ENCLOSURE (1)

UNITED STATES
NUCLEAR REGULATORY COMMISSION
ADVISORY COMMITTEE ON REACTOR SAFEGUARDS
WASHINGTON, D. C. 20555

October 20, 1981

MEMORANDUM FOR: William J. Dircks, Executive Director for Operations

FROM: Raymond F. Fraley, Executive Director

SUBJECT: BOLT FAILURES IN NUCLEAR POWER PLANTS

In the last few years there has been a significant number of incidents of failed or severely degraded bolts in systems essential to cope with design basis accidents or in closures to the primary pressure boundary. Examples are: pressure vessel supports (Midland), steam generator supports (Haddam Neck), primary pump bolts (Calvert Cliffs and Fort Calhoun), steam generator primary manway closures (Oconee), and core internals (Oconee). These clearly represent a deterioration of essential lines of defense in protecting against accidents and thus are safety issues meriting consideration.

Some of these failures have occurred in steels believed to have been specified in conformance with good practice, with respect to composition and strength. Other failures may have been associated with an unconventional use of ultrahigh-strength material.

Of the various phenomena involved here, the one of most concern is the stress corrosion cracking of high-strength bolts since these can break without warning.

We believe it is essential that the Staff begin an active program to establish:

(1) Whether ultrahigh-strength bolts with high-pretension are necessary in the nuclear applications where they have been used.

(2) Which plants may be using such bolts and the conditions under which their use should be allowed.

(3) What conditions may have led to failure in bolts believed to have been specified to conventional good practice.

(4) What regulatory actions are needed to avoid challenges to primary system integrity from bolt failures.

DEC 0 2 1981

MEMORANDUM FOR: Raymond F. Fraley, Executive Director
Advisory Committee on Reactor Safeguards

FROM: William J. Dircks
Executive Director for Operations

SUBJECT: BOLT FAILURES IN NUCLEAR POWER PLANTS

In response to your memorandum of October 20, 1981, the NRC staff is currently addressing many of the concerns about bolting and bolting materials that you described.

Work sponsored by the Materials Engineering Branch and performed by the Lawrence Livermore National Laboratory (LLNL) has led to the preparation of a report, "Lower Bound K_{ISCC} For Bolting Materials - A Literature Survey," which will be published this month. This report will document work that has been performed on the stress corrosion cracking susceptibility as a function of the magnitude of preload for low alloy bolting steels and maraging steels in various environments (air, water, sea water, sulfur gases). This report will be utilized jointly by the staff and our contractor, Brookhaven National Laboratory (BNL), to prepare an NRC position on the actions that should be taken to prevent stress corrosion cracking in safety-related bolting and bolting materials. Since the LLNL work did not include age-hardened alloys (such as I conel X750 or A286), it will be necessary for BNL to compile similar data for these alloys prior to the preparation of the NRC position.

We anticipate that this position will provide a basis for improved control of bolting practice which will reduce the incidence of bolting failures in both operating plants and plants under construction. As part of the development of the staff position, we will prepare a family of curves of strength (or hardness) versus preload limit for various environments which will specify pretension limits for the use of the so-called ultra high strength bolting materials. These limit curves would also apply to the lower strength bolting materials. The position may also include new requirements related to design, material selection, materials testing,

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and improved inspection and quality control. Implementation of this position will probably require some changes in inspection as cur ently performed (e.g., addition of hardness testing) both in situ and a receipt at a plant site. Some voluntary activity in the area of hardness testing is currently underway by tilities. The results thus far show a wide variation of hardness values from the specified values. A survey at Midland performed on various bolting applications indicates that many bolts (207 of 334) ordered to a specific hardness range are supplied grossly out of that hardness range. T s results in stress corrosion failure when a pretension based on the a umed hardness is applied.

The Chemical Engineering Branch, after its initial evaluation of the borated water corrosion wastage of primary pump bolts at Ft. Calhoun and other plants, contracted with BNL to perform a review of all such incidents. Based on the initial BNL report, which indicated that little detailed information is available about this type of corrosion, BNL was directed to expand their investigation into field experience and laboratory work and provide recommendations regarding the need for plant surveillance requirements. An initial report was provided on November 10, 1981, and the final BNL recommendations are scheduled for January 15, 1982.

The Division of Safety Technology (DST) is placing this subject on their list of Generic Issues for prioritization. DST plans to complete the prioritization by March 1982. If warrented by this prioritization, DST will develop and carry out a plan for resolution.

Implementation of the resolution would require participation of other groups within the NRR staff. The Mechanical Engineering Branch would identify systems containing those bolts or bolting materials used which are "safety related." A survey of the utilities would be necessary to identify such bolts or bolting material applications. Such a survey would identify those plants using combinations of actual bolting material strength (as determined by hardness) and pretension outside of the limit curves which would result in bolts highly susceptible to failure.

Implementation of some actions by licensees and applicants may be required somewhat earlier if results of this study indicate a need for urgent action.

In addition, the Operating Reactors Assessment Branch is preparing a report that describes the recent adverse bolting experience, describes the safety implications, and outlines current and future recommended regulatory actions. The objective of this report is to provide NRC management with an updated summary of the problem and the status of its resolution.

OFFICE							
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It is anticipated that the combination of the survey, identification of safety-related bolting, and the application of the NRC bolting position would significantly reduce challenges to primary system integrity from failures of bolting or bolting materials.

We will keep the ACRS informed of developments in this area.

(Signed) William J. Dircks

William J. Dircks
Executive Director for Operations

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UNITED STATES
NUCLEAR REGULATORY COMMISSION
WASHINGTON, D. C. 20555

APR 0 6 1982

MEMORANDUM FOR: Darrell G. Eisenhut, Director
Division of Licensing

FROM: Stephen H. Hanauer, Director
Division of Safety Technology

SUBJECT: CORROSION OF RCP STUDS

REFERENCE: Memo, D. G. Eisenhut to S. H. Hanauer and R. H. Vollmer, "Corrosion of RCP Studs," dated March 24, 1982.

In the referenced memorandum you recommended that prompt attention be given to fully assessing the safety implication of the corrosion of studs in PWR coolant systems. We agree that this issue has high priority because it contributes significantly to risk and would be relatively inexpensive to correct.

We estimate that the frequency of small-break LOCAs that could result from corrosion of stud bolts would be about 6×10^{-3} per PWR-year, based on the operating experience to date. Based on the WASH-1400 distribution of release categories resulting from small-break LOCAs, the estimated risk is 1.5×10^{4} Ci/PWR-year. If regular visual inspection of studs is required we believe that the risk can be reduced by at least a factor of ten. Over the life of a plant, the additional cost of visual inspection is estimated to be approximately $110,000. Thus the priority score for this issue is 1×10^{5} Ci/RY/10^{6} dollars, which is a high score relative to other issues that have been prioritized. The details of the evaluation are enclosed.

ORAB is coordinating the work of several branches and is preparing a general report on all mechanisms of bolting degradation. We recommend that this report be completed quickly. The report contains a number of recommendations including the recommendation for visual inspection. While the recommendations in the report would provide a comprehensive resolution to the issue of bolt failures, we believe that the recommendation to visually inspect bolts in reactor coolant systems has the highest priority and should be implemented without waiting for the comprehensive resolution.

Stephen H. Hanauer, Director
Division of Safety Technology

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Enclosure:
Priority Score for RCP Stud Corrosion

cc: M. Ernst
W. Minners
T. Ippolito
P. Wagner
H. Vander Molen
V. Benaroya

ENCLOSURE

PRIORITY SCORE FOR RCP STUD CORROSION

Frequency

Twenty-three incidents of boric acid corrosion of bolts or studs have been reported in (currently) about 350 PWR-years of experience. Thus, the frequency of corrosion initiation is 23/350, or 6.6×10^{-2} events per reactor-year.

In the most recent event at Ft. Calhoun, studs degraded 1/4" in one 18-month fuel cycle. Thus, if we assume a constant linear rate of degradation, it would take about seven years for a 3 1/2" stud to degrade to 1 1/8" diameter, as had happened earlier at Ft. Calhoun.

One of the degraded studs in the first Ft. Calhoun incident failed at a stress of 15,500 psi, which is just about equal to the residual stress in the studs when they are held by the nuts. Therefore, we will assume, based on judgment, that after 10 years the studs will fail and small (S2) LOCAs will result.

During this 10-year latency, there is a good chance that the corrosion will be discovered and the studs replaced. We will assume, again based on judgment, that 9 times out of 10 the studs will be discovered and replaced before they fail. The result is then an S2 frequency of $(6.6 \times 10^{-2}) \times (0.1)$ or 6.6×10^{-3} S2 LOCAs/PWR-year.

Consequences

An S2 (very small) LOCA can result in a wide spectrum of consequences, depending on whether or not the engineered safety features function. We have recalculated the WASH-1400 release category probabilities with and without this new source of S2 LOCAs. The appropriate probability estimates are in Table V 3-14 of WASH-1400. The results are:

RELEASE CATEGORY	FREQUENCY (PER R-Y)	CURIES RELEASED	Δ Fn Rn
PWR-1	6.6×10^{-7}	1.2×10^{9}	7.9×10^{2}
PWR-2	2.0×10^{-6}	9.3×10^{8}	1.8×10^{3}
PWR-3	2.0×10^{-5}	5.2×10^{8}	1.0×10^{4}
PWR-4	2.0×10^{-6}	2.8×10^{8}	5.5×10^{2}
PWR-5	2.0×10^{-6}	1.3×10^{8}	2.6×10^{2}
PWR-6	1.3×10^{-5}	1.0×10^{8}	1.3×10^{3}
PWR-7	1.3×10^{-4}	2.1×10^{6}	2.8×10^{2}
		TOTAL:	1.5×10^{4} Ci/PWR-year

Costs

The proposed fix is to visually inspect the bolts whenever UT inspection is required. This is a minor increase in surveillance, and should not require more than one man-week of extra effort per plant per refueling outage, provided the thermal insulation is readily removable. For the first inspection we will triple this to allow for insulation removal. We will double the 40-year result to allow for administrative overhead. At 100K per staff year, the cost is 0.11 million dollars.

For NRC cost, we will assume two man-months to send out a generic letter. This is 1.7×10^{-2} million dollars.

Score

There are 43 PWRs operating. The score is therefore:

$$S = \frac{(43)(1.5 \times 10^{4})}{(1.7 \times 10^{-2}) + (43)(0.11)}$$

$$S = 1 \times 10^{5} \text{ Ci/year/million dollars.}$$

It should be noted that this score does not include refinements such as the avoided cost of cleanup after an accident, or after a successfully-mitigated small LOCA. Such refinements would result in a still higher score.

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EXAMINATION OF #2 STEAM GENERATOR
COLD LEG PRIMARY MANWAY COVER
STUDS FROM MAINE YANKEE

J. F. HALL
G. C. FINK

APRIL, 1982

COMBUSTION ENGINEERING, INC.
NUCLEAR POWER SYSTEMS
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1.0 SUMMARY

Primary manway cover studs from #2 steam generator (cold leg) Maine Yankee were examined at C-E because several studs fractured during their removal from the manway flanges. The examinations included visual examination of failed studs, tensile testing of cracked studs, metallographic examination by light and scanning electron microscopy of failed studs, analysis of lubricant and Furmanite scrapings and hardness testing. Results showed that the failures were the result of environmentally assisted cracking (stress corrosion or hydrogen embrittlement cracking). Other than cracking, the studs showed no significant general or localized corrosion. The microstucture and hardness of the studs were typical for SA 540 Grade B24 alloy Steel. Qualitative analysis of lubricant scrapings showed that Ni, Cu, S, and Mo were present, with the current lubricant the source of the Ni and the source of the other elements unidentified. However, Cu, Mo and S are also used in lubricants. Traces of Mo and/or S were detected on the crack surfaces. Examination of the Furmanite sealant used to seal the leaking manway detected no contaminant elements that are known to be aggressive toward alloy steels.

