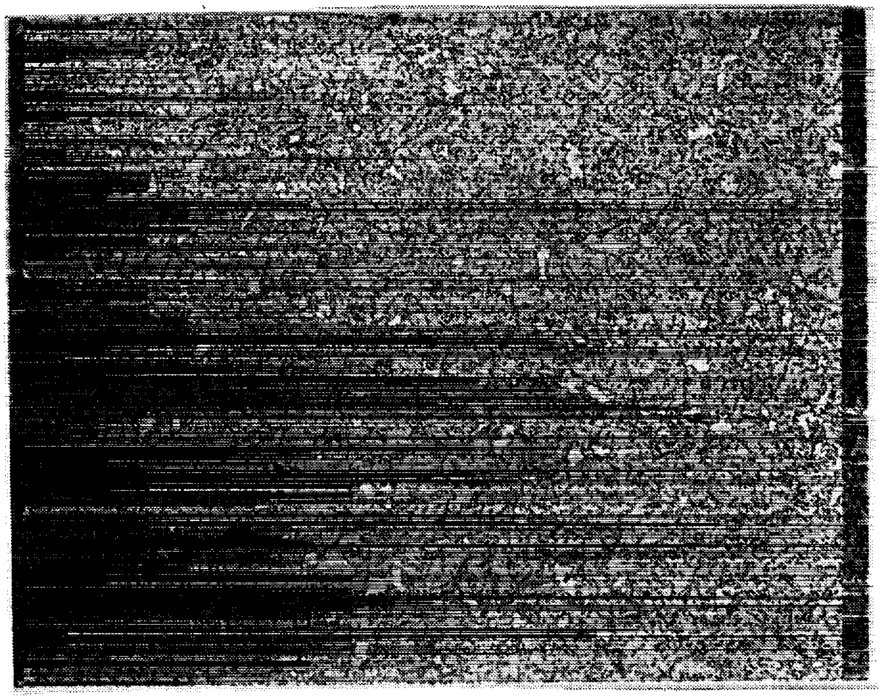

Figure 1. 440C microstructure normal to direction of hot rolling (400X).

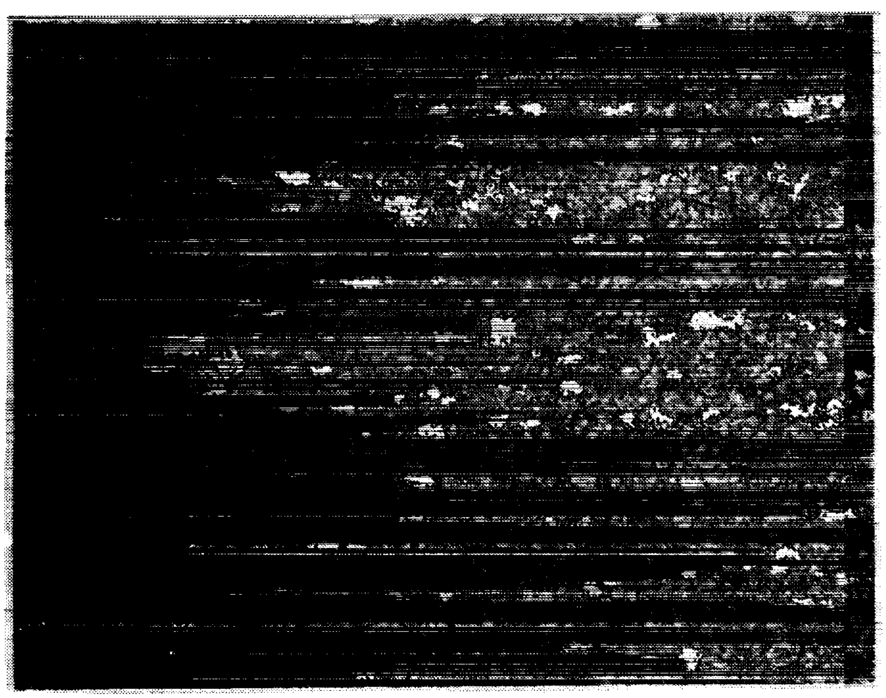

Figure 2. 440C microstructure parallel to direction of hot rolling.
Notice slight amount of carbide banding. (400X)

