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Examination of Failed Studs From No. 2 Steam Generator at the Maine Yankee Nuclear Power Station

Prepared by C. Czajkowski

Brookhaven National Laboratory

**Prepared for
U.S. Nuclear Regulatory
Commission**

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ABSTRACT

Three studs removed from service on the primary manway cover from steam generator #2 of the Maine Yankee station were sent to Brookhaven National Laboratory (BNL) for examination. The examination consisted of visual/dye penetrant examination, optical metallography and Scanning Electron Microscopy/ Energy Dispersive Spectroscopy (SEM/EDS) evaluation. One bolt was "through cracked" and its fracture face was generally transgranular in nature with numerous secondary intergranular cracks. The report concludes that the environmentally assisted cracking of the stud was due to the interaction of the various lubricants used with steam leaks associated with this manway cover.

1.0 INTRODUCTION

During March 1982, at a maintenance outage of the Maine Yankee Power Plant, 5 of 20 primary manway studs failed during removal of the (cold leg) primary manway cover from steam generator #2.

Prior to this outage, the manway cover had been leaking, with retorquing of the studs and injection of sealant into the stud used by the utility as methods of controlling the leakage.

Since failure of this cover would result in a breach of the primary pressure boundary, the Materials Engineering Branch of the United States Nuclear Regulatory Commission (U.S.N.R.C.) requested that BNL perform an independent failure analysis on three of the bolts from the #2 steam generator manway cover.

The examination for this analysis included:

1) Visual inspection/photography/dye penetrant
2) Optical metallography
3) SEM/EDS analysis

2.0 VISUAL INSPECTION/PHOTOGRAPHY/DYE PENETRANT

The three studs received at BNL were approximately 10 1/2" long, and 1 1/2" in diameter and were surveyed by BNL Health Physics personnel as having a dose rate reading of approximately 2 mR/hr at contact.

The first stud examined (Figure 1) had apparent disruption to the thread area which had the appearance of damage caused by the use of a vise grip-type tool. There was also a white fibrous type coating on the opposing thread area of the stud (discussed in the SEM section).

The second stud examined (Figure 2) appeared to have residue from prior dye penetrant examinations and had a nut threaded on to one end of the stud. This nut was removed prior to dye penetrant examination by cutting (due to the extremely tight fit to the stud).

Figure 3 is a photograph of a "through cracked" stud. The crack is approximately 2 1/2" into the thread area "inserted end" of the stud. The fracture face of the stud (Figure 4) was relatively flat and had a black oxide appearance.

Dye penetrant examination was performed on the first two studs (Figures 1 and 2) using Spotcheck Brand (all Formula B) penetrant type SKL-HF/SKL-S developer type SKD-NF and cleaner/remover type SKC-NF. There were no relevant indications seen on the two studs. It should be noted that there was a tight black adherent film on the thread area of all studs examined, possibly from prior lubricant application (see SEM section).

No attempt was made to dye penetrant examine the cracked stud in order to keep exterior contamination to a minimum prior to SEM/EDS evaluation.

3.0 OPTICAL METALLOGRAPHY

A longitudinal section was made of the small end of the cracked stud, perpendicular to the main fracture face. It was then mounted, etched and polished (Figure 5). It can be clearly seen in the photomicrograph that numerous secondary cracks are present, initiating at the main fracture (areas A-F). These cracks had some fern-like branching associated with them which is normally indicative of environmentally assisted cracking. The major secondary cracks appeared to be generally transgranular in nature. The microstructure of the stud material (Figure 6 & 7) was that of a quenched and tempered martensite with a fine grained microstructural appearance. This microstructure is typical of an ASTM A540-B24 steel (Table 1).

4.0 SEM/EDS

The fracture face and a limited amount of thread area of the "through cracked" bolt was examined by SEM/EDS examination prior to any deoxidizing treatments in hopes of determining if any corrodent species were present.

The "as received" surface of the fracture face had a very flat and heavily oxidized surface (Figure 8) with the oxide varying from a nodular type (Figure 9) to an acicular shape (Figure 10). Initial SEM observations were that the fracture face was predominantly transgranular with no evidence of any ductile tearing. After some sections were deoxidized by an Endox 215 solution, however, various areas of apparently intergranular secondary cracking were revealed (Figures 11-13). This type of secondary cracking would be a definite indication of an environmentally assisted corrosion phenomenon.

An EDS scan of the base metal was performed (Figures 14 and 15) and showed characteristic peaks of Fe, Cr, Mn and Ni. These peaks would be typical for this alloy.

Various particles and areas of apparent material smearing were scanned for constituents using EDS. The first particle scanned from stud No. 2C6 was fibrous in appearance (Figures 16 & 17) and had quite high peaks of silicon and nickel in relation to the Fe alloy background. The scan also showed trace amounts of S, Ca and Cr. The high silicon content is probably related to the use of the silicon base Furmanite sealer used by the utility to seal the leaking stud holes. The high nickel content is attributed to the utility's use of Fel-Pro-5000, a nickel-base lubricant. (For typical certifications of materials used at Maine Yankee (see Tables 2-5.)) A second fibrous particle scanned (Figures 18 & 19), however, showed only peaks of Fe, Cr and Ni.

Another area scanned (Figures 20 & 21) appeared to be a pit and had a lead (Pb) peak in addition to Si, Cr, Fe and Ni. The appearance of lead may be attributed to the Fel-Pro which allows up to 25 ppm total lead in its specification.

EDS analysis of a smeared area (Figures 22 & 23) showed peaks of Si, S, Ca and Fe only.

A second smeared area (Figure 24) had such a high peak in sulfur (Figure 26) that a wavelength dispersive spectrographic scan was accomplished to determine if molybdenum was present with the sulfur. It can be clearly seen (Figures 27 & 28) that this particular area is quite concentrated in Mo which is indicative that a molybdenum disulfide type lubricant may have been used sometime in the service history of the stud.

5.0 DISCUSSION

Since the cracking of the steam generator manway studs was associatd with leaking gaskets, it is worthwhile to examine the observed effects of pressurized water reactor (PWR) primary coolant on high strength low alloy bolting materials. A review [1] was performed by BNL on incidents of boric acid wastage corrosion at seven nuclear units. All incidents involved a primary coolant leaking mechanism and in no instance was cracking observed. All degradation occurred by a general wastage/corrosion mechanism.

This being the case; it is therefore logical to assume that the addition of other environmental variables must be made in order to crack these materials in either a transgranular or intergranular manner. The most obvious source of these contaminants is the lubricant applied to these materials in service.

Work done at BNL [2,3] on turbine disc steels has shown that molybdenum disulfide lubricants can have a marked effect on lowering the ultimate tensile strength of high strength low alloy steels when exposed to a steam environment on notched tensile specimens.

A metallurgical failure analysis [4] performed on steam generator manway studs at the Oconee Unit 3 power station ascribed the intergranular attack on the bolts to the use of molybdenum disulfide lubricants.

Kay [5] has cited that MoS_2 can oxidize in the presence of air and moisture to produce molybdenum dioxide and sulfuric acid which would be quite detrimental to a martensitic steel.

Finally, a report issued by the Swansea Tribology Centre [6] on molybdenum disulfide lubrication has listed these precautions on the use of the lubricant:

(quoted in part)

"1) Always remember that where conditions exist which will tend to cause corrosion, the presence of molybdenum disulphide may increase the extent of the corrosion.

