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TESTING OF NUCLEAR GRADE LUBRICANTS AND THEIR EFFECTS ON A540
B24 AND A193 B7 BOLTING MATERIALS *

Carl J. Czajkowski
Brookhaven National Laboratory
Department of Nuclear Energy
Upton, New York 11973

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ABSTRACT

An increase in the number of bolting failures attributed to lubricant/coolant interaction at nuclear power plants has caused a great deal of concern regarding the more judicious use of lubricants by the nuclear power industry. An investigation was performed on eleven commonly used lubricants by the nuclear power industry. The investigation included EDS analysis of the lubricants, notched-tensile constant extension rate testing of bolting materials with the lubricants, frictional testing of the lubricants and weight loss testing of a bonded solid film lubricant. The report generally concludes that there is a significant amount of variance in the mechanical properties of common bolting materials; that MoS₂ can hydrolyze to form H₂S at 100°C and cause stress corrosion cracking (SCC) of bolting materials, and that the use of copper-containing lubricants can be potentially detrimental to high strength steels in an aqueous environment. Additionally, the testing of

various lubricants disclosed that some lubricants contain potentially detrimental elements (e.g. S, Sb) which can promote SCC of the common bolting materials. One of the most significant findings of this report is the observation that both A193 B7 and A540 B24 bolting materials are susceptible to transgranular stress corrosion cracking in demineralized H₂O at 280°C in notched tensile tests.

1.0 INTRODUCTION

In the first quarter of 1983, the United States Nuclear Regulatory Commission (U.S. NRC) reported [1] 44 distinct instances of bolting degradation at nuclear power plants between October 1964 to March 1982. These 44 occurrences were broken down into the following subclassifications: 19 involved a stress corrosion cracking (SCC) mechanism, 13 were attributed to boric acid wastage corrosion, 3 were fatigue induced, 1 instance of erosion-corrosion and 8 additional incidents involving material related

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problems or where apparent cause could not be identified. It is clearly obvious that the largest single cause of bolting degradation was SCC. (At least two [2,3] analyses involving SCC of fasteners attributed the failure to a lubricant/ moisture interaction which resulted in a corrosive environment.)

It was this lubricant/moisture interaction which was of sufficient concern to the Office of Nuclear Regulatory Research (ONRR) of the U.S. NRC to provide funding to Brookhaven National Laboratory (BNL) for an investigation into the effects of lubricants on fastener degradation.

The program consisted of:

- 1) EDS chemical analysis of the lubricants.
- 2) Steaming tests of MoS₂.
- 3) Coefficient of friction tests.
- 4) Notched tensile constant extension rate tests.
- 5) Weight loss tests.

2.0 CHEMICAL ANALYSIS OF THE LUBRICANTS

For purposes of this program, a total of 11 lubricants were used. Some tests were also performed on a bonded solid film lubricant. (Note: due to time restrictions and other constraints, only one type of coating was tested, and these results should not be extrapolated to other types of coating without additional testing).

These 11 lubricants were types commonly used in construction and post operational lubrication at nuclear power plants. The 11 lubricants tested were numbered one through eleven and were classified as follows:

Lubricant #	Type
1	Chemically pure MoS ₂
2	Commercially available MoS ₂ spray
3	Commercial MoS ₂ spray (different manufacturer)
4	Commercial MoS ₂ spray (3rd manufacturer)
5	Graphite in isopropanol
6	Graphite in ammonia
7	Nickel + graphite lubricant
8	Copper + graphite lubricant
9	Anti-seizing lubricant
10	Nickel based never seizing lubricant
11	Never seizing lubricant

Each of these lubricants was either sprayed or smeared onto carbon blocks and then loaded into a scanning electron microscope (SEM) and subjected to energy dispersive spectroscopy analysis of constituents.

(Note: EDS will only discern elements with atomic numbers greater than Na so certain light elements will not be detected). The scans for the majority of lubricants displayed the expected result with three notable exceptions.

Figure 1 is an EDS scan of a commercially available MoS₂ (lubricant #2) spray. The scan detected both antimony and titanium in addition to the molybdenum and sulfur. The titanium is possibly used for coloring. The significance of the antimony addition is unknown.

The EDS scan of lubricant #4 which is another moly-lube spray is shown in Figure 2. This scan showed only a sulfur peak with no molybdenum present at all.

Lubricant #5, which is a graphite and isopropanol lubricant, was subjected to EDS and the resultant scan is shown in Figure 3. This lubricant showed a high sulfur peak with a trace of silicon also present. The presence of sulfur would not be normally anticipated in a graphite + alcohol lubricant and might be detrimental to highly alloyed steels under the proper conditions.

The bonded solid film type lubricant was not subjected to EDS analysis due to significant outgassing of the material when subjected to an electron beam. The manufacturer's reported chemical analysis limit of this lubricant are therefore included:

Fluoride	29 ppm
Chlorine	200 ppm
Sulfur	98 ppm
Lead	2 ppm
Mercury	1 ppm
Arsenic	0.05
Zinc	1 ppm

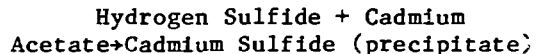
It can be clearly seen from the different EDS scans that there is a significant variance in the chemical analysis of even similar type lubricants. The appearance of possibly detrimental elements in lubricant #2 (antimony) and #5 (sulfur) plus the total absence of molybdenum in the moly-lube type lubricant #4 give evidence that independent chemical analysis of lubricants prior to their general use in critical applications would be a prudent course of action.