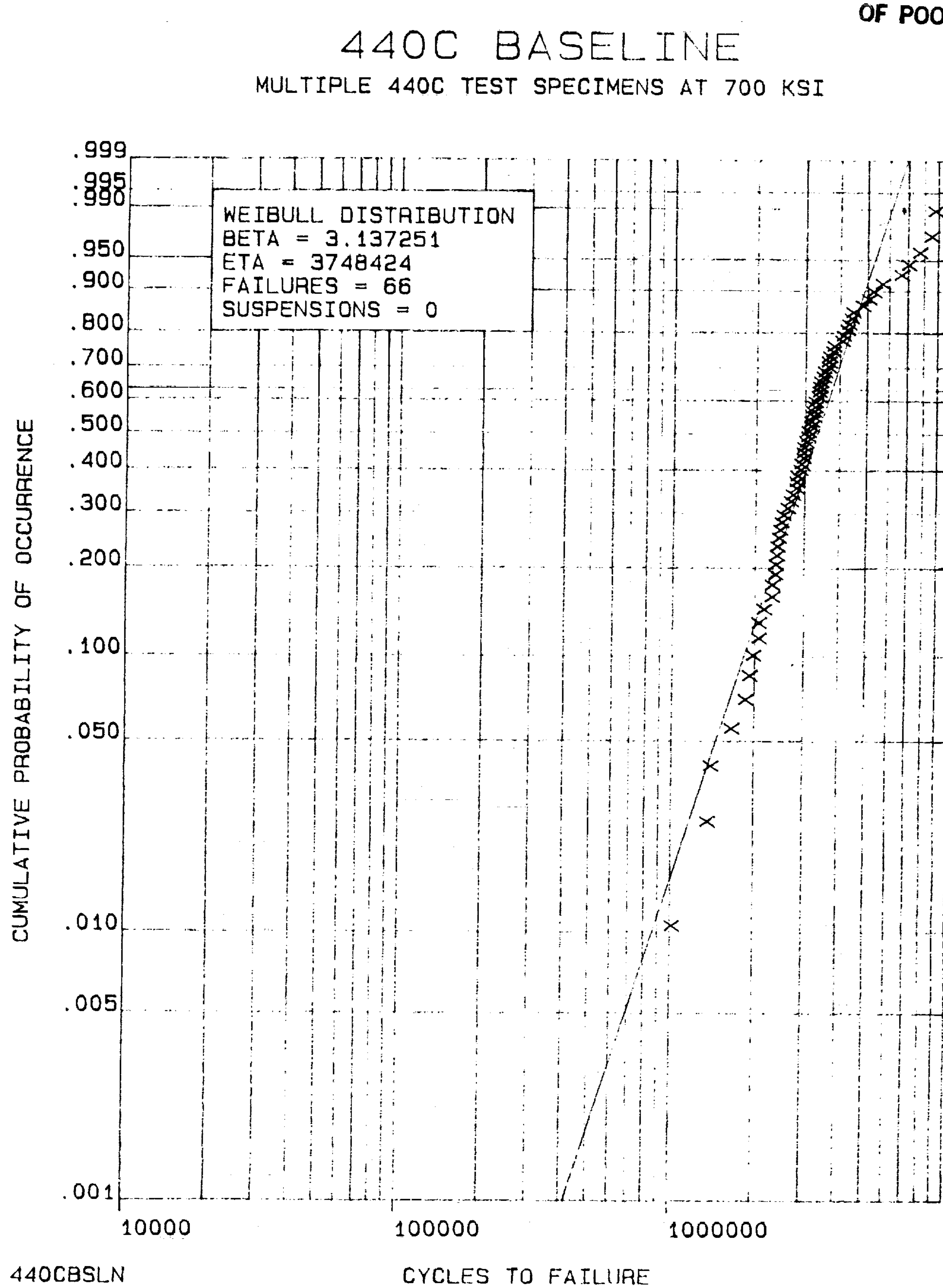

Figure 3. Weibull plot of 440C baseline data. Points are from five different test specimens.