2) Wherever possible eliminate the presence of corrosive materials such as acids, brines, or water and use corrosion - resistant substrates."

It is, therefore, logical to assume that some interaction between the leaking steam and the sulfur containing lubricants on the bolts may have led to the premature failure of the bolt by a stress corrosion crack mechanism.

6.0 CONCLUSION

1) The cracked bolt failed in a generally transgranular mode with numerous secondary intergranular cracks.

2) This cracking is considered environmentally assisted in nature and was typical of a stress corrosion cracking phenomenon.

3) Since prior investigations have shown that high strength low alloy steels in primary coolant will have a wastage-type corrosion only, this cracking is considered to be the result of the interaction of primary water/steam with the use of sulfur containing lubricants.

7.0 ACKNOWLEDGEMENT

The author wishes to thank R. Sabatini for the SEM/EDS work; L. Gerlach and D. Becker for the metallography; O. Betancourt for her typing and Dr. J. R. Weeks for his continuing support.

8.0 REFERENCES

1. Czajkowski, C., BNL-NUREG-31098, March, 1982.
2. Czajkowski, C., BNL-NUREG-28724, October, 1980.
3. Czajkowski, C., BNL-NUREG-29964, March, 1981.
4. Burck, L. H., Foley, W. J., Report No. IE-123, April, 1981.
5. Kay, E., Wear, 12 (1968), 165-171.
6. Lansdown, A. R., Report No. 79/419 ESA (ESTEC) Contract No. 2261/74.

TABLE 1

TYPICAL CHEMICAL AND MECHANICAL PROPERTIES OF

ASTM A540-B24 STEEL

Chemical Requirements		Product Variations %, over or under
Carbon	0.37-0.44	0.02
Manganese	0.70-0.90	0.04
Phosphorous, max.	0.025	0.005
Sulfur, max.	0.025	0.005
Silicon	0.15-0.35	0.02
Chromium	0.70-0.95	0.05
Nickel	1.65-2.00	0.05
Molybdenum	0.30-0.40	0.02
Vanadium	-	-

Mechanical Requirements

Grade	Class	Diameter
B24	3	to 3, incl.

Tensile Strength	Yield Strength	Elongation	% Red. of Area,	Surface Hardness, Brinell	
min.	0.2 offset,min.	% in 2 in.	min.	min.	max.
145 ksi	130 ksi	12	40	293	363

FURMANITE
INCORPORATED

529 VIKING DRIVE • VIRGINIA BEACH, VIRGINIA 23452 • PHONE (804) 486-8182 • TELEX 823-675

TABLE 2

TYPICAL CERTIFICATION FOR SEALANT USED (LOT 505)

MATERIAL CERTIFICATION
REVISION 1

August 14, 1981

Material: Furmanite Nuclear Grade Compound F-500, Lot No. 505

Expiration date for Compound: July 31, 1982

This is to certify that the analysis results of a typical sample of the above compound, analyzed by an independent test laboratory, is as follows:

	TOTAL	LEACHABLE
Chlorine	38	26
Flourine	11	<1
Sulfur (As S)	14	≤2
Antimony	*	≤0.03
Arsenic	*	≤0.03
Cadmium	*	≤0.03
Lead	*	≤0.3
Tin	*	≤3
Zinc	*	≤0.05
Mercury	*	0.002

Note: Results in microgram/gram
* Not Measured

D. F. Limroth
Engineering Manager

Ref: CT&E - 8/6/81

DFL/tfm

MAINE YANKEE ATOMIC POWER CO.
QUALITY CONTROL INSPECTED
SAT. ✓ UNSAT. .....
DATE 10/24/81 INSPECTOR R. Lantz

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TABLE 3

TYPICAL CERTIFICATION FOR SEALANT USED (LOT 701)

MATERIAL CERTIFICATION

October 19, 1981

Material: Furmanite Nuclear Grade Compound F700, Lot No. 701

Expiration date for Compound: October 16, 1982

This is to certify that the analysis results of a typical sample of the above compound, analyzed by an independent test laboratory, is as follows:

	TOTAL	LEACHABLE
Chlorine	179.5	32.7
Fluorine	19.0	0.88
Sulfur	62	18
Antimony	*	0.11
Arsenic	*	< 0.02
Cadmium	*	< 0.005
Lead	*	< 0.05
Mercury	*	< 0.002
Tin	*	< 0.05
Zinc	*	< 0.05

Note: Results in microgram/gram
* Not Measured

D. F. Limroth
Engineering Manager

Ref: Jennings Laboratories, Inc.

DFL/tfm

MAINE YANKEE ATOMIC POWER CO.
QUALITY CONTROL INSPECTED
SAT. ✓ UNSAT. .....
DATE 10/24/81 INSPECTOR R. Linton

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TABLE 4

TYPICAL CERTIFICATION FOR SEALANT USED (LOT 702)

MATERIAL CERTIFICATION

December 4, 1981

Material: Furmanite Nuclear F-700-N, Lot #702

Expiration date for compound: December 4, 1982

This is to certify that the analysis results of a typical sample of the above compound, analyzed by an independent test laboratory is as follows:

	Total	Leachable
Chlorine	246.2	29.6
Fluorine	24.0	.65
Sulfur	15	84.0
Antimony	*	0.18
Arsenic	*	<0.02
Cadmium	*	<0.005
Lead	*	<0.05
Mercury	*	<0.002
Tin	*	<0.05
Zinc	*	<0.05

Note: Results in microgram/gram
* Not Measured

D. F. Limroth
Engineering Manager

REF: Jennings Laboratories, Inc.
12/04/81

DFL/tfm

MAINE YANKEE ATOMIC POWER CO.
QUALITY CONTROL INSPECTED
SAT. ✓ UNSAT. .....
DATE 12-22-81 INSPECTOR .....

TABLE 5

TYPICAL CERTIFICATION FOR LUBRICANT USED (BATCH 55)

FEL-PRO N-5000
NUCLEAR GRADE
ANTISEIZE LUBRICANT

BATCH NO. 55

DATE OF MFR. 6/10/81

CERTIFICATE OF COMPLIANCE

It is hereby certified that the above batch of Fel-Pro N-5000 meets the following purity standards:

	MAXIMUM ALLOWABLE PER N-5000 SPECIFICATION	TEST RESULTS SAMPLE #1	SAMPLE #2	SAMPLE #3
Total Fluorine	200 ppm	25 ppm	24 ppm	15 ppm
Total Chlorine	50 ppm	<20 ppm	<40 ppm	<20 ppm
Total Sulfur	100 ppm	<25 ppm	<25 ppm	<25 ppm
Total Lead	25 ppm	8.5 ppm	7.5 ppm	8.8 ppm
Total Tin	25 ppm	11.6 ppm	—	—
Total Zinc	25 ppm	6.4 ppm	—	—
Total Cadmium	2 ppm	0.8 ppm	—	—
Total Mercury	2 ppm	0.17 ppm	—	—
Total Copper	50 ppm	2.6 ppm	—	—

NOTE: Original test report on this batch of N-5000 is on file at FEL-PRO INCORPORATED. Copies are available upon request. Test results are further shown on each can filled from this batch of materials.

BY: FEL-PRO INCORPORATED
7450 N. McCORMICK BLVD.
Skokie, Ill. 60076

W L Schaefer
Officer of Company

Prod. Mgr., Chem. Prod. Div.
Title

SUSCRIBED AND SWORN TO BEFORE ME THIS DATE:

07 Aug 1981

Notary Public

NOTARY PUBLIC
COOK COUNTY, ILL.