2.1 Steaming Tests on MoS₂

Prior BNL reports [2, 4, 5] had referenced various papers and reports which showed that in moist conditions, MoS₂ could hydrolyze and form detrimental sulfides which could result in a SCC failure in low alloy steels.

These conditions involved frictional tests in flowing nitrogen/water mixtures. Since rubbing forces are only at work at the torquing down of a bolt and would not normally come into play during the bolts' service life; some steaming tests were performed to see if steam alone could interact with MoS₂ in order to form potentially detrimental compounds.

Three tests were performed in order to determine if a gaseous sulfide could be produced by steaming MoS₂. The first test was conducted in a stainless steel autoclave. For this test, distilled water was poured into the autoclave, after which a glass dish with chemically pure MoS₂ was suspended above the water; the autoclave head tightened down and the water then heated to 100°C. The autoclave head was vented into a plastic tube with a metering valve into a test tube of water and then into a test tube of cadmium acetate. The unbalanced chemical reaction of this experiment is:



The amount of cadmium sulfide produced is then weighed and sulfur also measured in the water after the experiment is completed. The results of the first experiment produced 0.889₃g of sulfur from the gaseous phase and 0.911 mg of sulfur in the H₂O. Since some sulfide experiments had been performed in this autoclave prior to this run of tests, two more tests were performed in a new glass dessicator. The test conditions of the second test were basically the same as the first experiment although this time the experiment was conducted with 71.10 gms of chemically pure MoS₂ placed on two glass dishes supported above 500 ml of distilled H₂O. The water was boiled on a hot plate for five hours, after which the amount of sulfur was measured in both the gaseous and liquid phases. A blank was also run of the experiment and

subtracted from the original results to take into account any sulfide which might be present in the surrounding air. This test produced 0.615 μg in the gaseous phase and 142.4 μg in the H_2O or approximately 2 ppm sulfur (total) produced from steaming 71.1 gms of MoS_2 .

The third experiment was conducted in the desiccator using 71.10 gms of MoS_2 under similar conditions as the second test. The only variation was that the surface area of the MoS_2 open to the steam was reduced to 614 mm^2 from 1486 mm^2 . This test produced 0.0 gms of sulfur in the gaseous phase and 0.351 mg of sulfur in the liquid phase after the blanks were subtracted.

These experiments show that the steaming of MoS_2 produces hydrogen sulfide (H_2S).

3.0 COEFFICIENT OF FRICTION TESTING OF THE ELEVEN LUBRICANTS

SCC requires three separate elements to be present, namely:

- 1) Susceptible material
- 2) Corrosive environment
- 3) Tensile stress

If any of the aforementioned elements are missing, SCC will not occur. In the case of bolting, the material and the environment may not be able to be altered, in which case the applied tensile stress (preload) may have to be altered (if design conditions allow) to keep the applied tensile stress below a critical level. Since preload is dependent on the coefficient of friction for a given lubricant a factor of 2-3 (some lubricants have ranges given) in this frictional coefficient may throw the applied stress into the critical range of SCC susceptibility.

It was felt that since the coefficient of friction can play such an important role in the potential susceptibility of a bolt to SCC; BNL should measure the coefficient of friction for these lubricants.

The technique used for these measurements was the "sled and bed" technique. The tests performed were similar to those outlined in ASTM D 1894-75 (Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting). Both static and kinetic measurements were made. Some of the lubricants were tested both wet (as sprayed or brushed) and dry (if the lubricants were of the air drying type).

The following are the results of these tests:

Lubricant #	Coefficient of Friction	
	Static	Kinetic
1a. dry	0.182	0.164
b. wetted with isopropanol	0.273	0.142
2a. dry	0.266	0.223
b. wet	0.250	0.149
3a. dry	0.223	-
b. wet	0.156	-
4a. dry	0.026	0.150
b. wet	-	0.130
5a. dry	0.130	0.160
b. wet	0.185	0.190
6 . dry	0.351	0.170
7 . wet	-	0.149
8 . wet	0.055	0.126
9 . wet	0.093	0.171
10 . wet	0.042	0.296
11 . wet	0.049	0.268

The results show that the coefficients of friction, for even similar types of lubricants, vary widely. For example, the static values for lubricants 1-4, (All purportedly molybdenum disulfide-based lubricants), ranged from 0.026 to 0.273 dependent upon manufacturer and condition (wet or dry).

4.1 Notched Tensile Testing

In order to ascertain if bolting materials (specifically A193-B7 and A540 B24 Class 2) would have an environmental interaction with any of the lubricants and steam, constant extension rate tests (CERT) were performed at 100°C and 280°C.

Since ordinary tensile tests on smooth specimens will not indicate whether a material is prone to brittle fracture in the presence of a stress concentration (similar to the threads on bolts), it was decided that a notched tensile specimen would be used. The specimens had a 50% diameter notch machined into them.

The fracture face of each of the specimens was examined by SEM after the CERT. Prior to examination by SEM, the specimens were electrolytically cleaned to remove the oxide film.

This investigation involved a total of 62 notched tensile CERT. In addition to 44 tests on A193 B7 and A540 B24 materials; 18 tests were conducted on both A193 B7 and A540 B24 Cl.2 material coated with a bonded solid film lubricant [Table 1].