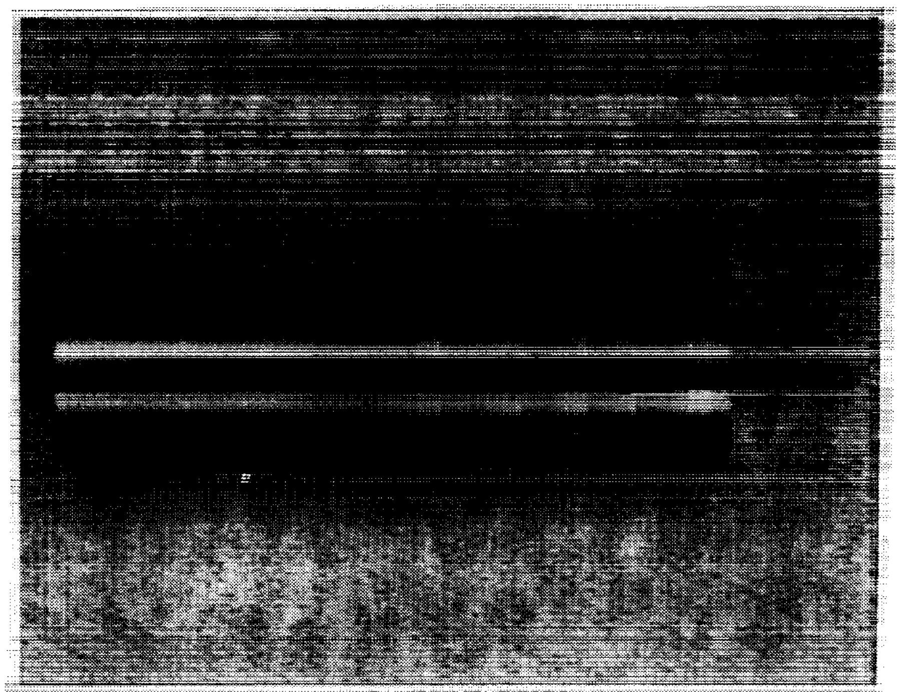

Figure 4. Test specimen 15s (nitrogen ion implanted, 1 x 10^{17} ions/cm^2, 40 kev) showing five test tracks with four of the five spalls in view. Area of ion implantation is slightly darker than unimplanted region.