2.0 INTRODUCTION

During a March 1982 maintenance outage at Maine Yankee, five steam generator primary manway cover studs failed during removal of the manway cover. Inspection of the remaining 15 studs by non-destructive techniques indicated that five additional studs were cracked. The studs were fabricated from SA 540 Grade B24 alloy steel. The affected manway was on the cold leg of steam generator #2. During the current fuel cycle, the manway leaked although the leakage never exceeded the technical specification limits for leak rates. Efforts to stop the leak included retorquing the studs to hydrotest levels (1100 ft-lbs) and later injecting Furmanite sealant into the stud holes. All of the studs in the leaking manway were replaced with new studs during the outage.

After removal from the manway and completion of NDE at the site, 19 of the studs were shipped to Combustion Engineering for examination. Examinations conducted at C-E included visual examination, optical metallography, scanning electron microscopy supplemented by energy dispersive spectrometry (EDS) and wavelength dispersive spectrometry (WDS), tensile testing of cracked studs and hardness measurements. This report documents the details of the various examinations and the results obtained.

3.0 VISUAL EXAMINATION

The as-received condition of the first two studs received at C-E is shown in Figures 3-1 and 3-2. Neither of these studs had been cleaned or decontaminated prior to shipment. Two pieces of the fractured stud identified as number 11, and one piece of the fractured stud identified as number 4 were received. Neither stud was visibly corroded. Most of the visible surfaces in the shank region were covered by a thin, tenacious, black film. Also observed were a few areas with white deposits, and a few small areas of shallow pitting, and thicker white deposits that were suspected to be the remains of the Furmanite sealant. This white deposit with a fiberous appearance, was observed in the threaded region and on the shank adjacent to the threads. Samples of this material were removed for further analyses.

Examination of the stud surfaces indicated that only minor general and pitting corrosion were evident. However, the presence of thick black deposits in the threaded region may have obscured the observation of cracks that were present. The deposits were presumably the remains of the lubricants used on the studs which were tightly adherent and could be removed only with forceful scraping. Samples of these suspected lubricant deposits were collected for further analyses.

The fracture surface of failed stud 4 was relatively flat; suggesting that the failure occurred without significant ductile yielding. Much of the surface was covered with a yellow-white deposit. The remainder of the surface was covered with a black oxide. The entire fracture of surface stud 11 was covered with a black oxide. The crack surface was essentially flat, but did have a central ridge running part way across the crack surface. The fracture surface exhibited numerous secondary cracks which were evident under low power magnification. Extensive crack branching and secondary crack formation is indicative of environmentally assisted cracking (stress corrosion cracking or hydrogen embrittlement cracking).

The conditions of three additional studs subsequent to tensile testing to determine remaining load bearing capability are shown in Figures 3-3, 3-4 and 3-5. These three studs had been cleaned at the site as part of the NDT program and were further cleaned on arrival at C-E to remove the residues from liquid penetrant testing. The surface conditions of these studs were similar to the conditions of broken studs 4 and 11. Only mild general corrosion and small areas of shallow pitting (other than the cracks) were observed. The entire surface of each stud was covered with a black tenacious film.

Tensile testing was conducted on a 60,000 lb capacity Riehle univeral testing machine at room temperature. Studs identified by C-E as A and B failed at 2,000 and 29,300 lbs., respectively. The stud identified by C-E as C was loaded to 60,000 lbs without failing.

The fracture surfaces of the two studs that failed during tensile testing showed that most of the cross section had been consumed by the cracking process. The cracked areas were relatively flat, were covered with black oxides and had a brittle-like appearance with no evidence of plastic deformation except where the final fracture occurred during tensile loading.

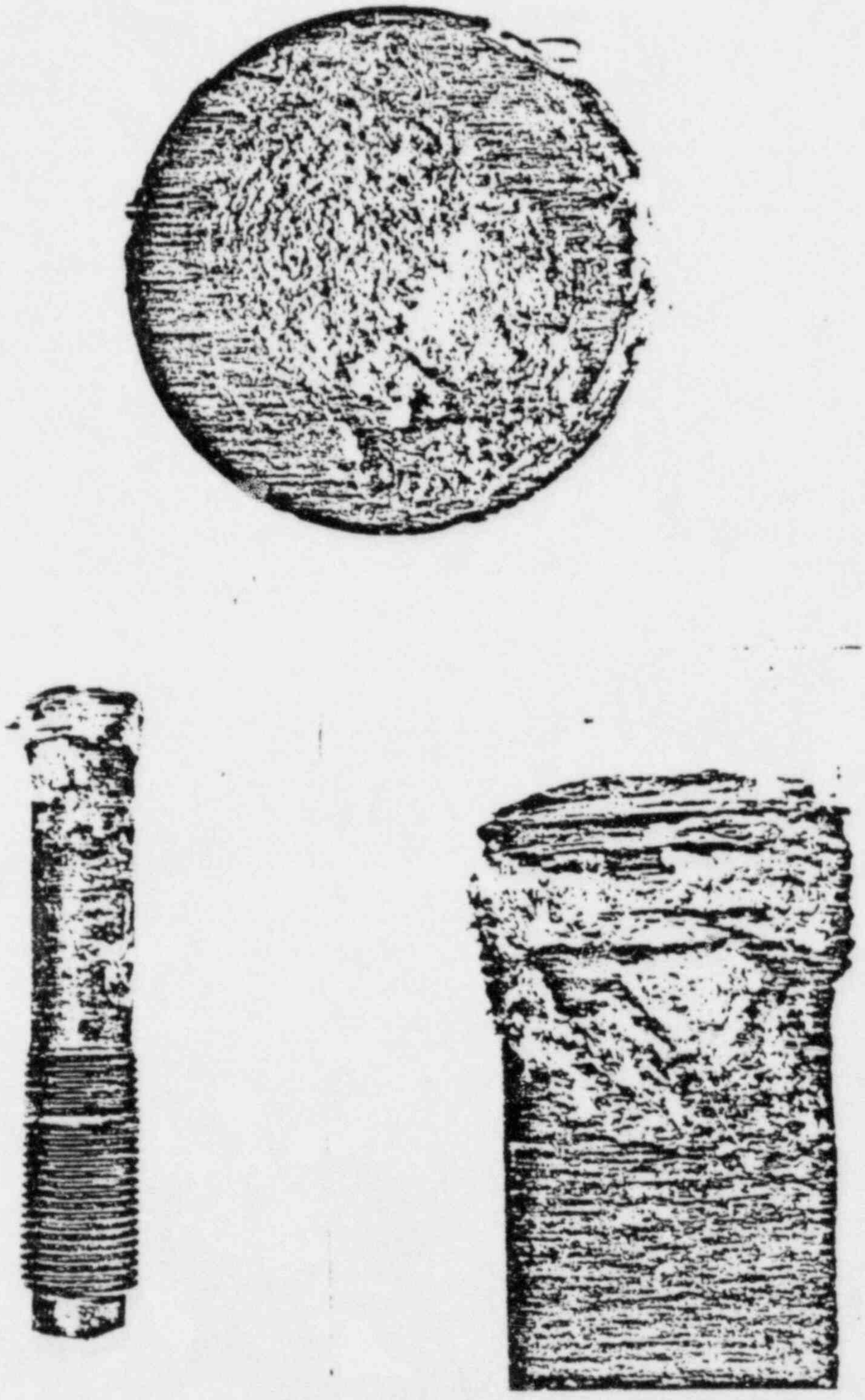


Figure 3-1. Views of the Fractured Stud Identified as 4.
(Negatives #53199, 53203, 53204)

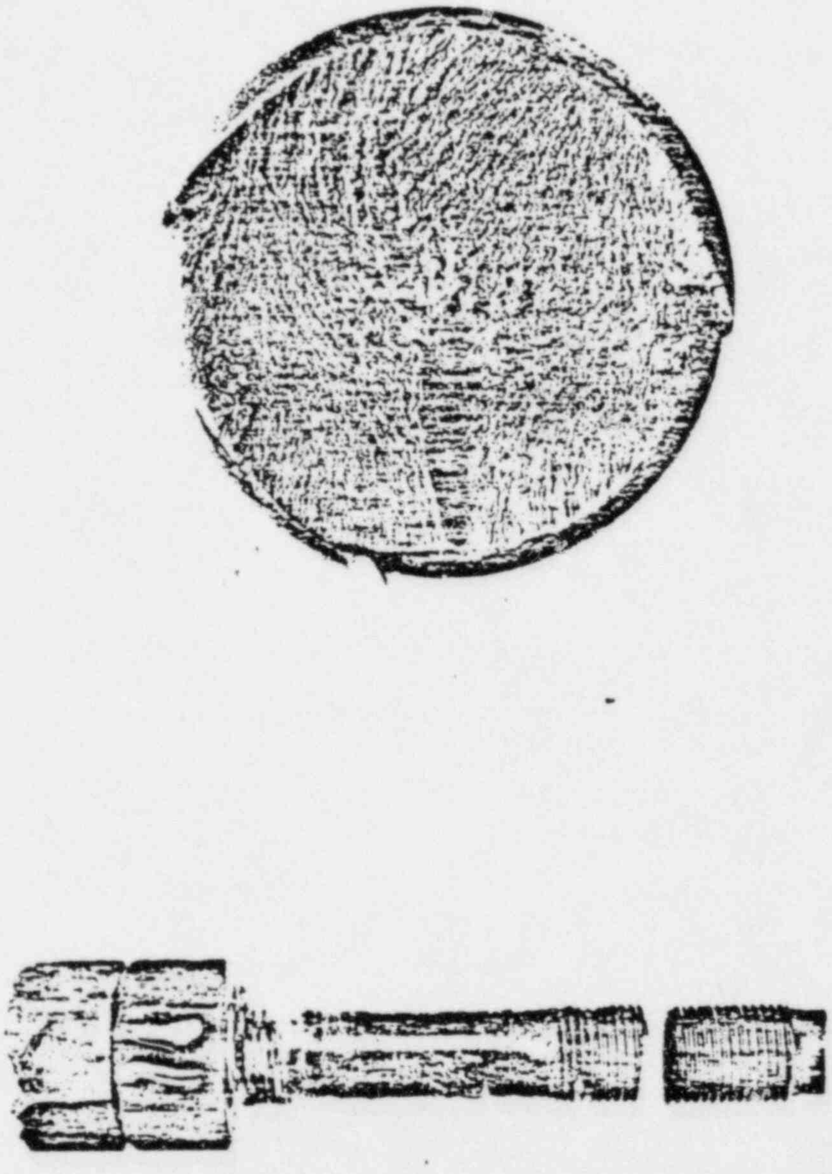
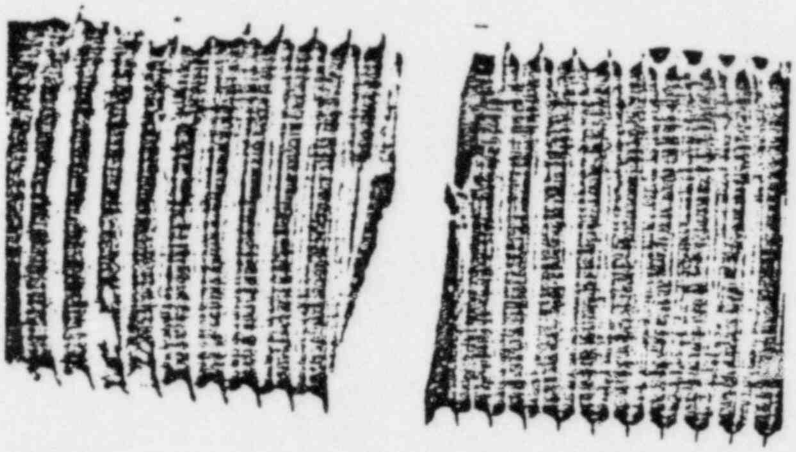