MAINE YANKEE ATOMIC POWER CO.
QUALITY CONTROL INSPECTED
SAT. ✓ UNSAT. ____
DATE 8-7-81
INSPECTOR

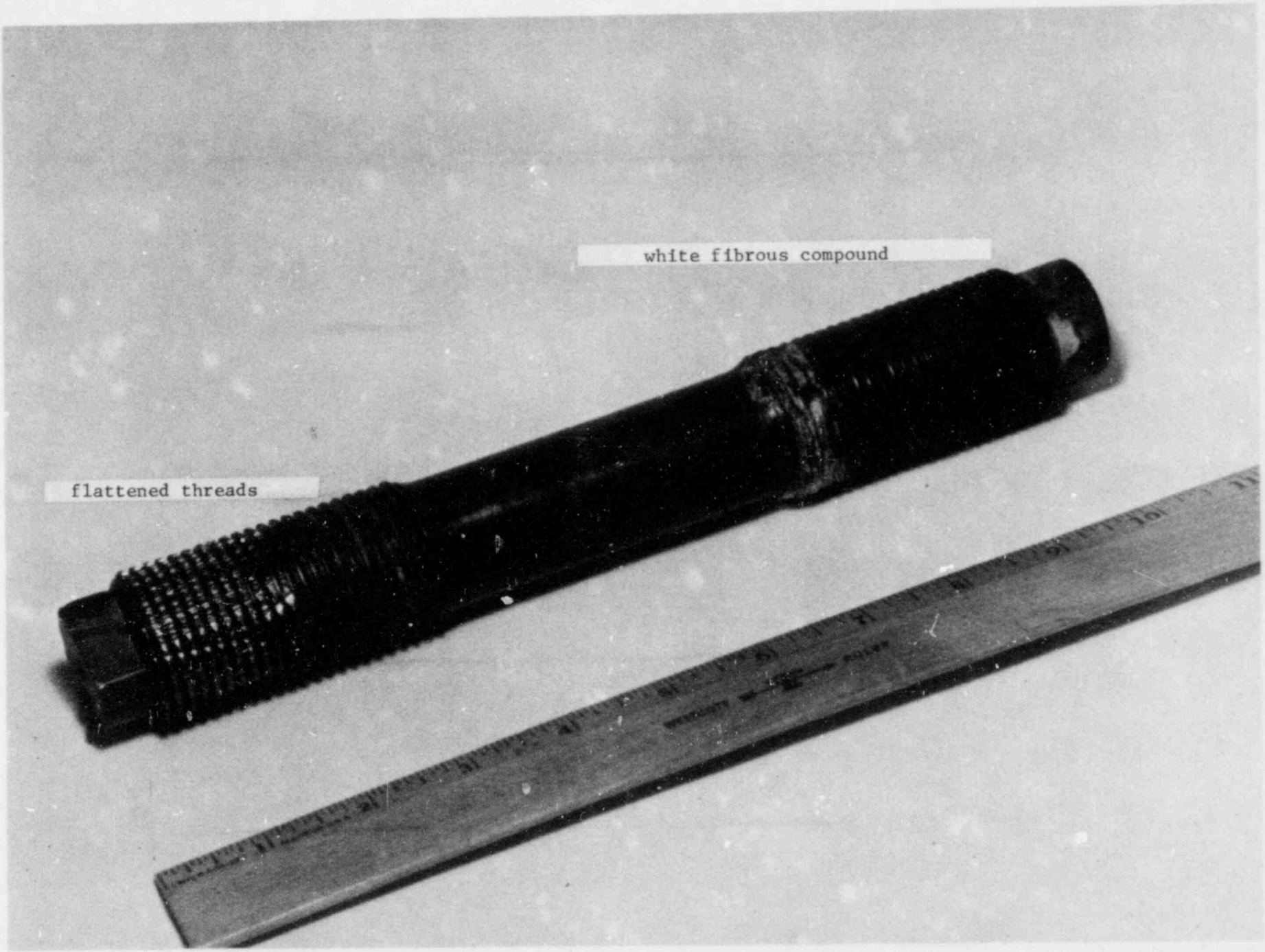


Figure 1. Photograph of "as received" stud from Maine Yankee. (Bolt had the following numbers stamped on the ends - 2C6, #46500, 6410).

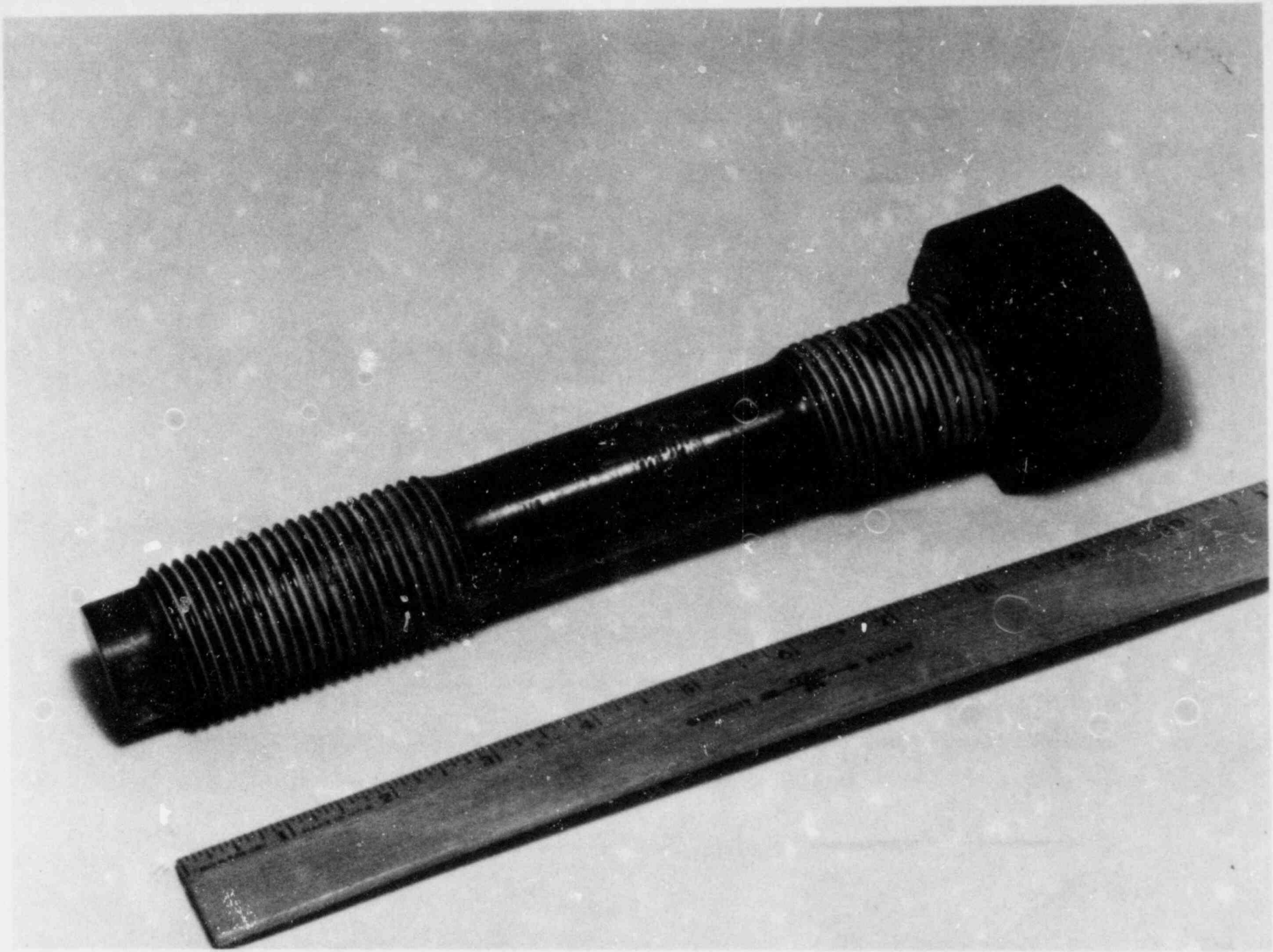


Figure 2. Photograph of second stud received from Maine Yankee. Photo depicts remnants of Dye Penetrant testing. (Bolt had the following numbers stamped on the ends (2C20, #64034)).