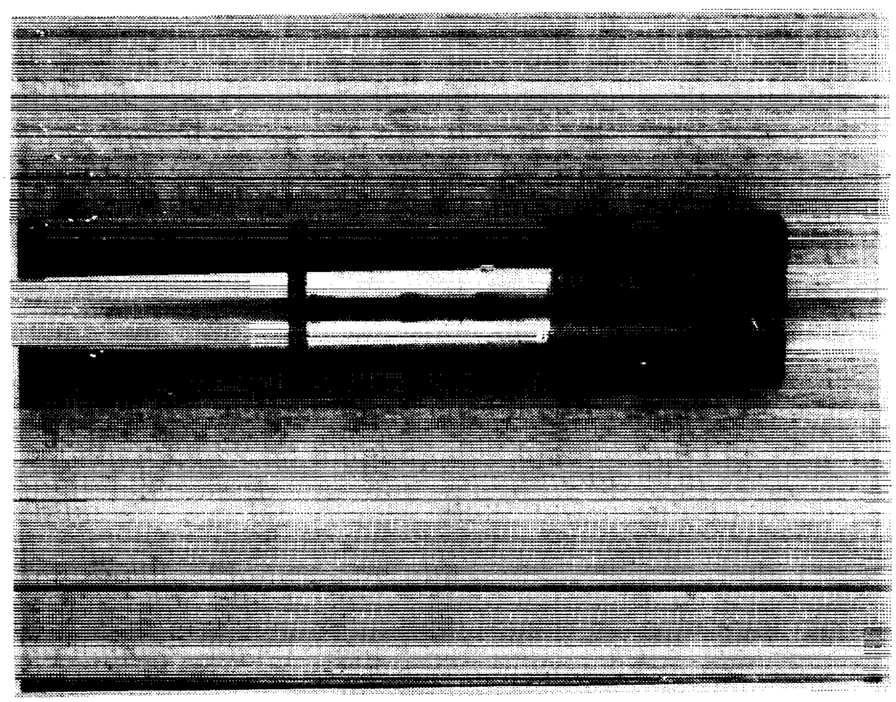

Figure 5. Test specimen 30g (0.8 μm thick titanium nitride) showing three test tracks, one track with a visible spall. Titanium nitride coating is a non-stoichiometric dark brown color. Extreme brittleness of coating on this sample is readily evident. Coating shows poor adherence in test tracks and even in area surrounding test tracks where no physical roller contact occurs.

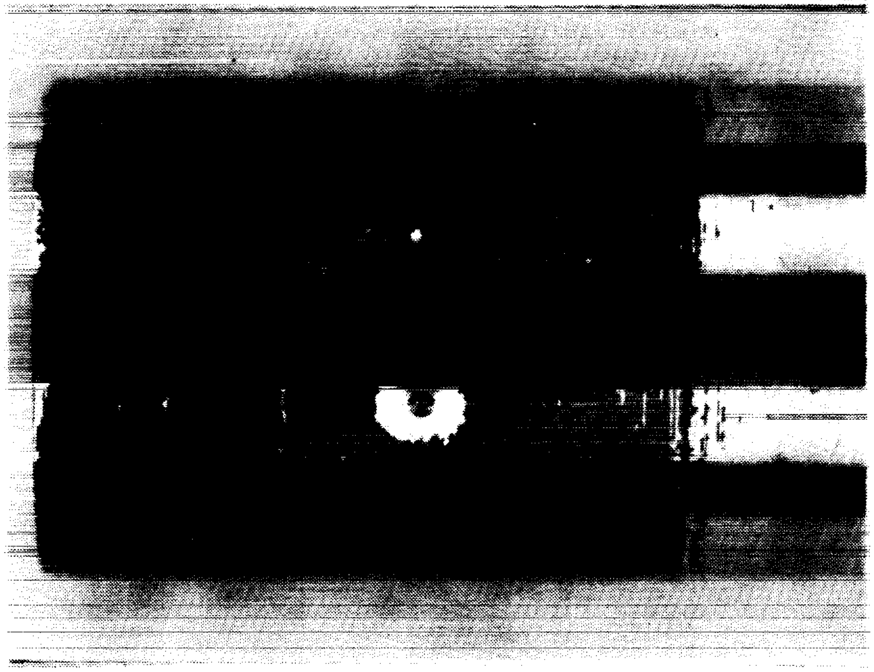

Figure 6. Test specimen 30g, showing Rockwell hardness indentation. 0.8 μm thick coating has cracked and flaked away from area in and around brale indentation.

Figure 7. Six test tracks are shown on this 0.8 μm thick titanium nitride test specimen (number 27g). Three of the six spalls are visible near the end. Note the large difference in apparent adherence between this sample and the one shown on Figures 5 and 6. Color of titanium nitride however is a dark brown similar to the color of the sample in Figures 5 and 6.