Figure 3-2. Views of the Fractured Stud Identified at 11.
(Negatives #53200, 53203)



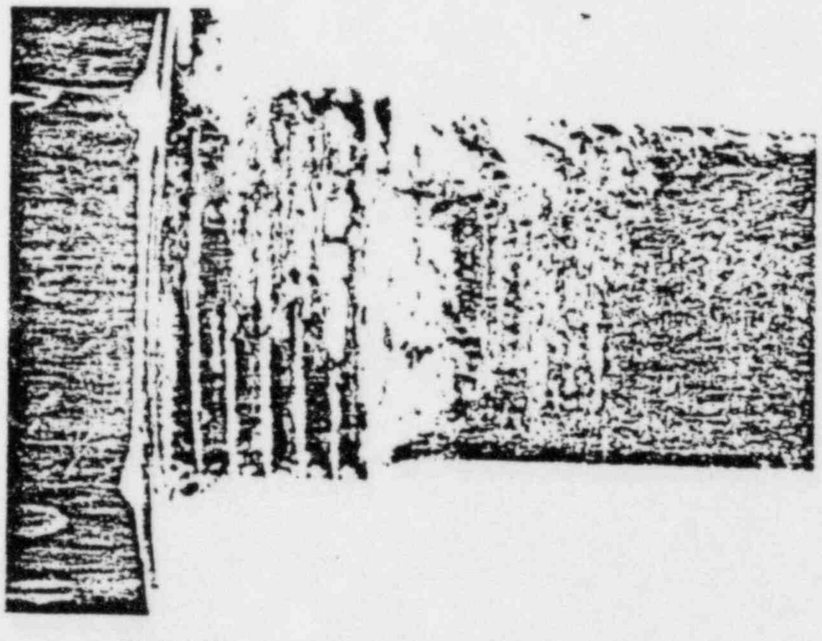


Figure 3-2. Continued (Negatives #53201, 53206)

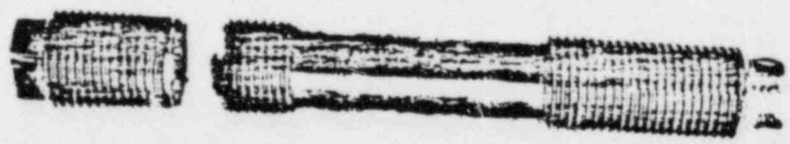


Figure 3-3. Views of Stud "A" After Tensile Testing.
(Negatives 53497, 53499)

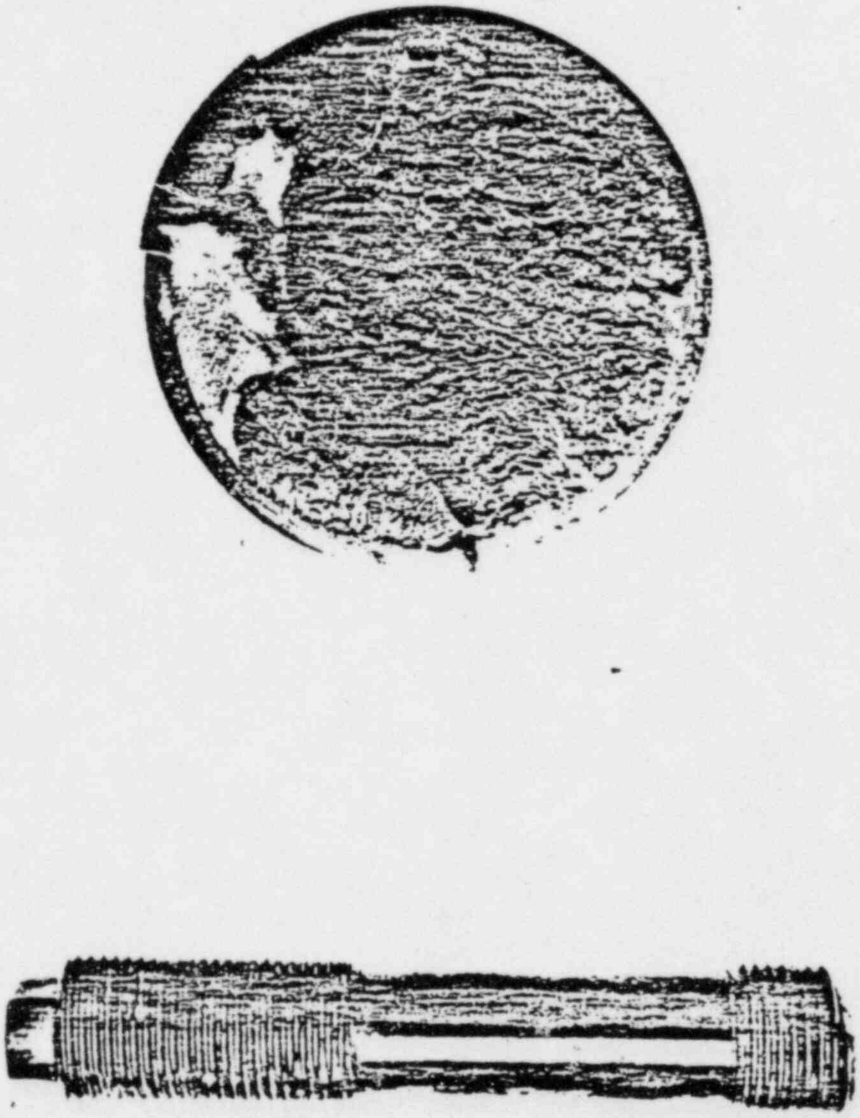


Figure 3-4. Views of Stud "B" After Tensile Testing.
(Negatives 53498, 53500)

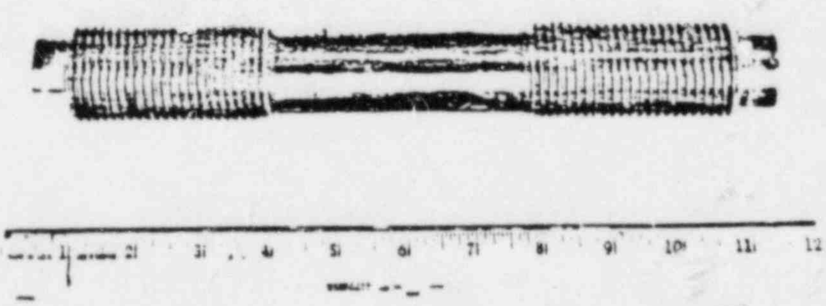


Figure 3-5. View of Stud "C" After Tensile Testing.
(Negative 53501)

4.0 LUBRICANT ANALYSES

The samples of lubricant scraped from the threaded regions of studs 4 and 11 were qualitatively analyzed to determine the elemental species present. The objective of these analyses was to determine if elements were present in the lubricant that are aggressive toward corrosion of alloy steel fastener materials. The instruments used in the analyses were a KEVEX 7000 energy dispersive spectrometer (EDS) and an ETEC wavelength dispersive spectrometer (WDS), both of which were coupled to an ETEC Autoscan scanning electron microscope. The EDS qualitatively indicates the presence of elements with atomic number 11 or greater (Na and above). However, because of the close proximity of two of the characteristic energy peaks for Mo and S, EDS cannot determine if S is present in a sample also containing Mo. The WDS can distinguish between Mo and S and was used for that purpose in this investigation.

The lubricant samples were mounted in carbon paste on an aluminum pedestal. Four EDS analyses were obtained from stud 4 lubricant scrapings and two analyses from the stud 11 scrapings. In addition, two WDS analyses for Mo and S were obtained for each sample.

Figures 4-1 to 4-6 show the lubricant sample EDS analysis results and low magnification scanning electron micrographs of the areas analyzed. Table 4-1 summarizes in tabular form the EDS results. Table 4-2 summarizes the results of the WDS analyses.

Fel-Pro N-5000, a nickel base lubricant, has been used at Maine Yankee in recent years as a manway cover stud lubricant, and thus, strong Ni indications in the lubricant scrappings were expected. The Fe indications probably resulted from iron oxides removed from the studs when the lubricant scrappings were obtained. Si and Ca may have been present in the sample as particles of Furmanite sealant removed along with the lubricant samples. The sources of the Cu, S and Mo present in the lubricant is not immediately evident. Contaminants in the Fel-Pro N-5000 would not appear to be the origin of these species since they are controlled in the lubricant to levels which would make detection by EDS difficult. Both Cu, and Mo and S (as MoS_2) are used as lubricants. Either or both of these type lubricants may have been used early in the stud life and not completely removed from the studs.

5.0 FURMANITE ANALYSES

The sample of Furmanite sealant removed from stud 11 was analyzed by EDS. Results of an analysis and a low magnification scanning electron micrograph are shown in Figure 5-1, and are included in the Table 4-1 tabulation. The results show Si as the major species present with significant amounts of Ca, Mg and Fe present and a lesser amount of Al present at the area examined. The Fe may be from oxides removed from the stud and the Al may be from the pedestal mount on which the Furmanite was mounted. There were no indications of Cl or S in this sample.

Table 4-1

SUMMARY OF EDS RESULTS

FROM LUBRICANT AND FURMANITE SCRAPINGS

			ELEMENTS PRESENT									
Material	Stud	Area	Ni	Fe	Cu	S/Mc	Si	Ca	Al	K	Mg	Figure
Lubricant	4	1	B	C	D	D	A	--	--	--		4-1
		2	A	E	D	E	E	--	E	--		4-2
		3	A	C	D	E	C	--	E	--		4-3
		4	A	C	D	E	B	E	--	E		4-4
	11	1	C	A	C	D	C	E	--	--		4-5
		2	B	A	D	E	B	E	--	--		4-6
Furmanite	11	1	--	C	--	--	A	C	D	--	B	5-1

Note: The presence of a letter indicates the presence of a specific element. The letters indicate the relative height of the specific peaks, as follows:

- A = The major peak, or any other peak greater than 75% of the major peak.
- B = 50-74% of the major peak
- C = 25-49% of the major peak
- D = 10-24% of the major peak
- E = Less than 10% of the major peak
- -- = Element not detected