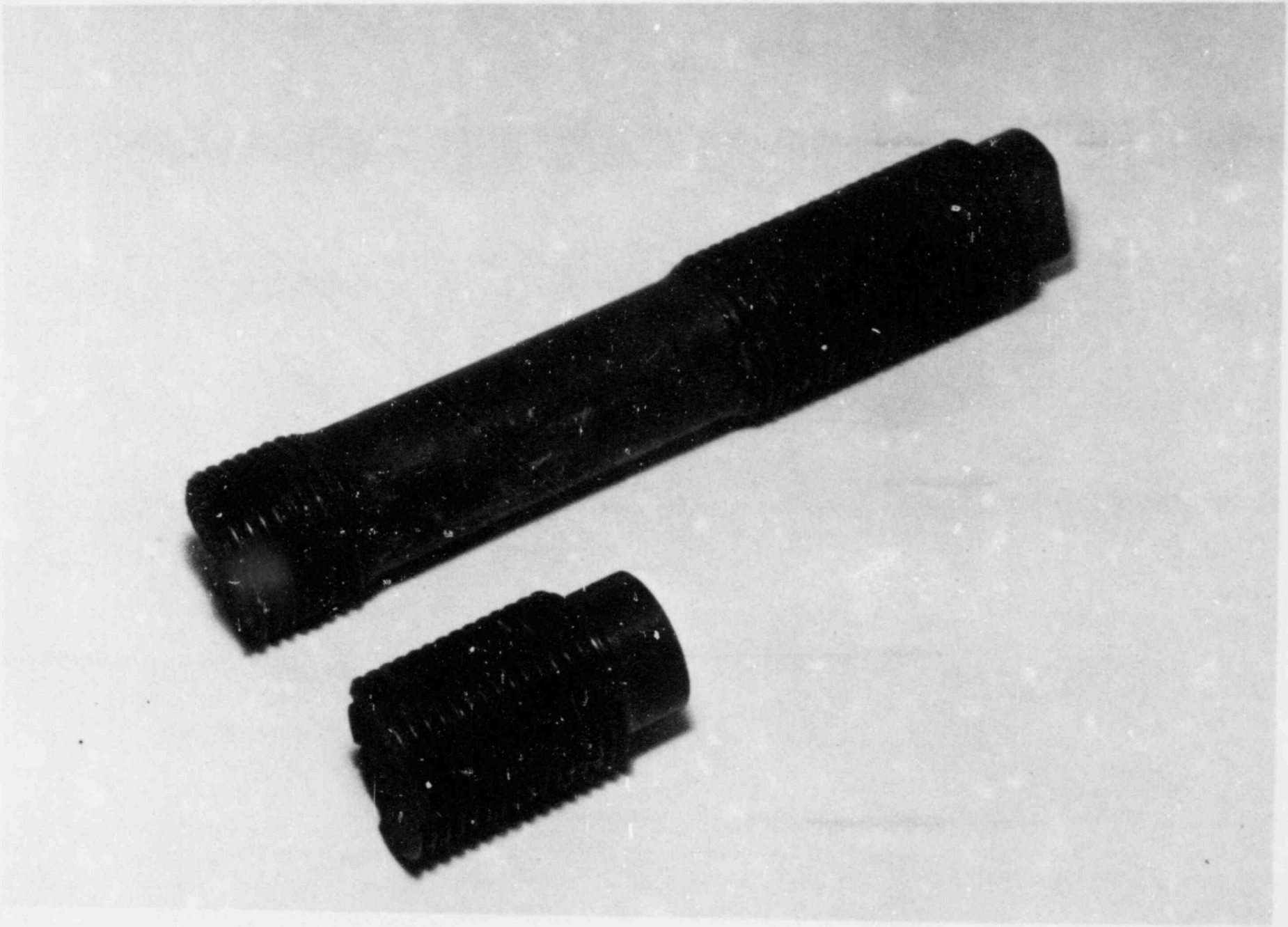


Figure 3. Photograph of "cracked" stud from Maine Yankee (Bolt had the following numbers stamped on the ends-2C8, 6418, #64034).

Figure 4. Frontal photograph of the fracture faces of the cracked bolt.