All of the tests were conducted in a stainless steel autoclave. Approximately 1000 ml of demineralized H₂O was added to the autoclave and then the specimen loaded above the water. The temperature of the solution was raised to test temperature (either 100°C or 280°C) through the use of resistance heating coils. After the required temperature was stabilized (approximately 30 minutes) the test pull was commenced. When the lubricant tests were conducted, the lubricants were either sprayed on or brushed onto the notched area of the specimen. All specimens were tested at a strain rate of approximately $5-9 \times 10^{-7}$ sec.⁻¹.

4.2.1 A540 B24 Class 2 Tests

The first six specimens were tested, at 100°C in demineralized H₂O with no lubricants added and at a

strain rate of approximately $5.0-9.0 \times 10^{-7}$ sec.⁻¹. Ductile fractures were seen in all of the SEM photographs (dimpled rupture). A surprising observation was the wide range of tensile strength results (195 Ksi - 277 Ksi) considering the fact that all of the A540 B24 specimens were machined from the same bar. On these specimens, there was no discernable difference in the fracture faces to account for the different strength levels.

The next three tests (Specimens #8, 9, 10) were also conducted at 280°C but exhibited a transgranular (quasi cleavage) area around the circumference of each specimen (Figures 4, 5). The tensile strength of these specimens ranged between 189 Ksi - 266 Ksi. This area of transgranular fracture would be indicative of a brittle fracture, probably induced by the environment.

Specimens #11 and 12 were tested at 100°C but had diametrically half of the notch coated with chemically pure MoS₂ lubricant #1. Both specimens exhibited a ductile overload failure, but differed in tensile strengths by almost 60 Ksi (#11-207 Ksi; #12-265 Ksi).

The next five specimens (tests #13-17) were all tested at 280°C with chemically pure MoS₂ on half of the notch. The fracture faces for these tests (specimens #13 and 14) had transgranular areas associated with them, indicating an environmental interaction. Specimens #15-17 all attained tensile values in excess of 240 Ksi and the fracture faces all exhibited a dimpled rupture appearance.

Specimens #18 and 19 were tested under the same environmental conditions as the prior five tests, except the strain rate was reduced by a factor of about 2. Both specimens attained very low notch tensile strengths and their fracture faces were primarily dimpled.

Specimens #20 and 21 were tested at 100°C and 280°C respectively with lubricant #2 sprayed on half of the notch. Both specimens attained notched tensile values in excess of 200 Ksi and both specimens had fracture faces exhibiting ductile failure.

The next five specimens #22-26 were all tested using lubricant #3. Specimens 22-24 were tested at 100°C and all attained notch tensile values in excess of 230 Ksi. The fracture faces for these specimens were all ductile in nature. Specimens 25 and 26 were tested at 280°C and both of these attained tensile values above 228 Ksi with dimpled fracture faces also evident.

Specimen #27 was tested at 280°C using lubricant #4. There was one area of transgranularity evident with the specimen attaining a value in excess of 220 Ksi.

Specimen #28 was tested with lubricant #5 (graphite + isopropanol) at 280°C. The fractographs of the fracture surface showed the failure to be ductile.

The fracture surface of Specimen #29 and #30 both showed ductile failures. These specimens were tested with lubricant #7 (nickel + graphite) at 280°C.

Specimens 31 and 32 were tested with the copper + graphite lubricant #8 at 280°C. Both specimens exhibited some transgranularity with #32 exhibiting only a 146.7 Ksi notched tensile strength. This specimen also had a circumferential crack running almost completely around the specimen. Specimens #33 and 34 were tested with lubricants #9 and 11 respectively at 280°C. Both specimens had dimpled rupture type fracture faces.

4.2.2 Al93 B7 Tests

The next ten specimens tested were machined from Al93 B7 bolting material.

Specimen #35 was tested in demineralized water at 100°C. The

specimen attained a notched tensile strength of 230.7 Ksi and showed a ductile failure.

Specimens #36-39 were all tested in demineralized water at 280°C. There was a large range of notched tensile values attained (114 Ksi - 232 Ksi). Transgranular areas were noted on specimens #38 and #39 while the fracture faces for the other specimens were predominantly ductile in appearance. The transgranular areas plus the wide range of tensile strengths are evidence of the susceptibility of this material to SCC in the demineralized water environment at 280°C. The lowest tensile value obtained on specimen #36 with no discernible transgranularity at this time must be considered an anomaly.

Specimens #40-#43 were all tested using chemically pure MoS₂ (lubricant #1) on one half of the notch.

Specimen #40 was tested at 100°C and attained a notched tensile strength and exhibited a ductile fracture mode.

The fracture faces of the specimens tested at 280°C with chemically pure MoS₂ all exhibited areas of transgranular fracture. The appearance of these areas in addition to the fact that these specimens only attained notched tensile strengths of 171 Ksi - 198 Ksi denote an environmental effect on this fracture.

Specimen #44 was tested at 280°C with lubricant #8 applied to half of the notch. Portions of the fracture face were transgranular in appearance.

4.3 Bonded Solid Film Lubricant Tests

A total of 18 tests were performed on bolts coated with a bonded solid film lubricant in order to determine if the lubricant would have a beneficial effect on the SCC resistance of the bolting materials tested. Twelve of the tests were performed on A540 B24 Class 2 materials and six tests were performed on Al93 B7 bolting materials.

Specimens #45-49 were all tested at 280°C in demineralized water. The fracture faces of specimens #45, 47, 48, and 49 all showed ductile overload. The notch tensile strengths of these specimens ranged between 224 Ksi and 261 Ksi. Specimen #46 attained a notched tensile strength of only 171.2 Ksi and exhibited transgranular areas on the fracture face defining a SCC susceptibility under these conditions.