Table 4-2

WDS ANALYSIS RESULTS

Stud	Area	Mo	S
4	1	--	X
	2	--	X
11	1	X	X
	2	X	X

Note: X = indicates element present

- = indicates element not detected

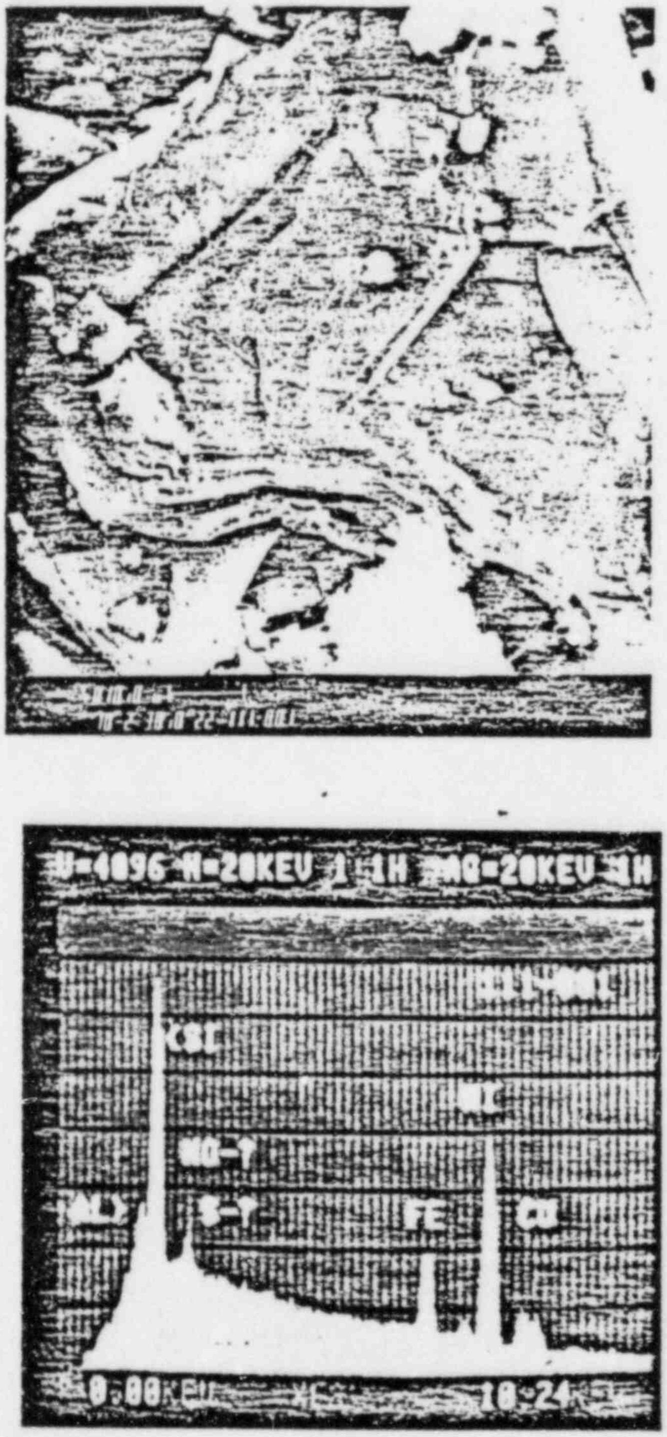


Figure 4-1. Scanning Electron Micrograph and EDS Results from Area 1 of the Failed Stud 4 Lubricant Scrapings. (1000X)

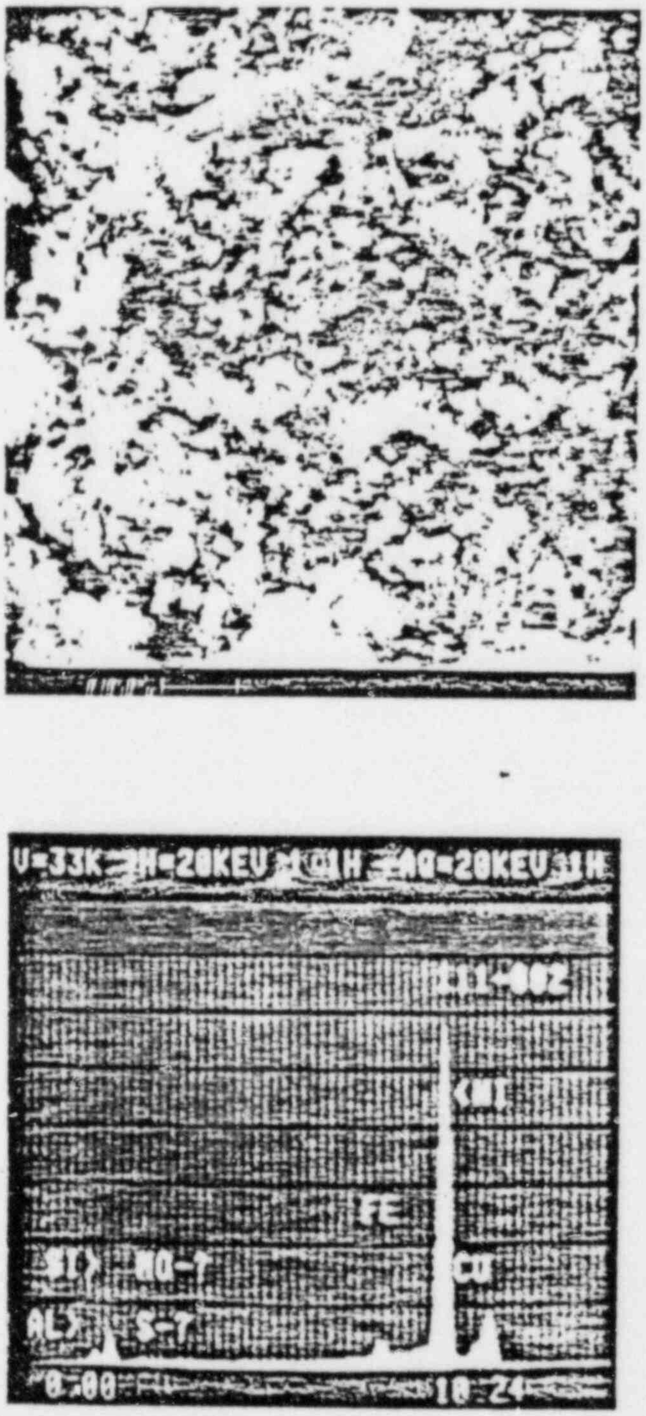
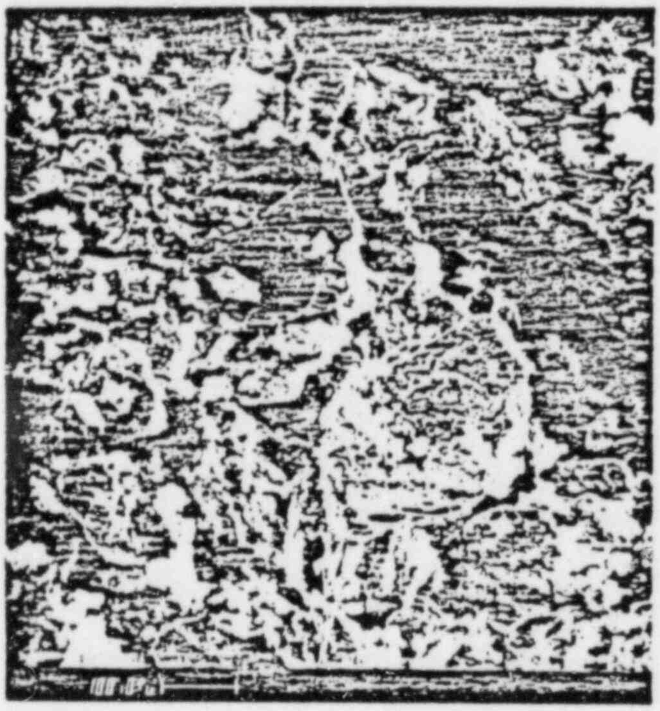


Figure 4-2. Scanning Electron Micrograph and EDS Results from Area 2 of the Failed Stud 4 Lubricant Scrapings. (1000X)



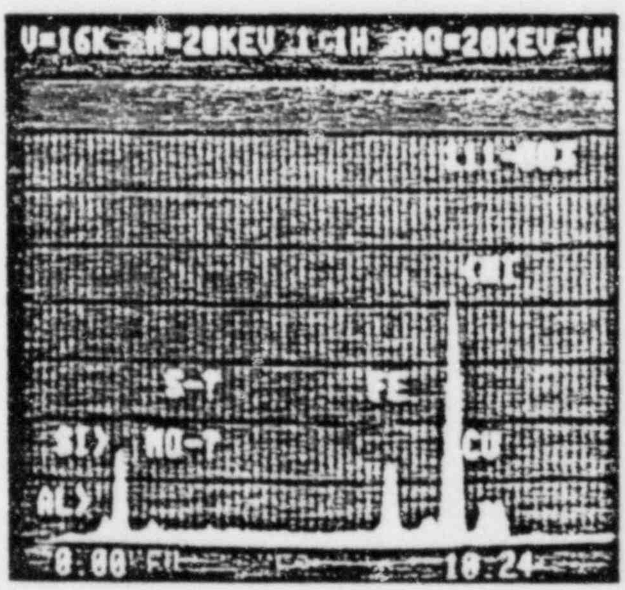


Figure 4-3. Scanning Electron Micrograph and EDS Results from Area 3 of the Failed Stud 4 Lubricant Scrapings. (100X)

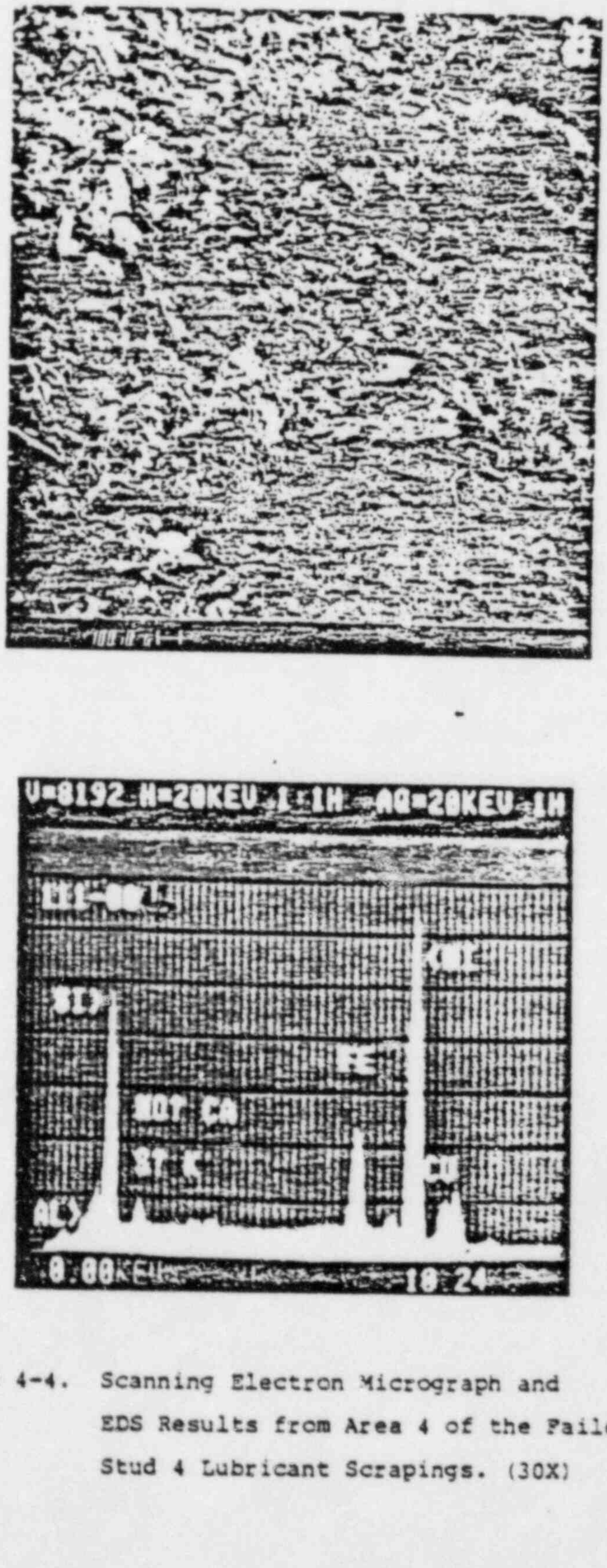


Figure 4-4. Scanning Electron Micrograph and EDS Results from Area 4 of the Failed Stud 4 Lubricant Scrapings. (30X)