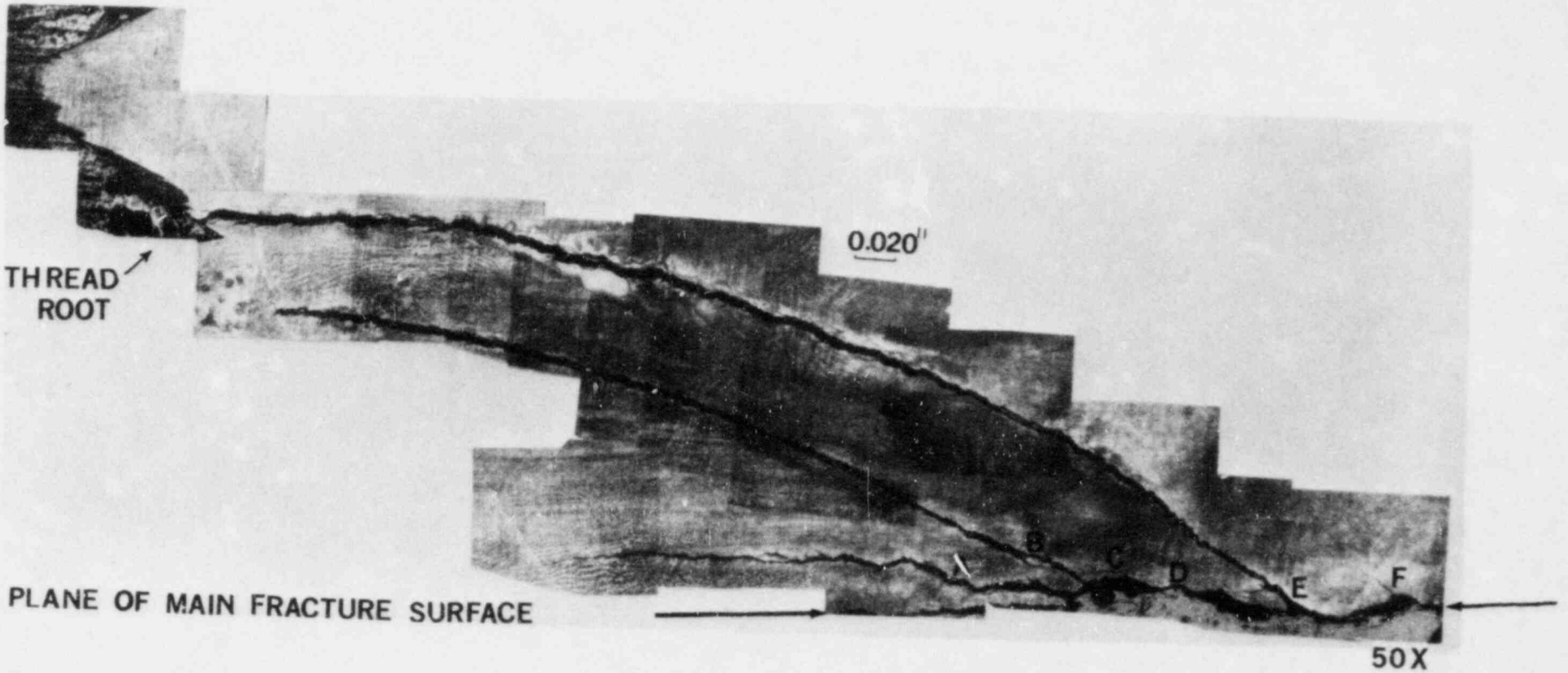
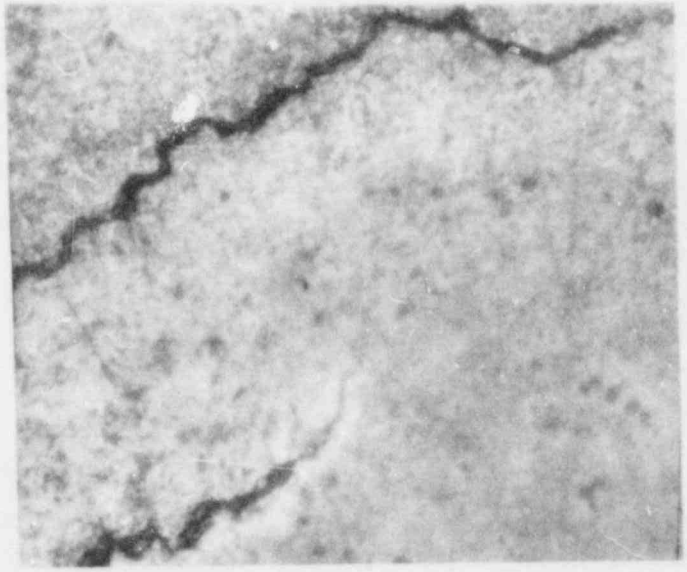
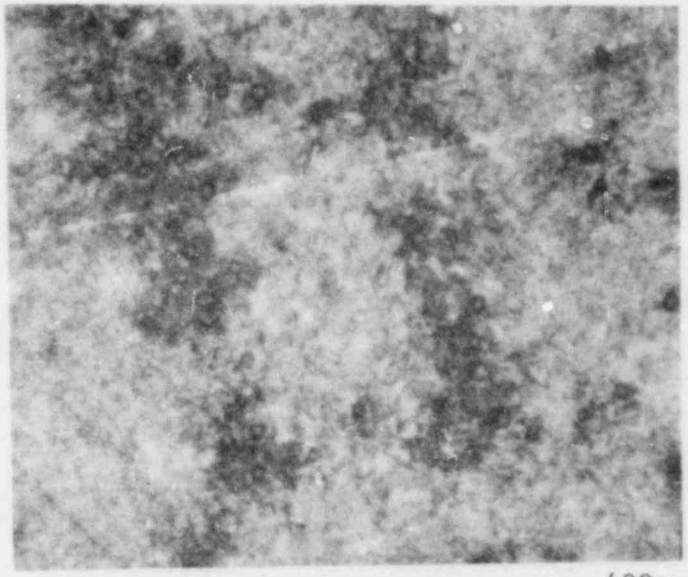


Figure 5. Optical photomicrograph of bolt section depicting extent of secondary cracking (areas A-F). Note fern like branching on larger secondary cracks.



500x

Figure 6. Optical photomicrograph of a secondary crack after etching.



400x

Figure 7. Optical photomicrograph of bolt's structure after etching.

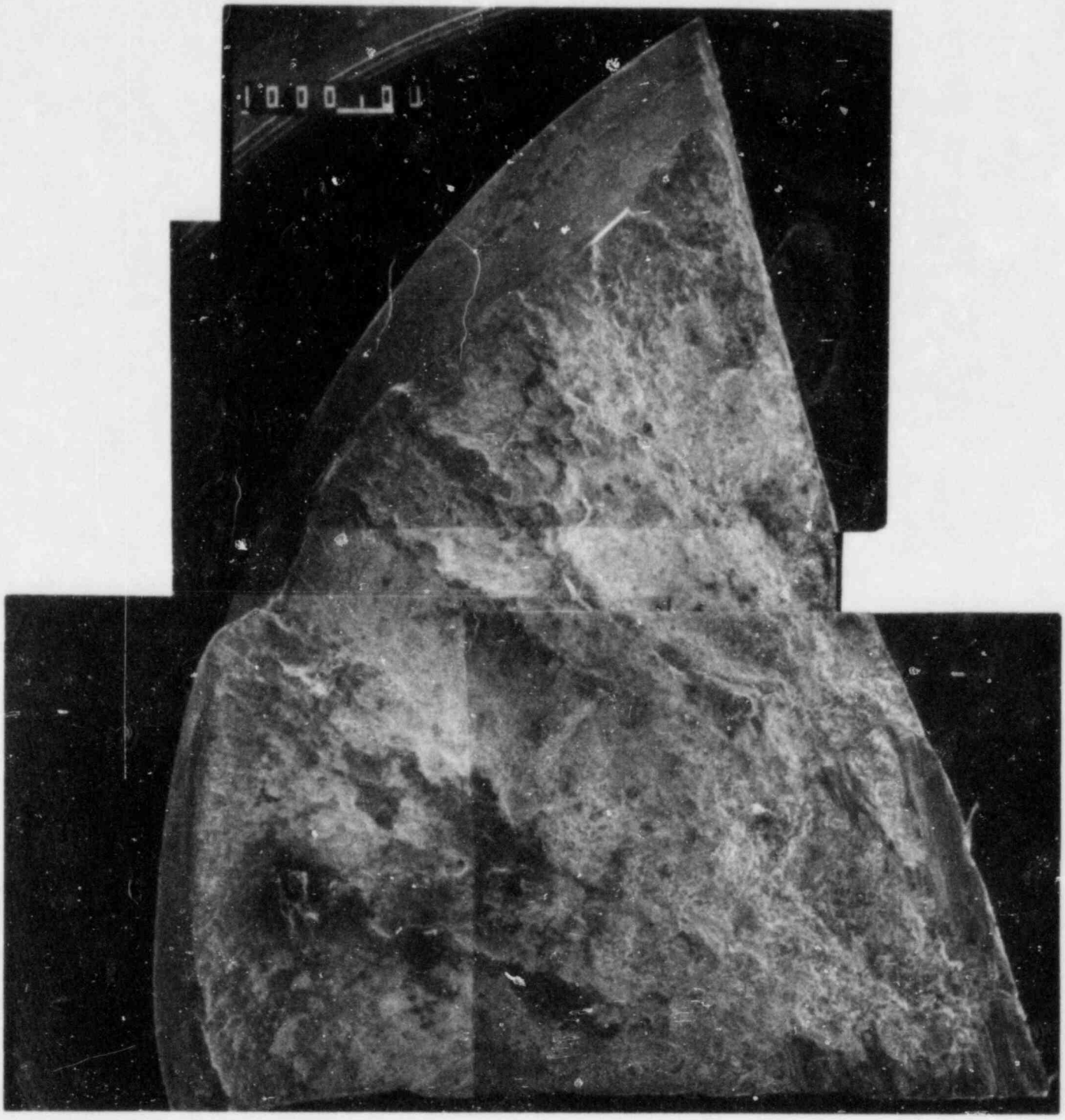
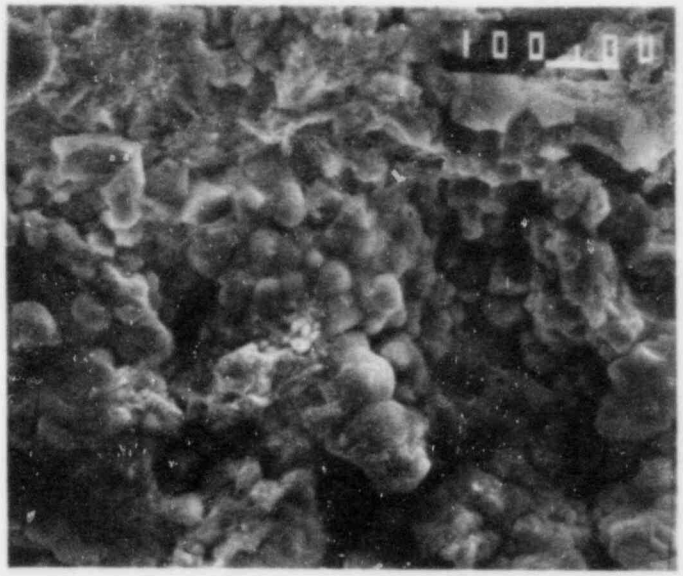
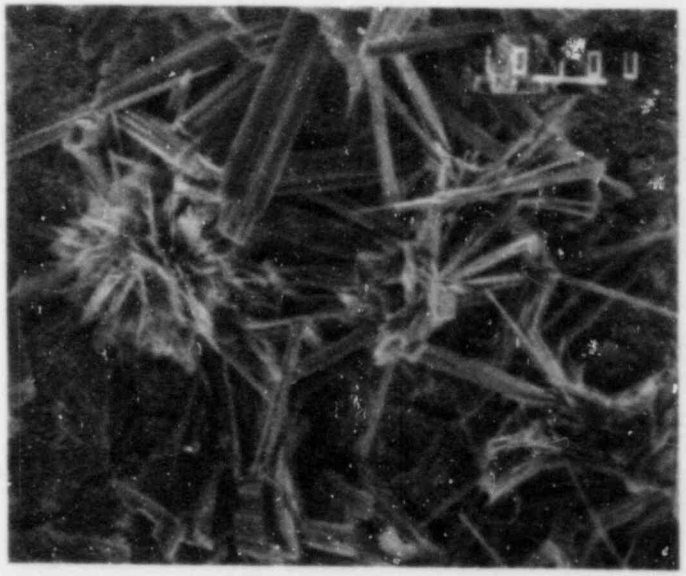