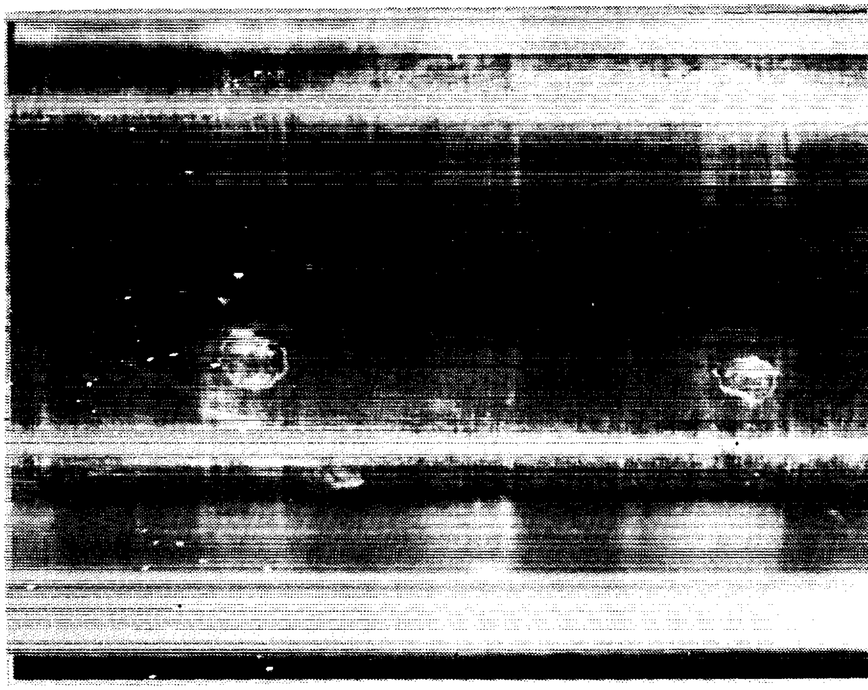

Figure 8. Close-up of previous sample, shown in Figure 7 (number 27g). This photo indicates the strong adherence of titanium nitride coating on this sample, particularly near the heavily damaged spalled areas.

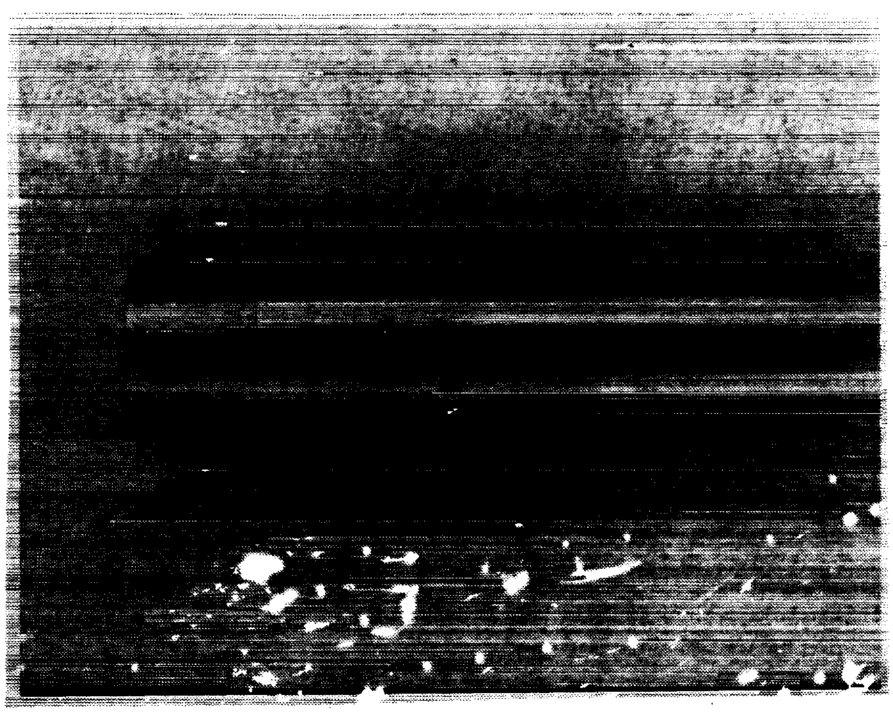

Figure 9. Baseline 440C test specimen number 47 showing five test tracks and three spalls.