Tests were performed at 280°C with chemically pure MoS₂ on half of the notch on specimens #50-53. Specimen #50 attained a notched tensile strength of 203.8 Ksi and had transgranular areas in evidence on the fracture face. Specimen #51 showed an entirely ductile fracture. Specimen #52 had large areas of transgranular fracture and only had a measured notched tensile strength of 171 Ksi. The final MoS₂ test (Specimen #53) was basically ductile in appearance and attained a notched tensile strength of 228.9 Ksi.

Specimen #54 tested at 280°C with lubricant #8 showed no environmental effect with a ductile fracture evident.

Since some bolts removed from power plants may have been previously exposed to MoS₂ lubricants prior to the application of a bonded solid film lubricant, two specimens (both A540, B24 material) were steamed at 100°C with MoS₂ for 1 hour; then cleaned, and sent out for coating. The two specimens #55 and #56 were tested afterward in demineralized water at 280°C. Specimen #55 exhibited a ductile fracture face (Figure 6) and had a notched tensile strength of 260.9 Ksi. A 60 Ksi reduction in the notched tensile strength of specimen #56 was observed, as well as some transgranularity of the fracture face (Figure 7). This specimen appeared to have suffered SCC while specimen #55 did not.

The last six notched tensile tests were performed on A193 B7 material. Specimens #57-58 were tested at 100°C in demineralized water. Specimen #57 had

areas of transgranular fracture evident on the fracture face. Specimen #58 achieved a tensile value of 232 Ksi and had a ductile fracture in evidence.

Specimen #59 was tested in demineralized water at 280°C. It had an area of transgranularity on the fracture face and had a notched tensile strength of 216 Ksi.

Specimens #60-62 were all tested at 100°C with chemically pure MoS₂ (lubricant #1) used on half of the notch. Both specimens #60 and #61 had dimple ruptured fracture faces.

The last specimen (#62) was tested at 280°C with chemically pure MoS₂ on one half of the notch. The fracture face exhibited transgranular areas and had a notched tensile strength of 220 Ksi.

5.0 WEIGHT LOSS TESTING OF BONDED FILM LUBRICANT

As previously mentioned [1], the U. S. NRC documented bolting degradation experience in 1983. The second largest contributor to the bolting degradation problem was boric acid wastage corrosion. BNL investigated this problem for the U. S. NRC, Chemical Engineering Branch [10] (CMEB) by conducting a literature search and by performing confirmatory experiments of boric acid wastage corrosion as outlined below:

Previously published data [11] were accumulated by BNL on an AISI 4135 steel in H₃BO₃ and H₃BO₃ - KOH solutions at 70°F and 140°F. In addition, BNL work on A193-B7 and AISI 4130 material at higher temperatures was plotted to determine if extrapolations were valid. The temperature dependence of the graph (Figure 7) is evident. As temperature increases to the boiling point of water, the corrosion rate increases at much faster rates. This increase continues until the boiling point is reached and then

starts to diminish, probably due to the loss of water in solution. The decrease in corrosion rate continues through at least a temperature of 352°F in $H_3BO_3 + LiOH$. As the higher temperatures are attained, water of hydration would also start to evaporate, which would probably deposit a lithium salt of meta-boric acid $LiBO_2$ ($LiBO_2$ has a melting point as high as 845°C in non-hydrated form). Other corrosion products may have a similar stifling effect in the case of H_3BO_3 by itself, accounting for the drop in rate after H_2O is lost, although they have not been examined in any detail.

A total of nine specimens coated with the bonded solid film lubricant were subjected to the same tests as the bare metal specimens. Five specimens were tested at 100°C (3 for 132.5 hours and 2 for 77 hours). Two specimens were also tested at 352°F (178°C) for 120 hours and two specimens at 300°F (315°C) for 130 hours. The working solution used for the testing was 4000 ppm boron (as H_3BO_3) + $H_2O + LiOH$ titrated to a pH of 7.3.

The data generated from these tests are plotted on the graph of the original test data in Figure 8.

It is evident that the corrosion rate at 178°C and 100°C for the solid bonded film lubricant is almost an order of magnitude less than for the bare metal alone. As no data were generated for the $H_3BO_3 + LiOH$ solution at 315°C, the corrosion rate for an A193 B7 material in 4000 ppm boron as $H_3BO_3 + H_2O$ solution is plotted. The point almost coincides with the bonded lubricant point denoting virtually no additional protection offered by the lubricant, at this temperature.

6.0 DISCUSSION

There has been a tremendous amount of controversy over the use of molybdenum disulfide as a lubricant on nuclear power plant components. BNL's involvement over this issue was initiated during prior investigations of turbine disc failures [4,5]. During these investigations, a very extensive

literature search was performed on MoS_2 and its possible role as a SCC contributor. An investigation [12] performed as a result of the Hinkley Point "A" disaster found that MoS_2 could reduce the crack initiation time of turbine steels (similar to bolting materials) by a factor of three in a steam environment. BNL tests [5] reduced the notched tensile strength of a disc steel by a factor of 3.5 over a corresponding steam test.

Other investigations [13, 14] have concluded that MoS_2 accelerates corrosive tendencies of metallic materials in moist environments.

Some of the earliest work found during this literature search [15] showed that MoS_2 could hydrolyze in a moist environment to produce H_2S and that this oxidation reaction was reversible [16].