Figure 8. SEM photograph of a quadrant of the "cracked bolt " fracture face.



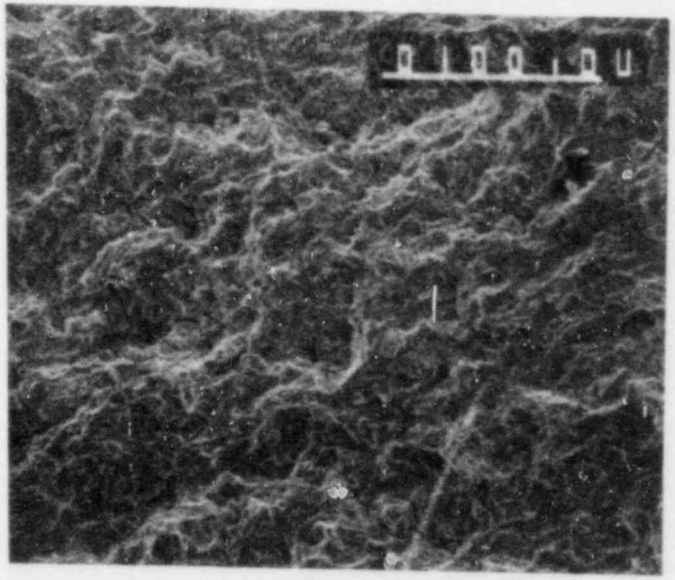
100x

Figure 9. SEM photograph of "nodular" oxide.



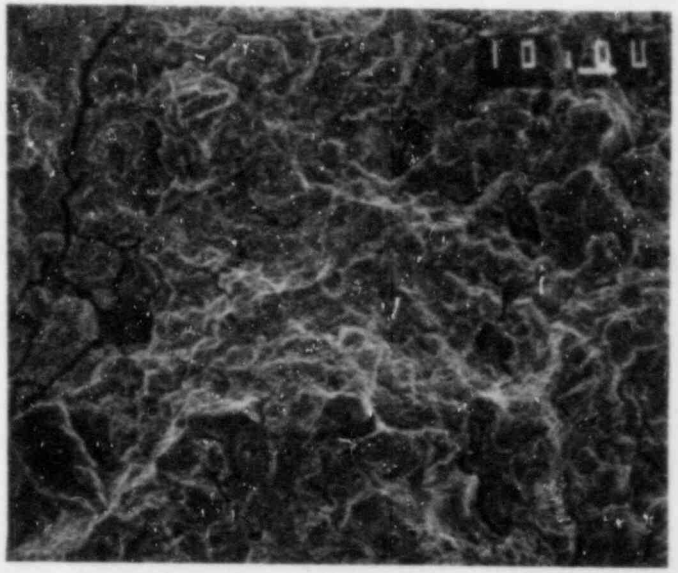
1000x

Figure 10. SEM photograph of "acicular" oxide.



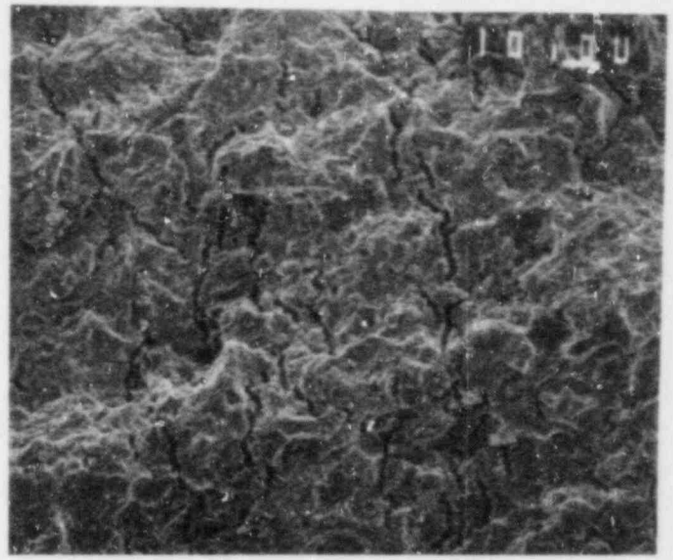
100x

Figure 11. SEM photo of fracture face prior to deoxidation treatments.