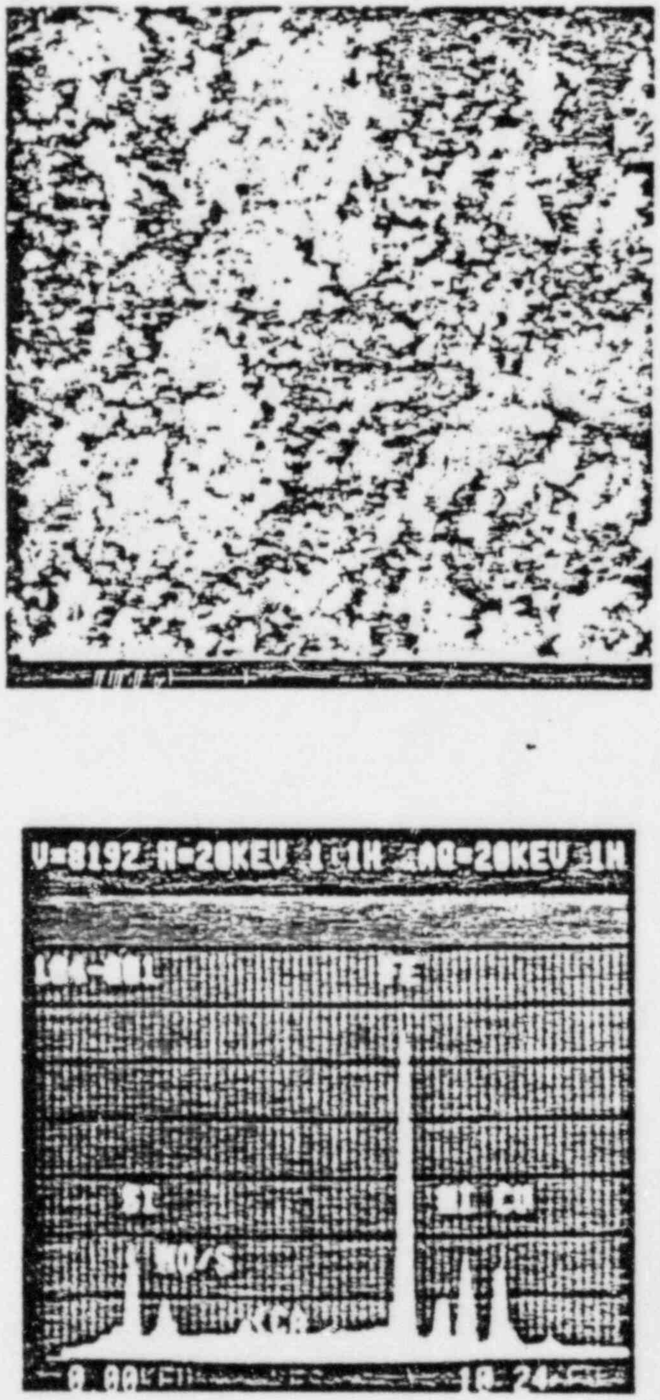
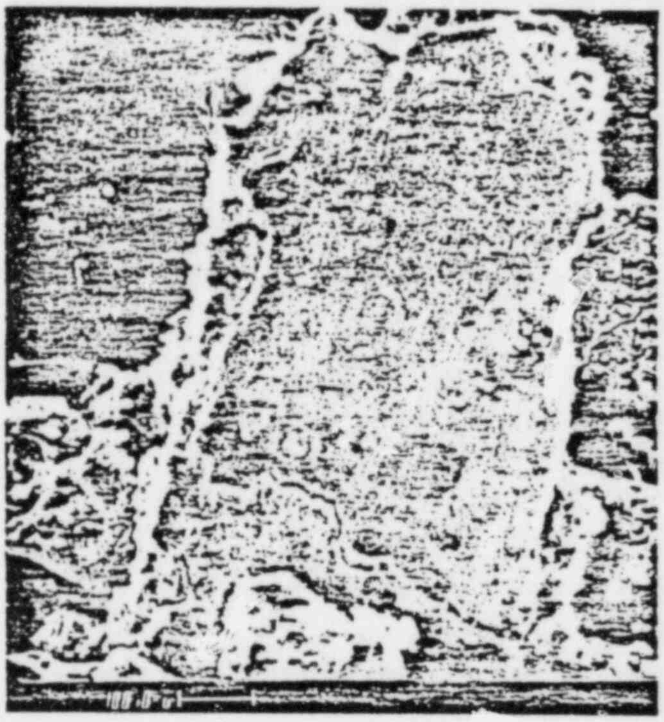


Figure 4-5. Scanning Electron Micrograph and EDS Results from Area 1 of the Failed Stud 11 Lubricant Scrapings. (1000X)



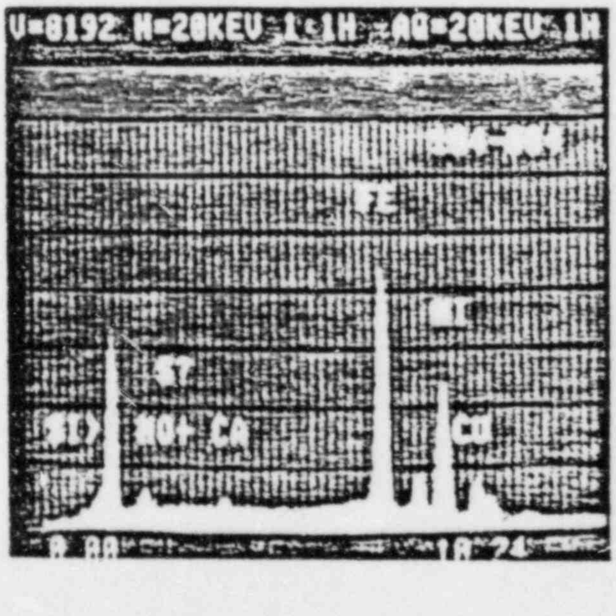
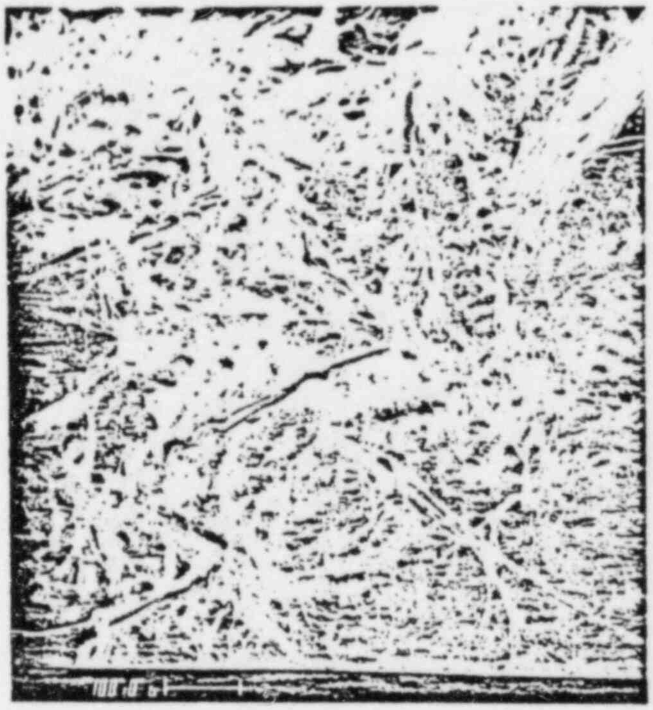


Figure 4-6. Scanning Electron Micrograph and EDS Results from Area 2 of the Broken Stud 11 Lubricant Scrapings. (100X)



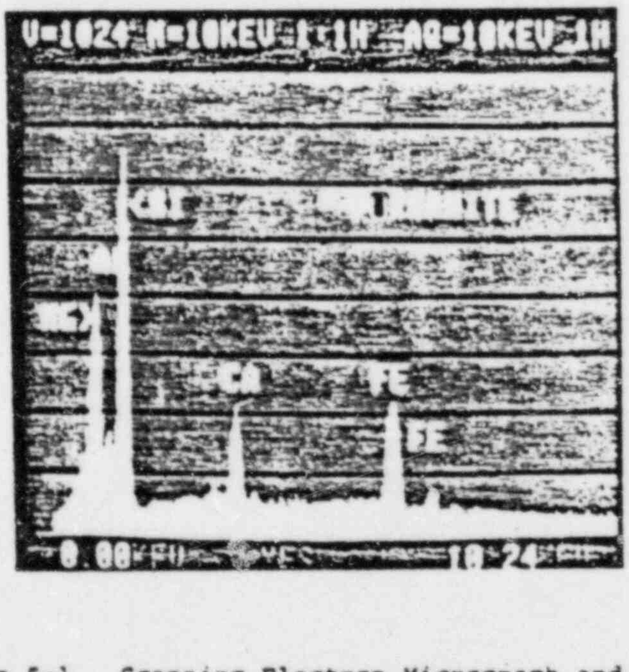


Figure 5-1. Scanning Electron Micrograph and EDS Results from Failed Stud 11 Furmanite Scrapings. (100X)

6.0 METALLOGRAPHIC EXAMINATIONS

6.1 OPTICAL MICROSCOPY

Studs 4 and 11 were longitudinally sectioned to produce metallographic samples of their threaded regions. Specimens were mounted, ground on SiC papers, polished in Al_2O_3 slurry and examined in both as-polished and after each of several etchants was applied. Typical photomicrographs of these specimens are shown in Figures 6-1 to 6-4.

The microstructures in both specimens were typical of quenched and tempered martensite with very fine grained microstructures making the resolution of grain boundaries difficulty even at high magnifications. There was no apparent differences in the microstructures of the two specimens.

Figure 6-1 to 6-3 are photomicrographs of part of one thread, including the thread root, of stud 4. This thread root was adjacent to the thread root from which the major failure crack originated. Two short cracks are evident in the figures; one of which originated near the thread root and one of which originated further up the flank. The higher magnification photomicrographs suggest the cracks followed prior austenite grain boundaries, at least over parts of their lengths. Even these short cracks show some crack branching, a phenomena usually associated with environmentally induced cracking. Corrosion product was present in the cracks.

Figure 6-1 also shows corrosion had occurred to the thread and thread root. The surface was irregular and numerous small oxide filled pits were present. Both cracks appeared to originate from the bottom of surface irregularities which may have been the result of the corrosion process.

The photomicrographs in Figures 6-4 show part of a long crack in stud 11 which originated in the root of the fourth thread below the fracture and propagated at an oblique angle toward the fracture surface. This crack also appeared to follow prior austenite grain boundaries and exhibited some crack branching over its length. Corrosion product of the fastener alloy was visible in the crack.

6.2 SCANNING ELECTRON MICROSCOPY

The scanning electron microscope, supplemented with the EDS, was used to further characterize the fracture surfaces including the mode by which the cracks progressed and the elemental species present on crack surfaces. Three studs were examined including studs 4 and 11 and stud B which was load tested to failure.

A pie shape section was cut from the stud 11 fracture section to provide a fracture surface on which to verify the ability of inhibited HCl to descale an oxidized surface without destroying details on the crack surface. This descaling procedure proved to be adequate.

Figures 6-5 to 6-9 show surface features of the pie shaped segment. Figure 6-5 is a 10X scanning electron micrograph showing the sample and areas examined at high magnification. Figure 6-6 is a montague of three higher magnification (30X) SEMs. At this magnification, the most significant feature was the large number of secondary cracks in the crack surface. These are present from the outer periphery to the tip of the specimen as shown by closer examination of Figure 6-5 and Figure 6-8. Figure 6-7 shows ductile dimples present at the outer periphery of the specimen, which was the root of a thread. The secondary cracks are characteristic of environmentally induced cracking and the ductile dimples characteristic of a ductile overload failure.