A report for the Royal Aircraft Establishment (U.K.) [17] cites accelerated corrosion of steel in contact with MoS_2 under conditions of high humidity. The formation products of this reaction are molybdenum dioxide and sulfuric acid. Sulfuric acid is very detrimental to low alloy steels.

At least five other investigations have attributed nuclear component failures to the possible use of MoS_2 [2, 18, 19, 24].

Work done by Atomic Energy of Canada Ltd. (AECL) [21] has shown that the following materials cracked under BWR conditions when contaminated by MoS_2 : sensitized 304SS, sensitized Inconel 600, Inconel 718, 17-4 PH in the H1025 and H1100 conditions, PH 13-8 Mo, Custom 455 and AM 355 SCT 1000.

The AECL work is substantiated by a G.E. Report [21] which documented stress corrosion tests on a number of materials with 12 different lubricants and partially concluded that " MoS_2 is the most aggressive lubricant. It readily cracked 17-4 PH

(1100) and cold worked 304SS. After longer exposures, even annealed 304 stainless steel cracked with this lubricant."

Recent work at Combustion Engineering [22] shows that MoS₂ can produce H₂S under PWR conditions.

The chemical instability of MoS₂ in aqueous media has been discussed in an EPRI report [23]. The report states that MoS₂ is not stable at pH values greater than 4.5. The stable compounds formed are HMoO₄⁻ or MoO₄⁻² with H₂S(aq.), HS⁻ or S⁻² also formed. These compounds could quite easily induce a sulfide SCC phenomenon in critical bolting applications where moisture is present and MoS₂ has been used.

The copper bearing lubricants have also been involved in at least one report as a crack initiator [21]. The particular lubricant used, (similar to this report's #8) produced cracks in 17-4 PH specimens. This result is complementary to BNL results where significantly lower notched tensile strength values were associated with the use of this lubricant. The SCC effect of copper on low alloy steels in as little as 1 ppm chloride solutions [25] has been observed. In this case, the copper is speculated to have shifted the potential into the cracking range in a CERT at 268°C. This potential shift might well be applicable to the lubricants.

7.0 CONCLUSIONS

1. The appearance of potentially detrimental elements in the chemical analysis (EDS scans) of the various lubricants clearly shows a marked difference in composition between supposedly similar (e.g. MoS₂ based) lubricants.

2. The steaming tests of the chemically pure MoS₂ show that in the presence of steam (100°C), MoS₂ will hydrolyze to form detrimental gaseous sulfides (H₂S).

3. The wide variation of measured coefficients of frictions for similar lubricants shows that generalizations

of this value for "same type" (e.g. MoS₂ based, graphite-based, copper-based or nickel-based) should not be made and that the coefficient of friction should be determined (or obtained) for the specific lubricant and substrate used.

4. The notched tensile tests of the bolting materials showed that both A540 B24 Class 2 and A193 B7 materials are susceptible to a SCC failure in steam at 280°C. The use of MoS₂ or a copper + graphite lubricant appear to enhance this susceptibility to SCC although this is not conclusive. The use of a solid bonded film lubricant does not significantly improve the bolting materials performance with either steam or MoS₂.

5. The variation in notched tensile strengths with no discernable difference on the fracture faces of the A540 B24 Class 2 material shows that bolts cut from the same rod may exhibit different mechanical properties. This observation is upheld by field experience and is adequate indication that more intensified inspections may be necessary on incoming bolting materials.

6. The weight loss experiments, using previously coated solid bonded film lubricant specimens, show a marked decrease in metal loss at 100°C and 178°C when compared to previously reported bare metal data. This lubricant protection disappears at 600°F (315°C), where the results on previously coated specimens differed little from results on bare metal specimens.

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9.0 REFERENCES

1. Koo, W. H., NUREG-0943, January 1983.
2. Czajkowski, C. J., NUREG/CR-2993, July 1982.
3. Burck, L. H., Foley, W. J., Report No. IE-123, April 1981.
4. Czajkowski, C. J., BNL-NUREG-28724, October 1980.
5. Czajkowski, C. J., BNL-NUREG-29964, March 1981.
6. Hudgins, C. M., McGlasson, R. L., Mehdizadeh, P., Rosborough, W. M., Corrosion, Vol. 22, August 1966.
7. Letter Report J. R. Weeks to D. Smith (U.S. NRC)-MTEB.
8. Telecon C. D. Sellers (U. S. NRC)-MTEB.
9. Cipolla, R. C., Cargill, R. L., Bersin, J. M., APTECH Report AES-8-08-79 dated May 1982.
10. Czajkowski, C. J., NUREG/CR-2728, March 1982.
11. YAEC-67, Westinghouse, November 1958.
12. Thornton, D. V., Mould, P. B., Patrick, E. C., Conference on Grain Boundaries, 1976 D13 (London: The Institution of Metallurgists).
13. Calhoun, S. F., Rock Island Arsenal Report #62-2752, August 15, 1962.
14. Perna, C. Picatinny Arsenal Report #DC3-1, January 1961.
15. Haltner, A. J., Oliver, C. S., Proc. ACS Pet. Div. Symp. on Chemistry of Friction and Wear, Vol. 3, No. 4, 1958, pp. A77-84.
16. Rowe, G. W., Sci. Lubn, Vol. 11, No. 10, 1959, pp. 12-15.
17. Kay, E., Wear, 12 (1968), pp. 165-171.
18. Goldberg, A., Streit, R. D., NUREG/CR-1884, November 1980.
19. Hall, J. F., Fink, G. C., Draft Copy of Combustion Engineering Report "Examination of #2 Steam Generator Cold Leg Primary Manway Cover Studs from Maine Yankee."
20. Private Communication - E. V. Murphy (AECL) 1983.
21. Rowland, M. C., Rose, T. C., G. E. Report, APED-4422, December 1963.
22. Hall, J. F., Presentation at EPRI Seminar, "Bolting Degradation or Failure in Nuclear Power Plants," November 1983.
23. Chen, C. M., Aral, K., Theus, G. J., EPRI NP-3137 Volume 1, Project 1167-2, Final Report, June 1983.
24. Jonas, O., Draft Paper, Corrosion/84 Paper #55.
25. Czajkowski, C. J., NUREG/CR 3614, December 1983.