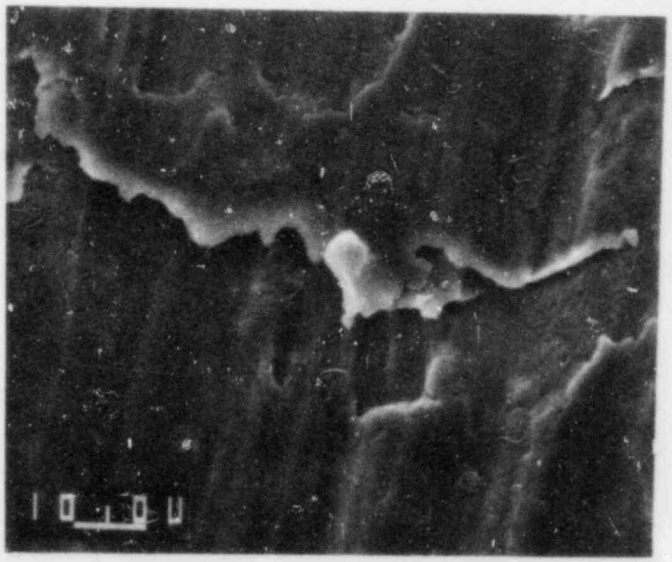
500x

Figure 12. SEM photo of fracture face depicting intergranularity of secondary cracks.



500x

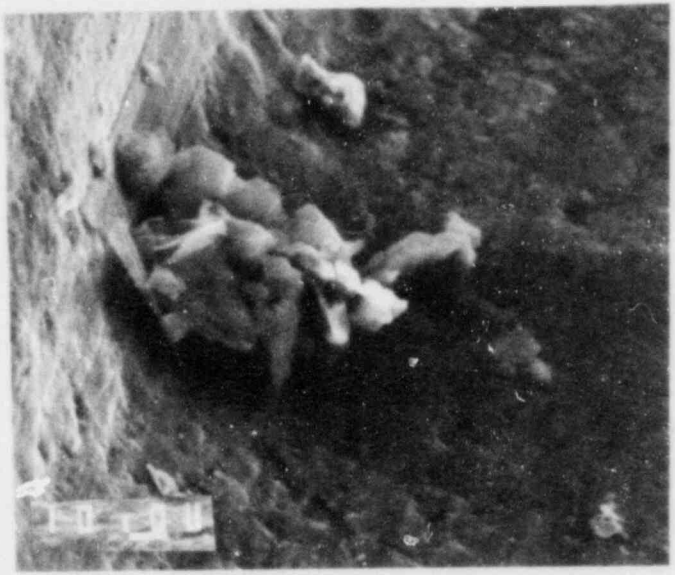
Figure 13. SEM photo of second area on the fracture face depicting secondary intergranular cracks.



1000x

Figure 14. SEM photo of base material area scanned.

Figure 15. EDS scan of base material for constituents.



500x

Figure 16. SEM photo of fibrous particulate.

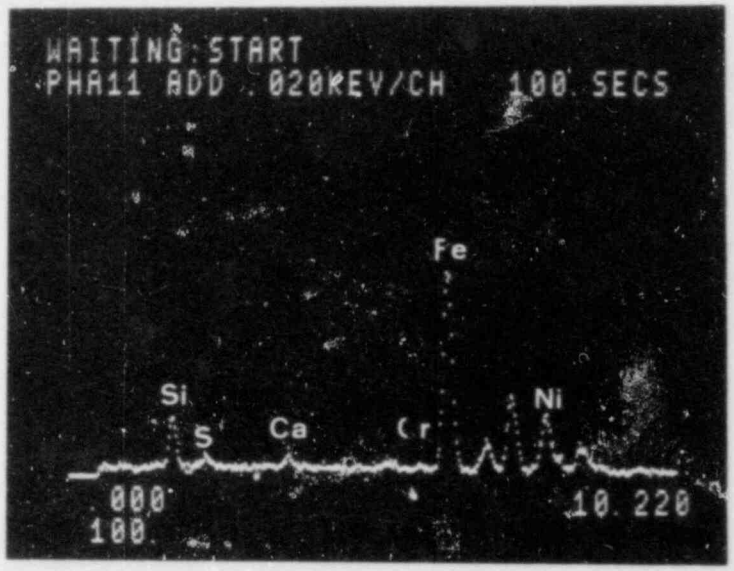
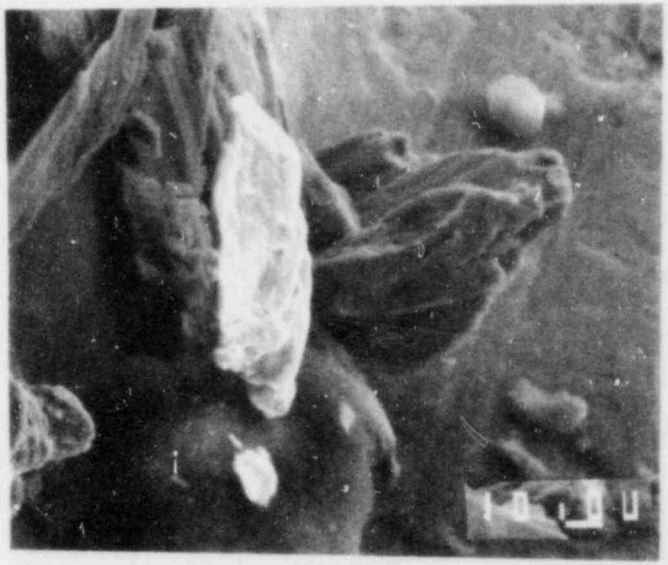


Figure 17. EDS scan of fibrous particle.



500x

Figure 18. SEM photo of second fibrous particle.

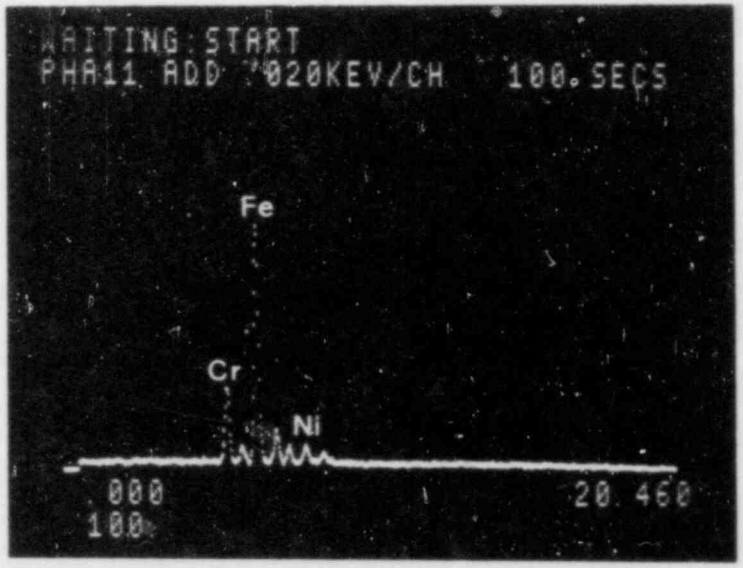
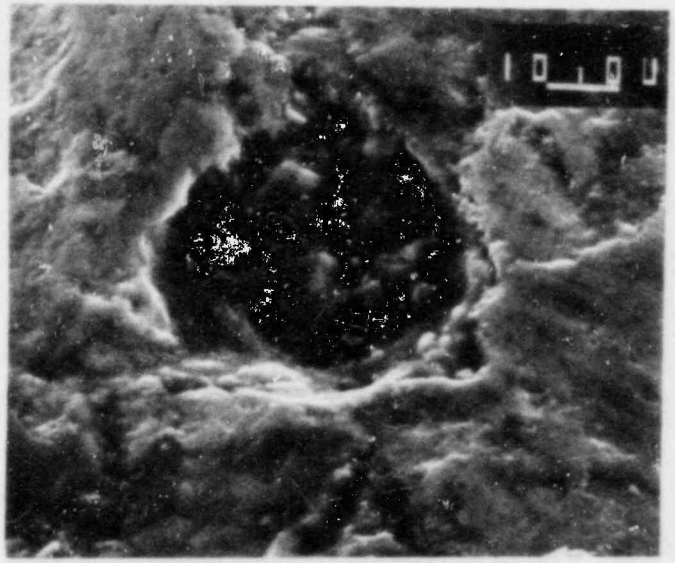