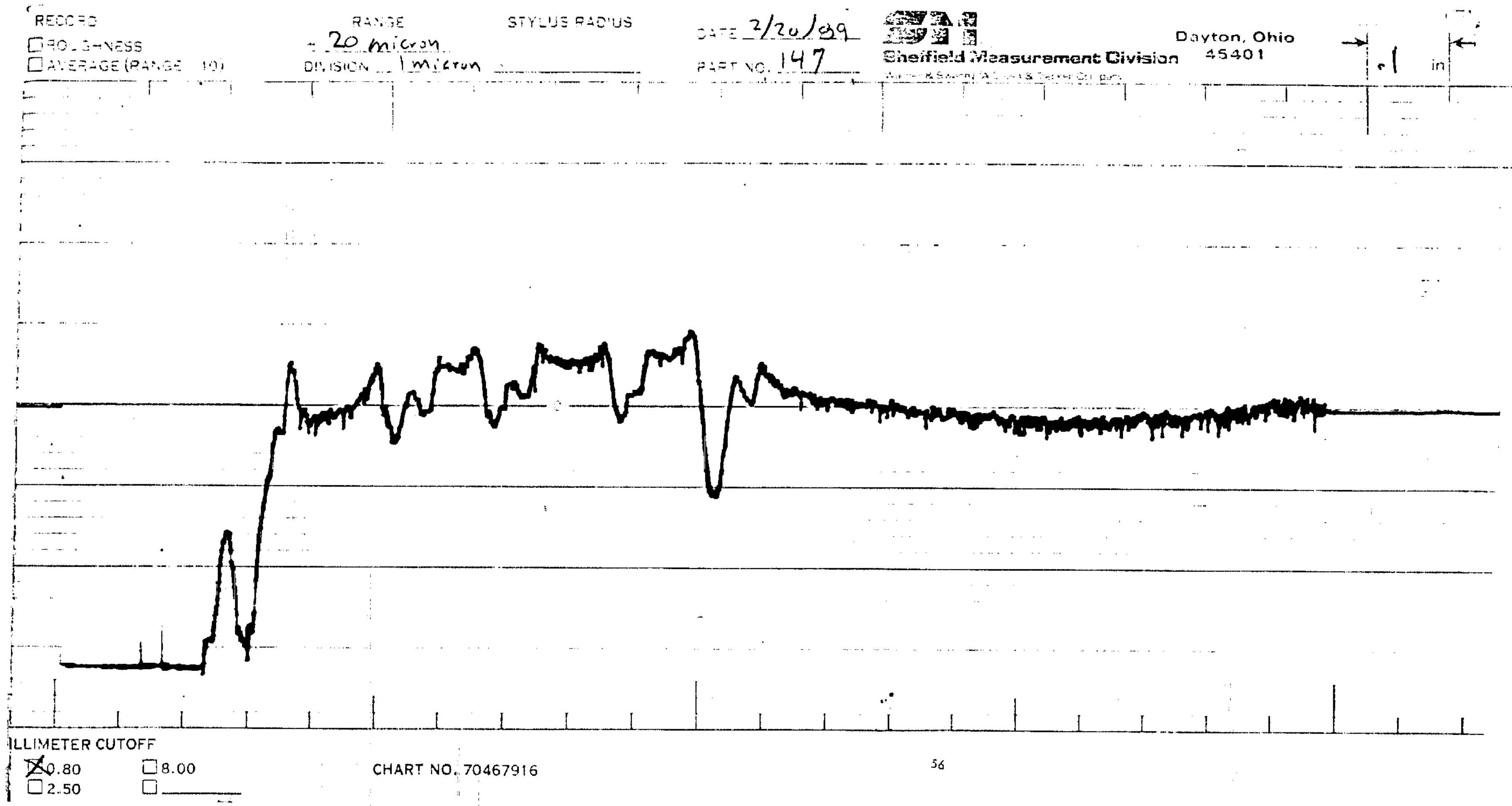

Figure 10. Profilometer trace of the five test tracks shown in Figure 9.