TABLE 1

NOTCHED TENSILE CONSTANT EXTENSION RATE TESTS

SAMPLE NO.	TEMP.	(hrs.)	TOTAL EXTENSION (in.)	EXTENSION RATE (Sec ⁻¹)	TENSILE STRENGTH (psi)	ENVIRONMENT
		TEST DURATION				
1	100°C	5:48	.010	4.8×10^{-7}	195,678	water
2	100°C	-	.028		277,056	water
3	100°C	6:40	.015	6.25×10^{-7}	228,291	water
4	100°C	6:48	.015	6.1×10^{-7}	275,580	water
5	100°C	7:01	.0225	8.9×10^{-7}	273,134	water
6	100°C	6:22	.0225	9.8×10^{-7}	240,521	water
7	280°C	7:28	.026	9.6×10^{-7}	201,303	water
8	280°C	4:56	.0245	1.38×10^{-7}	195,678	water
9	280°C	5:03	.020	1.1×10^{-6}	189,971	water
10	280°C	5:46	.0185	8.9×10^{-7}	216,877	water
11	100°C	7:08	.020	7.0×10^{-7}	207,093	molybdenum disulfide
12	100°C	6:31	.023	9.8×10^{-7}	264,981	molybdenum disulfide
13	280°C	-	.0225	-	154,912	molybdenum disulfide
14	280°C	6:43	.0175	7.2×10^{-7}	171,218	molybdenum disulfide
15	280°C	7:40	.031	1.1×10^{-6}	242,152	molybdenum disulfide
16	280°C	7:12	.027	1.0×10^{-6}	269,058	molybdenum disulfide
17	280°C	7:13	.0365	1.4×10^{-6}	242,152	molybdenum disulfide

TABLE '1 CONT'D

SAMPLE NO.	TEMP.	(hrs.) TEST DURATION	TOTAL EXTENSION (in.)	EXTENSION RATE (Sec ⁻¹)	TENSILE STRENGTH (psi)	ENVIRONMENT
18	280°C	15:26	.031	5.58×10^{-7}	143,846	molybdenum disulfide
19	280°C	17:47	.0225	3.5×10^{-7}	119,853	molybdenum disulfide
20	100°C	7:14	.0225	8.6×10^{-7}	203,832	lube #2
21	280°C	6:46	.026	1.06×10^{-6}	228,291	lube #2
22	100°C	5:54	.0265	1.2×10^{-6}	232,368	lube #3
23	100°C	7:25	.028	1.0×10^{-6}	252,751	lube #3
24	100°C	6:50	.026	1.0×10^{-6}	240,521	lube #3
25	280°C	7:19	.025	9.0×10^{-7}	228,291	lube #3
26	280°C	7:41	.030	1.1×10^{-6}	238,891	lube #3
27	280°C	5:53	.0225	1.06×10^{-6}	220,138	lube #4
28	280°C	7:35	.0265	9.7×10^{-7}	244,598	lube #5
29	280°C	8:12	.030	1.02×10^{-6}	252,019	lube #7
30	280°C	-	-	-	-	
31	100°C	6:24	.025	1.08×10^{-6}	228,291	lube #8
32	280°C	5:40	.0235	1.15×10^{-6}	146,700	lube #8
33	280°C	8:05	.030	1.0×10^{-6}	244,598	lube #9
34	280°C	7:00	.0265	1.05×10^{-6}	244,598	lube #11
35	100°C	6:07	.0325	1.47×10^{-6}	230,737	water
36	280°C	6:19	.013	5.0×10^{-7}	114,146	water
37	280°C	7:03	.0175	6.9×10^{-7}	232,368	water

TABLE 1 CONT'D

SAMPLE NO.	TEMP.	(hrs.)	TOTAL EXTENSION (in.)	EXTENSION RATE (Sec ⁻¹)	TENSILE STRENGTH (psi)	ENVIRONMENT
		TEST DURATION				
38	280°C	4:49	.0015	8.64×10^{-8}	167,142	water
39	280°C	5:33	.0215	1.07×10^{-6}	220,138	water
40	100°C	5:33	.0265	1.3×10^{-6}	231,553	molybdenum disulfide
41	280°C	4:28	.02	1.2×10^{-6}	171,210	molybdenum disulfide
42	280°C	4:14	.0165	1.08×10^{-6}	179,372	molybdenum disulfide
43	280°C	5:04	.019	1.04×10^{-6}	198,124	molybdenum disulfide
44	280°C	7:00	.030	1.2×10^{-6}	256,828	lube #8
45	280°C	7:20	.0145	5.5×10^{-7}	244,598	water
46	280°C	14:32	.018	3.4×10^{-7}	171,218	water
47	280°C	14:43	.015	2.8×10^{-7}	224,215	water
48	280°C	14:01	.0325	6.4×10^{-7}	260,905	water
49	280°C	13:38	.021	4.3×10^{-7}	244,600	water
50	280°C	5:06	.018	9.8×10^{-7}	203,832	molybdenum disulfide
51	280°C	24:00	.032	3.7×10^{-7}	293,518	molybdenum disulfide
52	280°C	8:08	.022	7.5×10^{-7}	171,218	molybdenum disulfide
53	280°C	14:36	.0175	3.3×10^{-7}	228,291	molybdenum disulfide
54	280°C	7:09	.0075	2.9×10^{-7}	256,828	lube #8