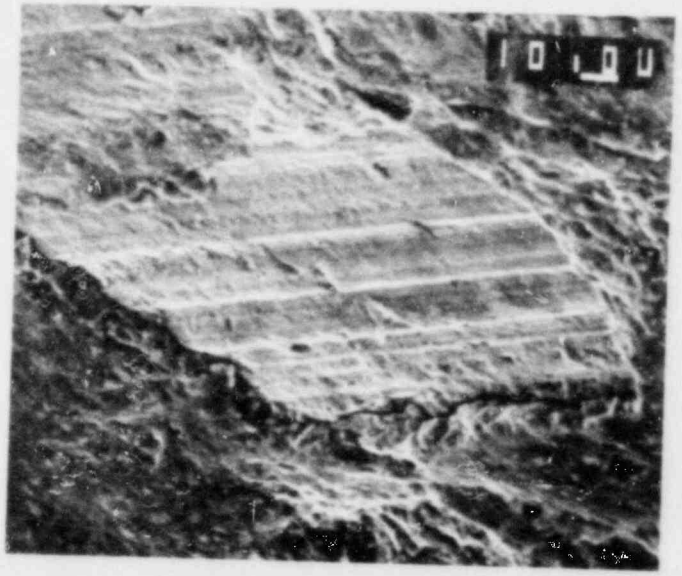
Figure 19. Constituents of second fibrous particle scanned by EDS.



1000x

Figure 20. SEM photo of apparent pit on thread of the cracked stud.

Figure 21. EDS scan of pit for chemical constituents.



500x

Figure 22. SEM photo of area of smeared material on thread area of the cracked bolt.

Figure 23. EDS scan of smeared material area.

1000x

Figure 24. SEM photograph of second "smeared" area on bolt.

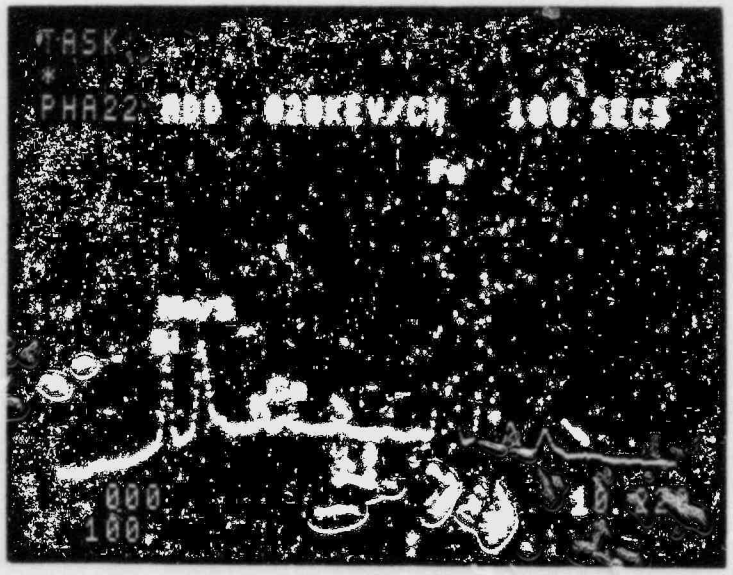


Figure 25. EDS scan of "smeared" area in Figure 25.

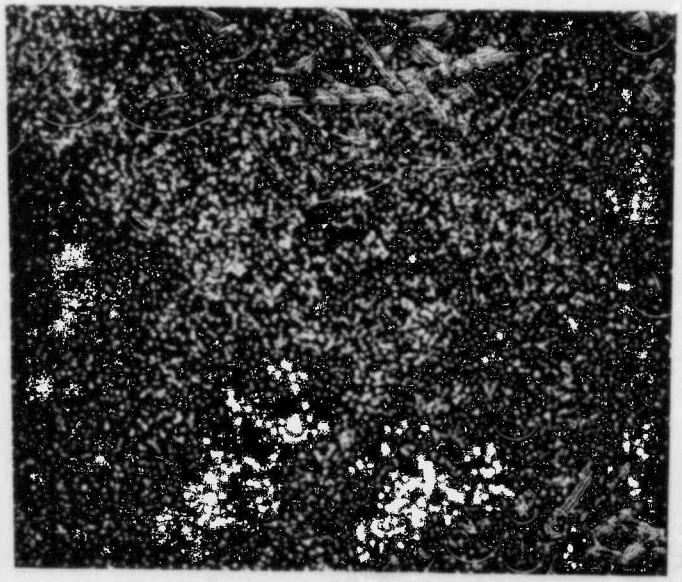


Figure 26. WDS scan of "smeared" area for molybdenum.

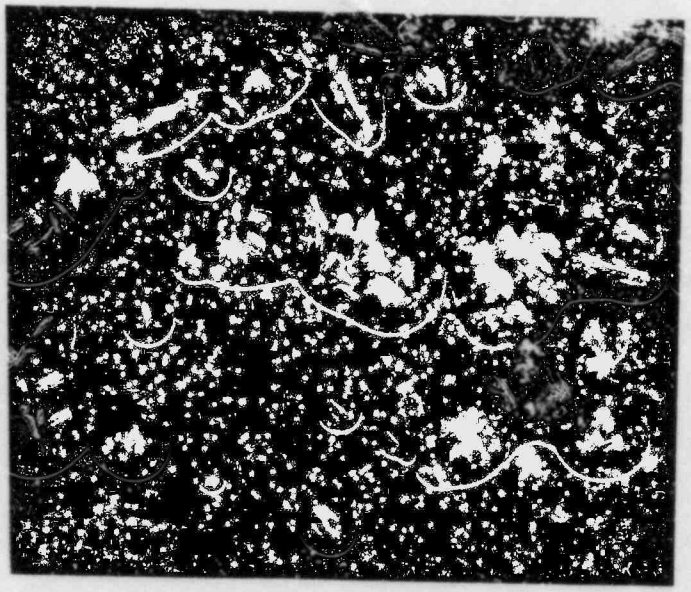


Figure 27. WDS scan of smeared area for sulfur.

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16. ABSTRACT (200 words or less)

Three studs removed from service on the primary manway cover from steam generator #2 of the Maine Yankee station were sent to Brookhaven National Laboratory (BNL) for examination. The examination consisted of visual/dye penetrant examination, optical metallography and Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) evaluation. One bolt was "through cracked" and its fracture face was generally transgranular in nature with numerous secondary intergranular cracks. The report concludes that the environmentally assisted cracking of the stud was due to the interaction of the various lubricants used with steam leaks associated with this manway cover.

17. KEY WORDS AND DOCUMENT ANALYSIS

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lubricant corrosion
threaded fastener degradation
transgranular cracking

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