The higher magnification SEMs in Figure 6-8 show the appearance of the fracture surface near the tip of this specimen, which was near the center of the stud. Although the features of the surface have been somewhat obliterated by corrosion, it is obvious that very little, if any, of the surface failed in a ductile manner. The features are more characteristic of intergranular fracture although there are some indications of transgranular crack propogation. Mixed mode cracking is also indicative of environmentally assisted cracking in high strength alloy steel fasteners.

Figures 6-10 to 6-14 are SEMs of the central area of stud 11. Figure 6-10 is a low magnification (10X) view of the central ridge running across the specimen and surrounding areas. The SEMs in Figure 6-11 are higher magnification SEMs of the central ridge and show ductile dimples. Figures 6-12 to 6-14 show no indications of ductile dimples but show indications similar to Figure 6-8. Also visible in these areas are numerous secondary cracks.

The remaining figures in this section (6-15 to 6-24) show the results of analyses of surface deposits on non-descaled fractures and within one large secondary crack in stud 11. The stud 4 surface which had much of the surface coated with deposits, and the fracture surface of stud 8 were also examined. The predominant species present was Fe, but there were also areas where Si was the major specie present. Present in very low, barely detectable quantities, were Mg, Ca, Cl, Ni, Cr, Mn, Al, P, and Mo/S. The EDS scan of the base metal (Figure 6-17) showed only Fe, Ni, Cr and Mn. Thus, the presence of Mo/S, Cl, Mg, Al, P and Ca on the fracture surfaces were apparently the result of contamination. The presence of Mo/S, which would indicate the presence of either Mo or S or both is significant since the only apparent source of these was the Mo and S detected in the lubricant scrapings.

Table 6-1

EDS RESULTS FROM STUD SPECIMENS

Stud	Location	ELEMENTS PRESENT											Figure
		Fe	Ni	Cr	Mn	Si	Mo/S	Cl	Al	P	Ca	Mg	
11	Crack	A	E	E	E	E	E	E	--	--	--	--	6-15
11	Crack Tip												
	Area 1	A	E	E	E	--	--	--	--	--	--	--	6-17
	Area 2	A	E	E	E	E	E	E	E	E	--	--	6-17
	Area 2	A	E	E	E	--	--	--	--	--	--	--	6-17
	Non-Corroded Metal	A	E	E	E	--	--	--	--	--	--	--	6-17
B	Crack Surface	B	E	--	E	A	E	E	--	--	E	--	6-18
11	Crack	A	E	--	--	D	--	--	--	--	--	--	6-19
	Crack Spot 1	E	--	--	--	A	E	--	--	--	E	A	6-20
	Area 1	A	E	--	--	E	--	E	--	--	--	--	6-20
	Area 2	A	E	--	--	D	--	--	--	--	--	--	6-20
4	Deposit - Area 1	A	E	E	--	C	E	E	--	--	--	Mg	6-21
	Area 2	A	E	--	--	E	--	--	--	--	--	--	6-21
	Area 3	A	E	--	--	E	--	--	--	--	--	E	6-21
4	Deposit	A	E	E	E	E	E	--	--	--	--	E	6-22
4	Deposit	A	E	--	E	E	--	--	--	--	--	--	6-23
4	Deposit	A	E	--	E	--	--	--	--	--	--	--	6-24

Notes: The presence of a letter indicates the presence of a specific elemen
The letters indicate the relative height of the specific peaks, as follow

A = The major peak, or any other peak greater than 75%

B = 50-74% of the major peak

C = 25-49% of the major peak

D = 10-24% of the major peak

E = Less than 10% of the major peak

- = Element not detected

Figure 6-1. Cracks in the Root of a Thread in Failed Stud 4. (As-Polished, 100X)

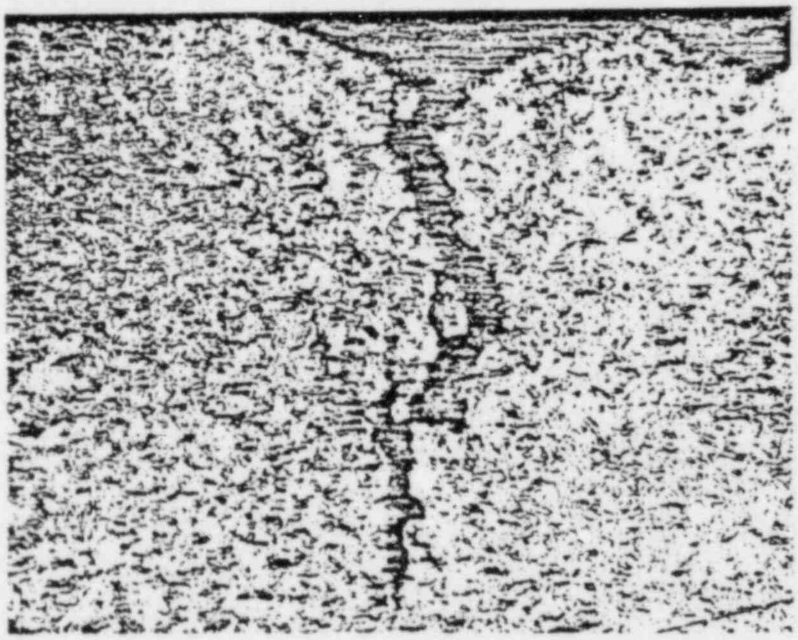


Figure 6-2. Photomicrograph of a Crack in Failed Stud 4. (Villella's Etch, 500X, Negative 53272)

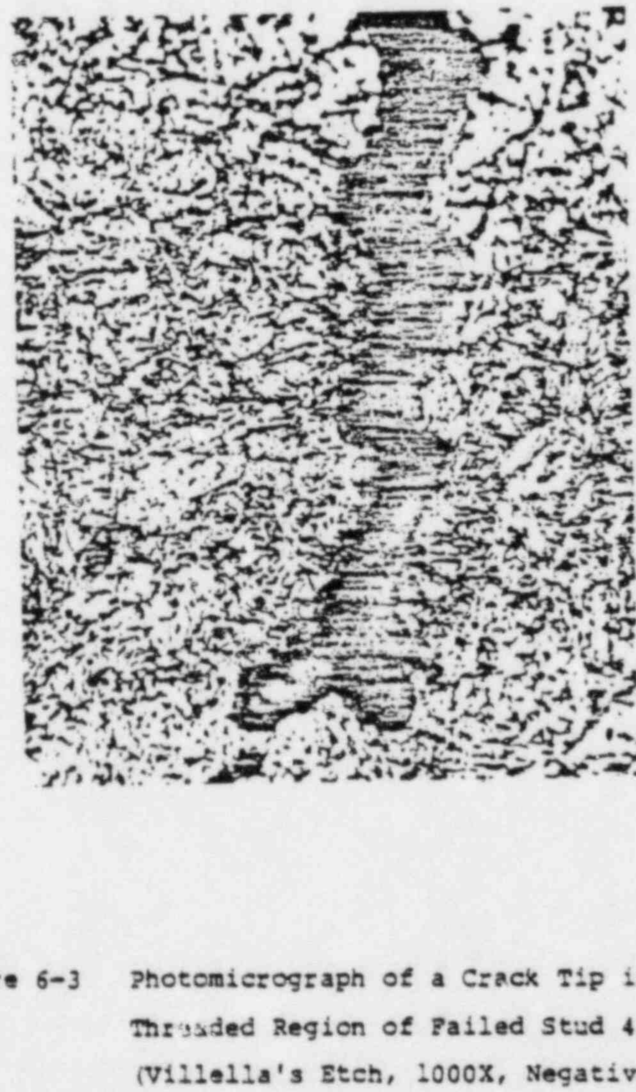
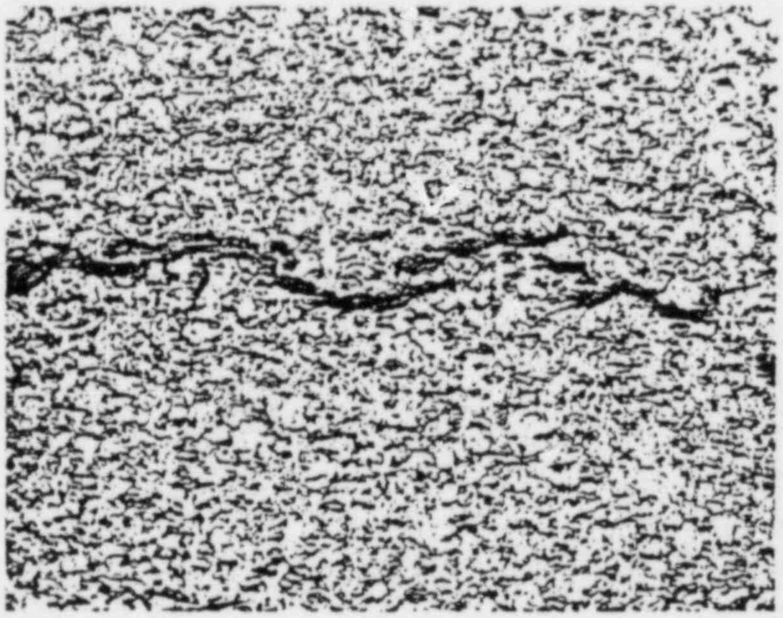
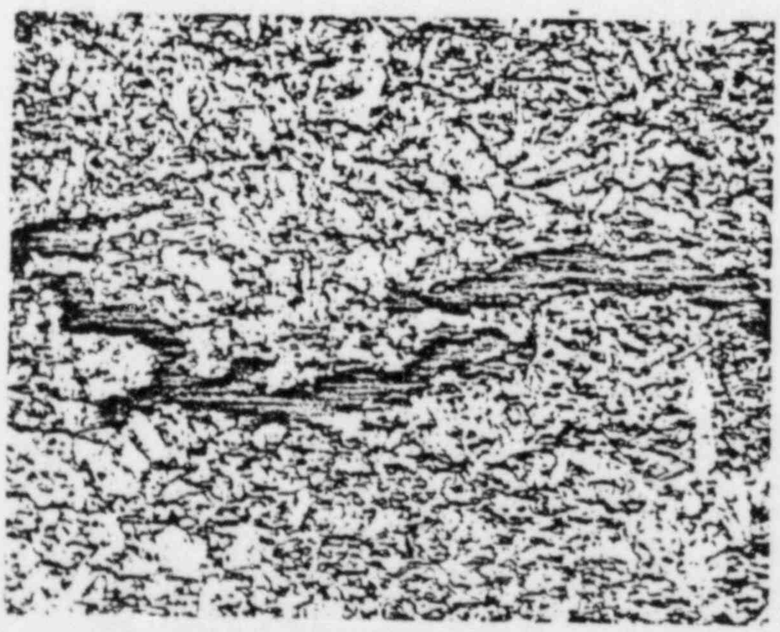


Figure 6-3 Photomicrograph of a Crack Tip in the Threaded Region of Failed Stud 4. (Villella's Etch, 1000X, Negative 53277)



500X



1000X

Figure 6-4. Photomicrographs of Part of a Crack in the Threaded Region of Failed Stud 11. (Villella's Etch, Negatives 53275, 53276)