TABLE 1 CONT'D

SAMPLE NO.	TEMP.	(hrs.)	TOTAL EXTENSION (in.)	EXTENSION RATE (Sec ⁻¹)	TENSILE STRENGTH (psi)	ENVIRONMENT
		TEST DURATION				
55	280°C	7:45	.022	7.87×10^{-7}	260,905	water
56	280°C	14:27	.013	2.5×10^{-7}	203,832	water
57	100°C	12:53	.0225	4.9×10^{-7}	216,061	water
58	100°C	12:01	.012	2.8×10^{-7}	232,368	water
59	280°C	13:30	.016	3.3×10^{-7}	216,061	water
60	100°C	12:51	.0125	2.7×10^{-7}	224,215	molybdenum disulfide
61	100°C	15:13	-----N.G.-----			molybdenum disulfide
62	280°C	12:50	.025 in.	5.4×10^{-7}	220,138	molybdenum disulfide

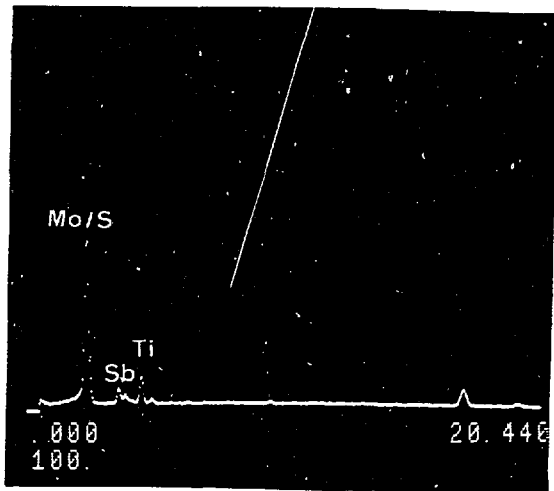


Figure 1 EDS scan of lubricant #2.

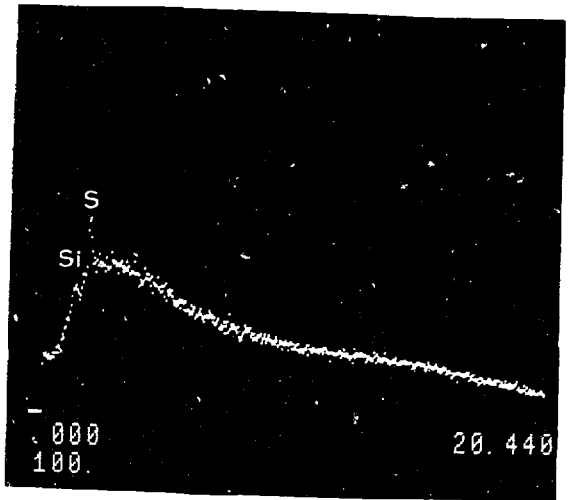


Figure 3 EDS scan of lubricant #5.

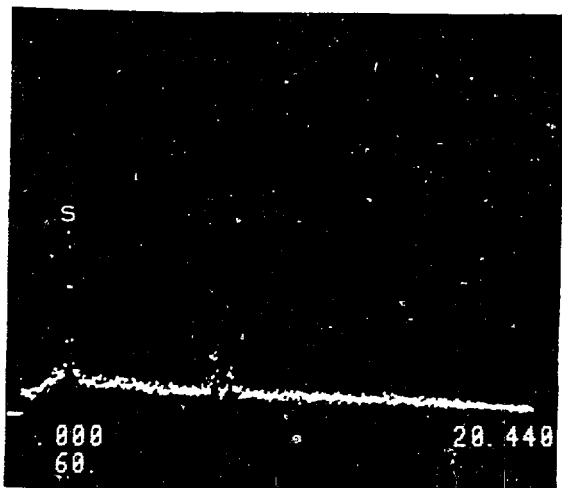


Figure 2 EDS scan of lubricant #4.

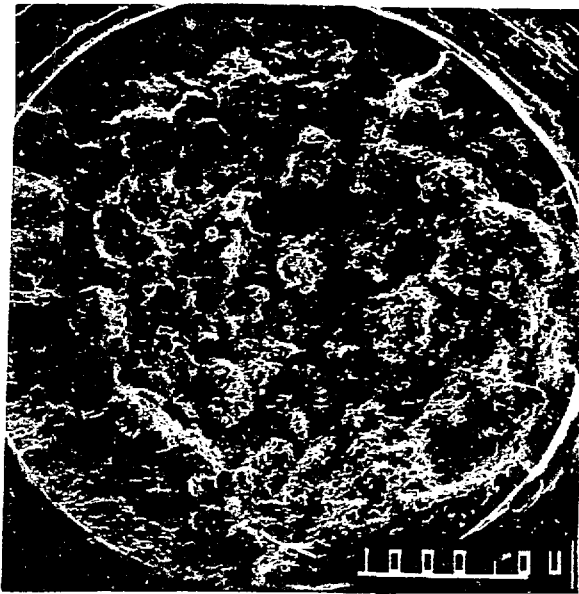


Figure 4 SEM photo of specimen #8 fracture face.