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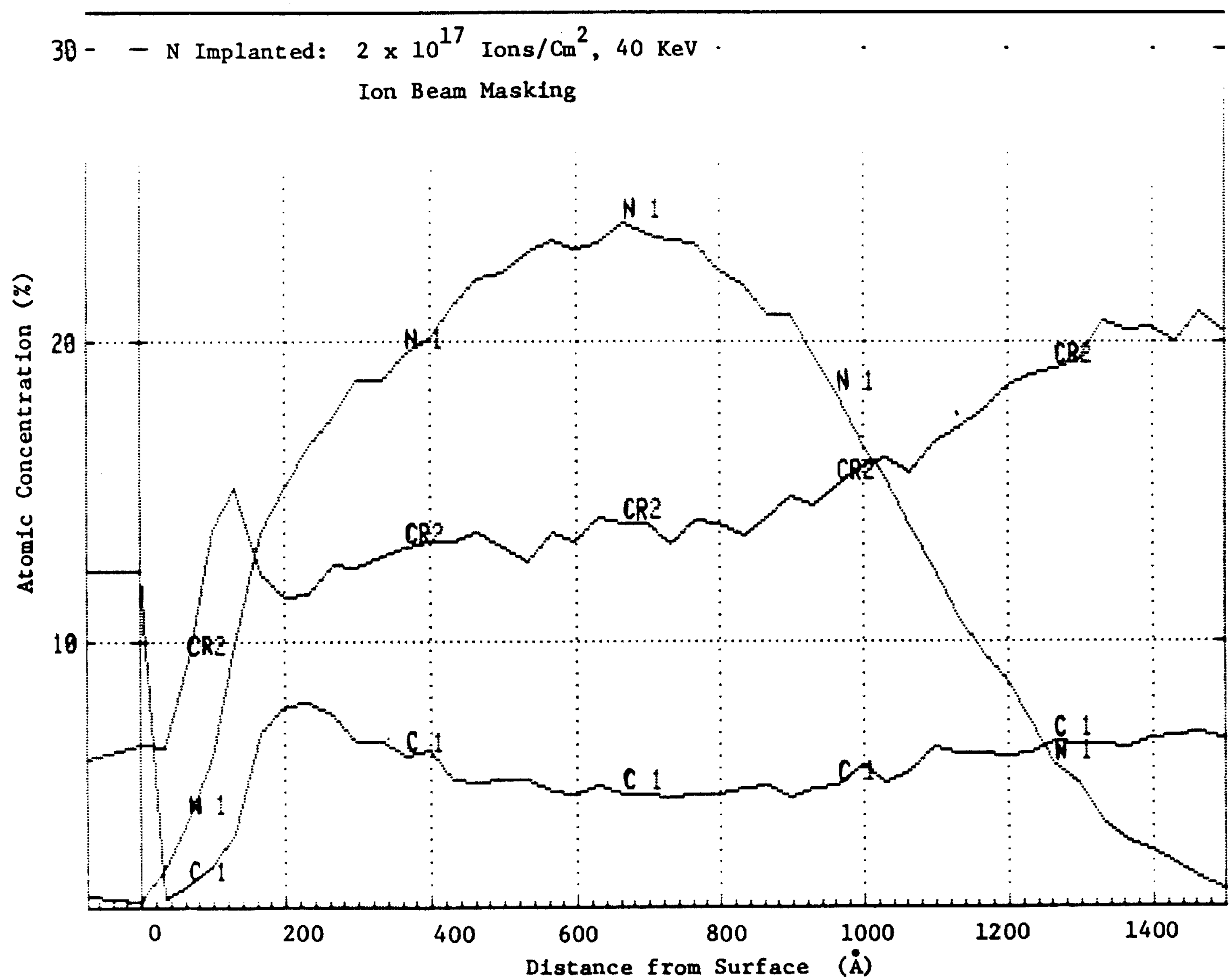

Figure 11. Auger concentration profile of nitrogen ion implanted test specimen (number 09s). Note depth of nitrogen is only about 0.14 μ (5.5 μin.).

APPROVAL

ROLLING CONTACT FATIGUE OF SURFACE MODIFIED 440C USING A "GE-POLYMET" TYPE DISC ROD TEST RIG

By Robert L. Thom

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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