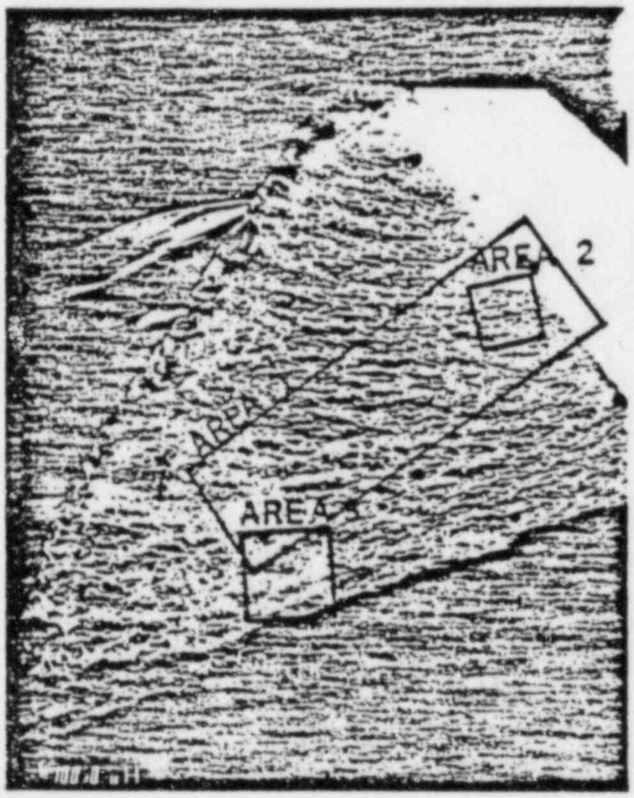


Figure 6-5 Low Magnification Scanning Electron Micrograph of a Section of the Crack Surface of Failed Stud 11 Showing Areas Examined at High Magnification. (10X, Negative 52792)

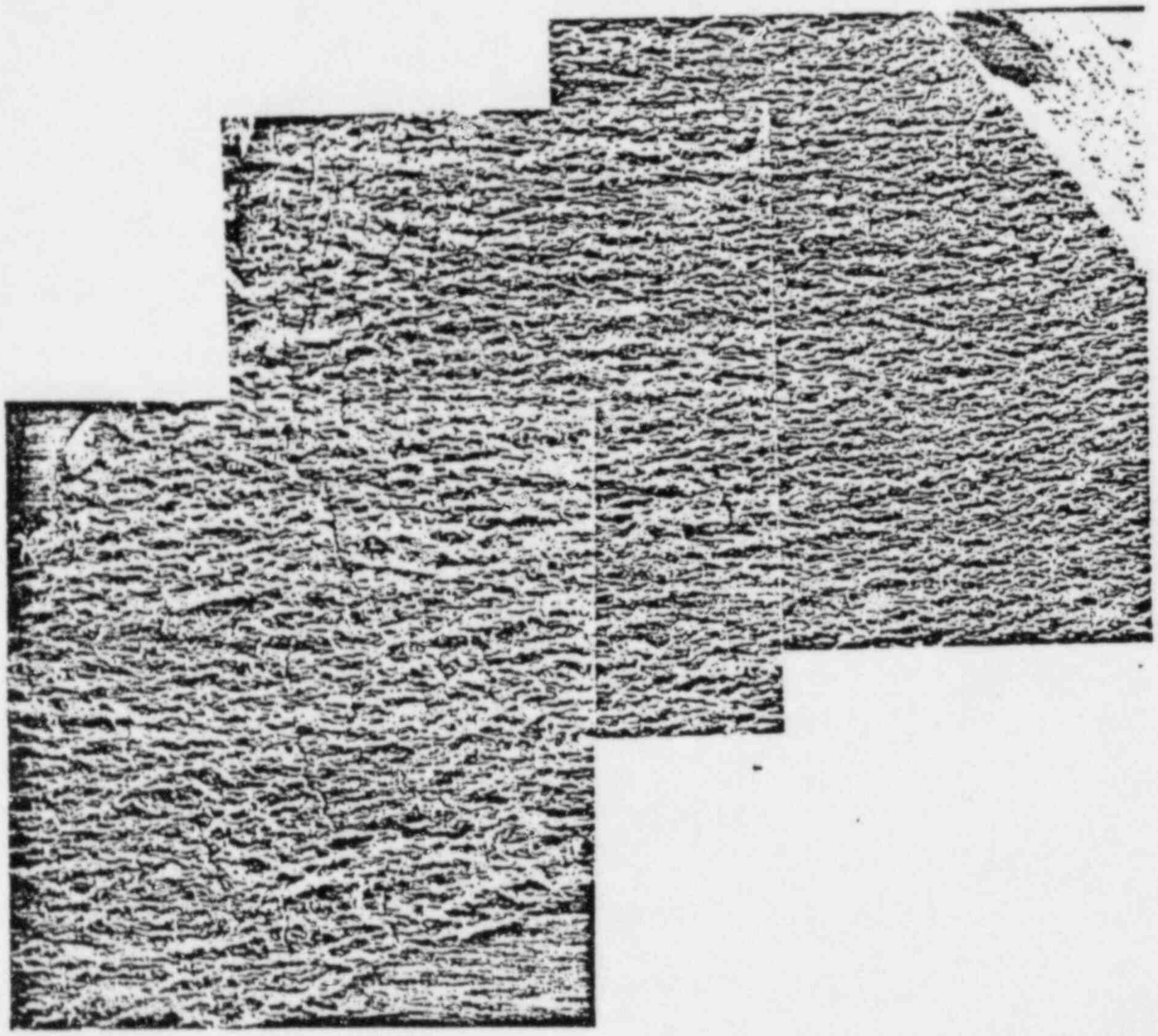
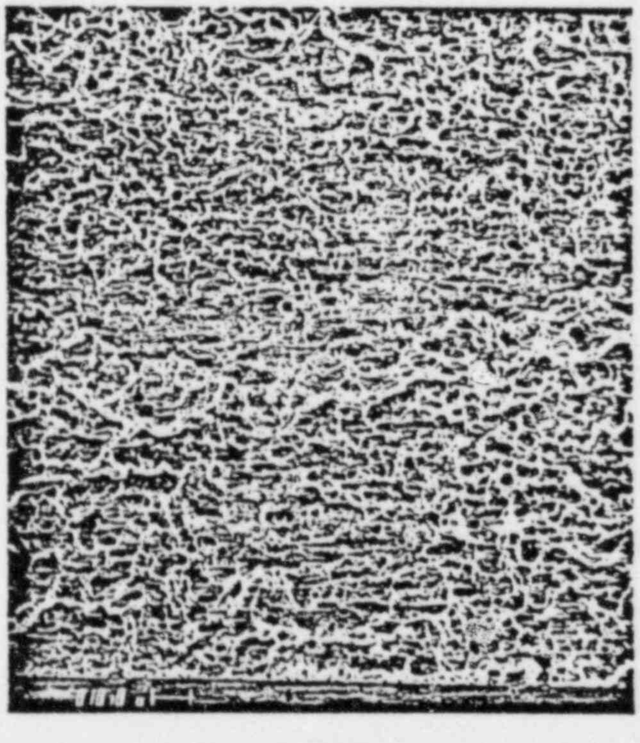


Figure 6-6. Scanning Electron Micrograph of Area 1 of Figure 6-5. (30X, Negatives 52793, 52794, 52799)

100X



500X

Figure 6-7. Scanning Electrion Micrographs of Area 2 Near the Periphery of the Specimen Shown in Figure 6-5. (Negatives 52795, 52796)

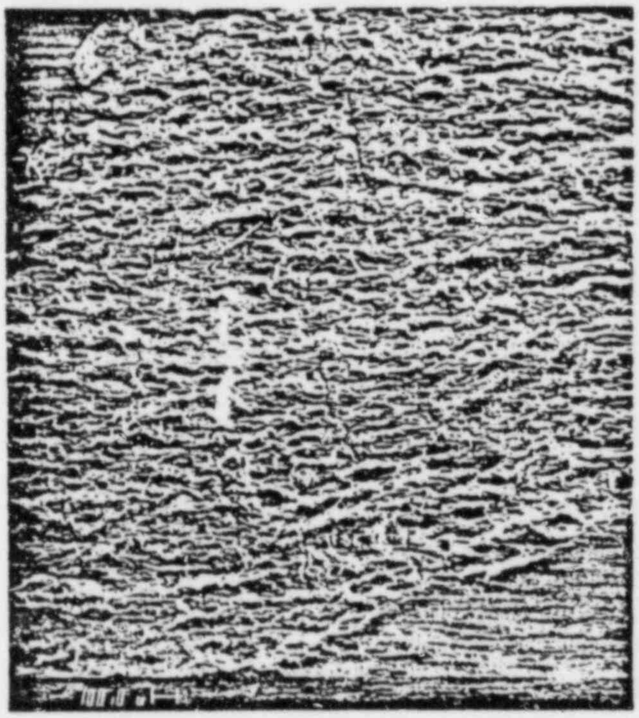
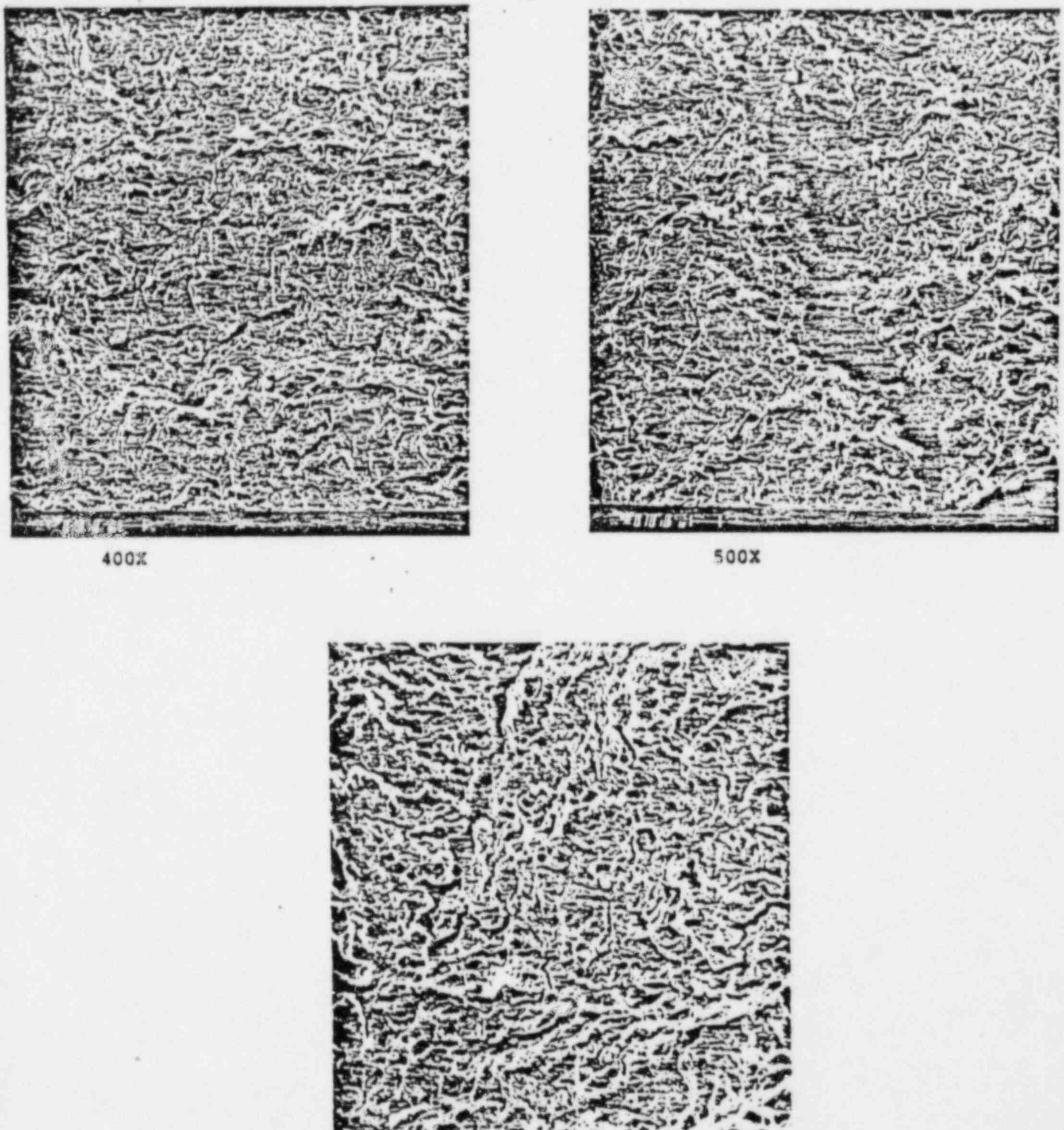