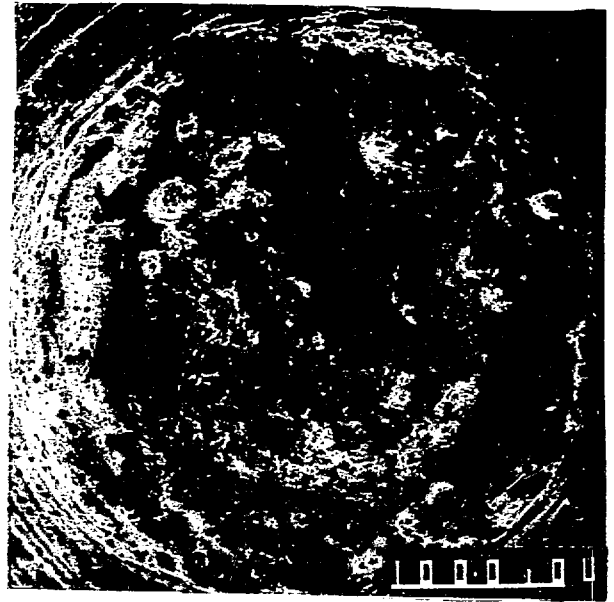


Figure 6 Low magnification SEM photo of specimen #55 (ductile)



Figure 5 Higher magnification fractograph of specimen #8 transgranular area.



Figure 7 Transgranular & ductile area of specimen #56.

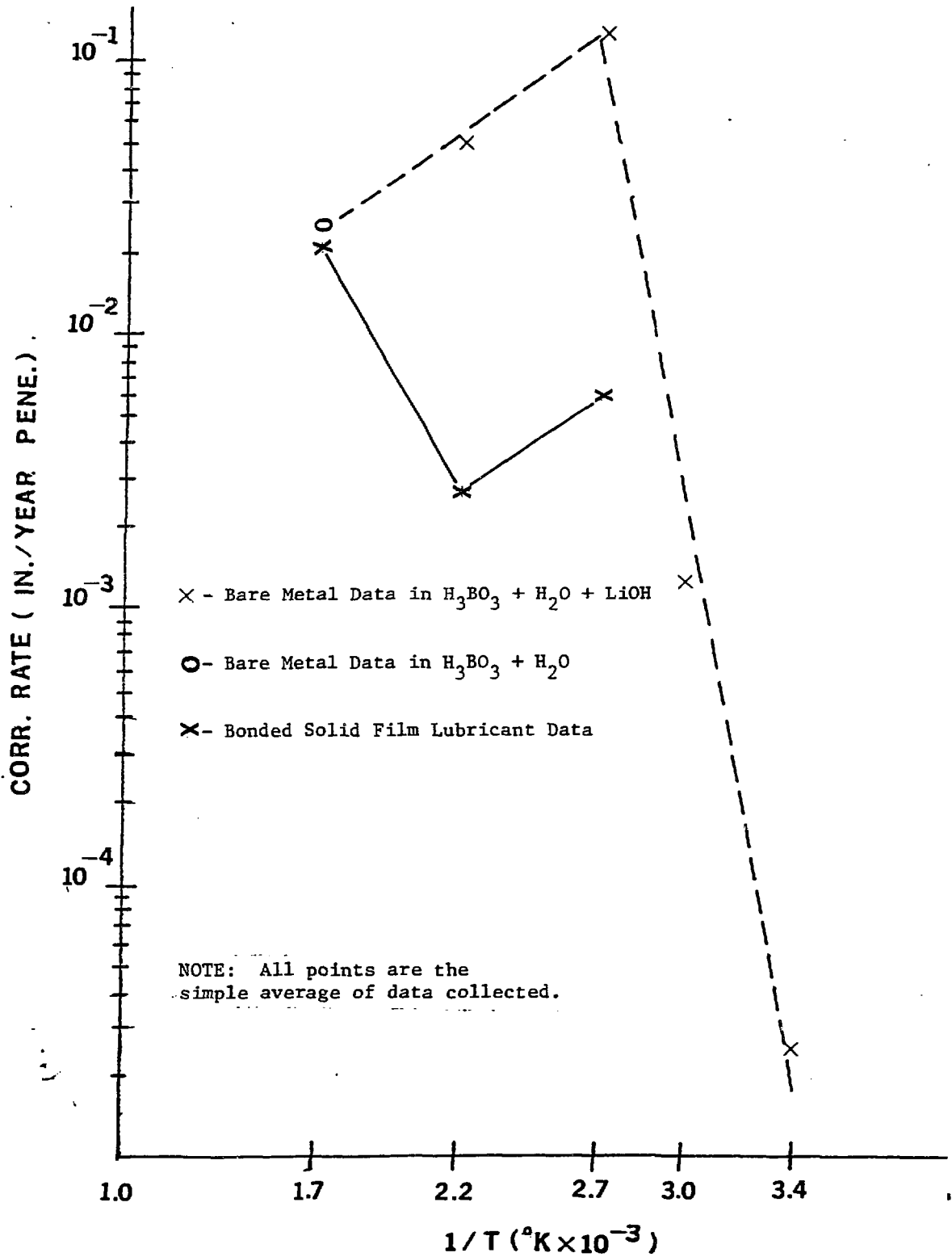


Figure 8 Graphical representation of weight loss experiments.