Figure 6-8. Scanning Electron Micrograph of Area 3 of Figure 6-5. (30X, Negative 52799)



400X

500X

1000X

Figure 6-9. Scanning Electron Micrographs of Area 3 of Figure 6-5. (Negatives 52797, 53798, 53302)

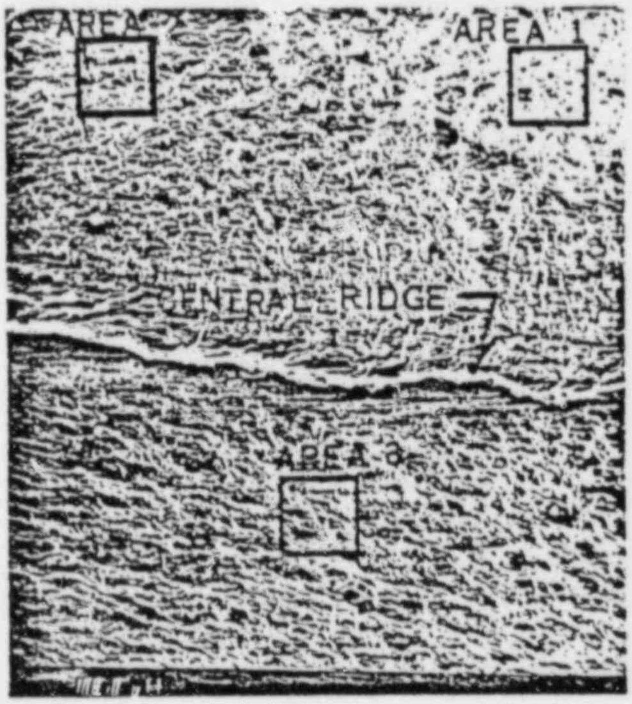
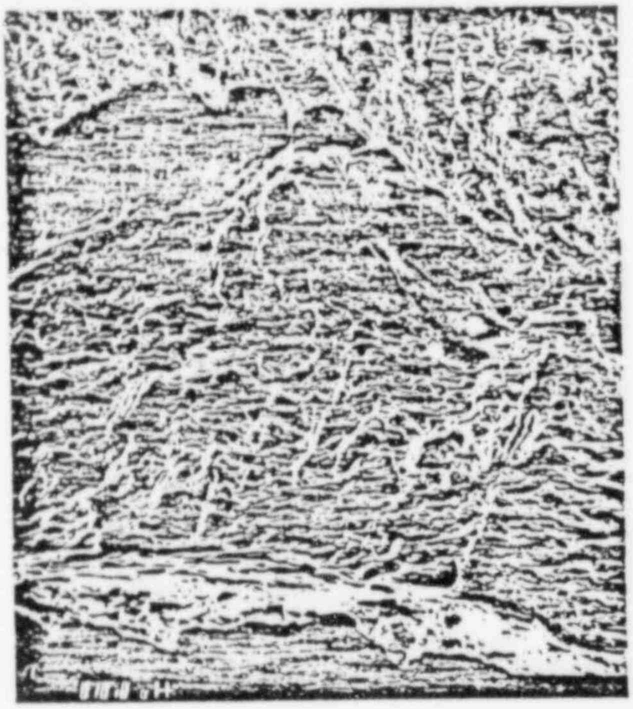
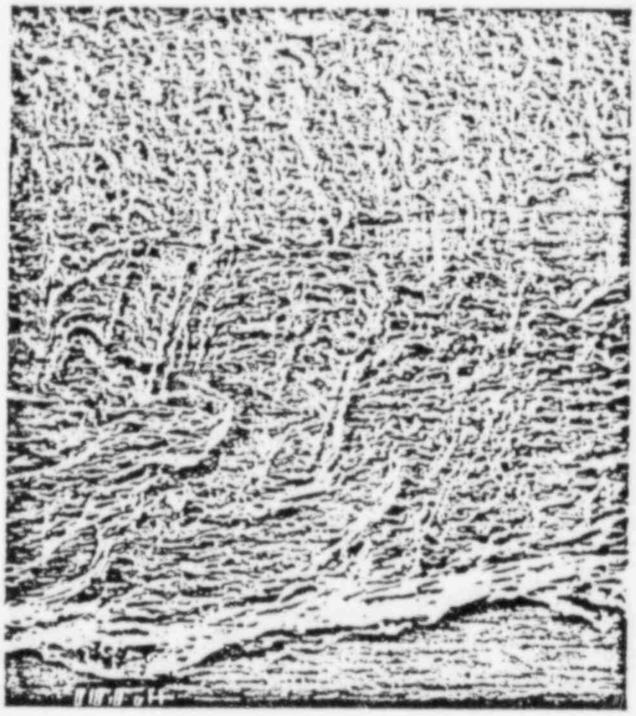


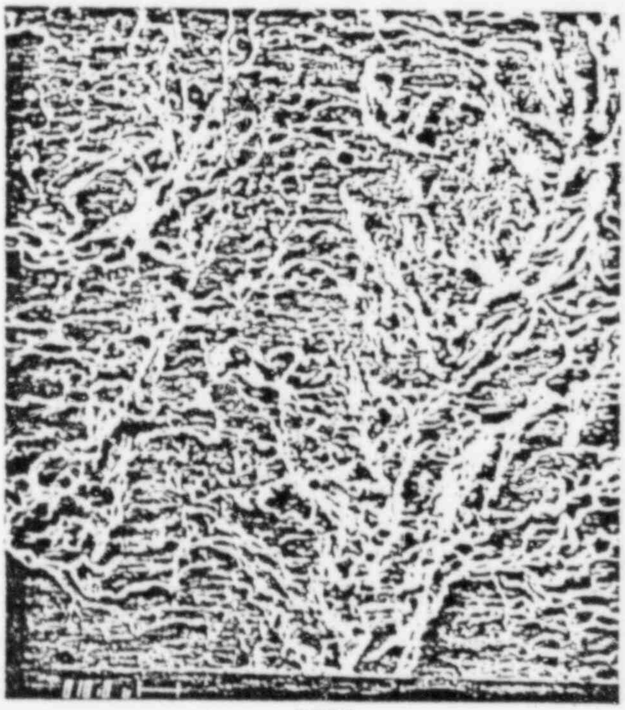
Figure 6-10. Scanning Electron Micrograph of the Central Ridge Region of Failed Stud 11. (10X, Negative 53303)



100X

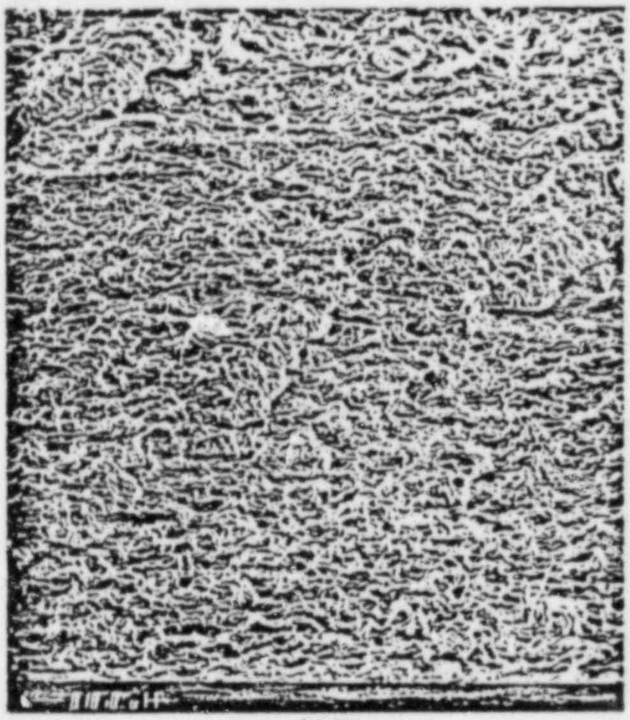


100X

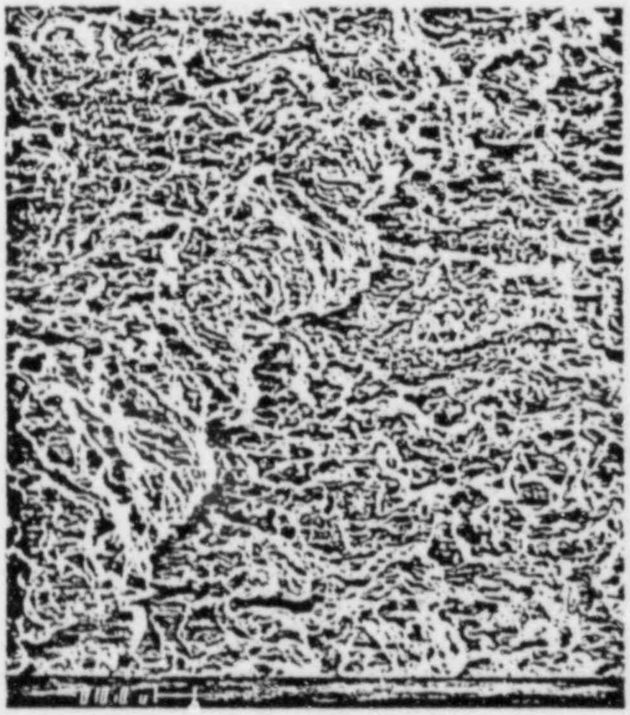


500X

Figure 6-11. Scanning Electron Micrographs of the Central Ridge Region of Figure 6-10. (Negatives 53304, 53305, 53306)

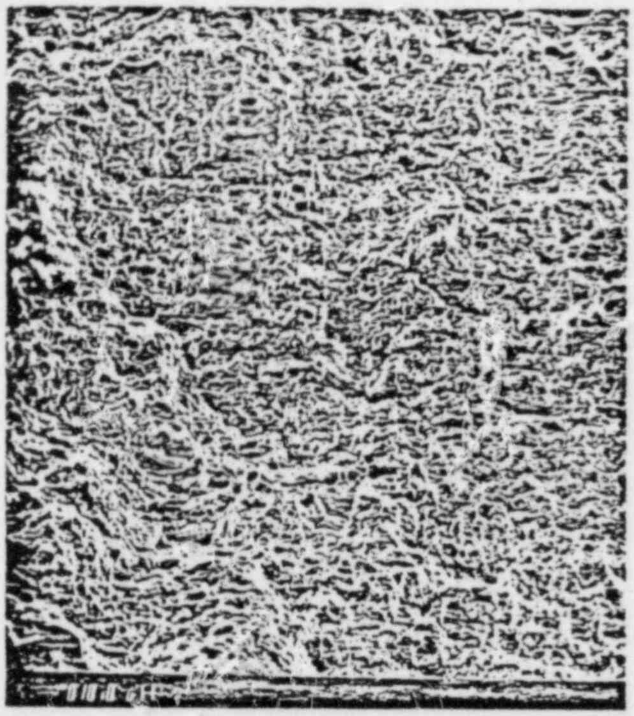


100X

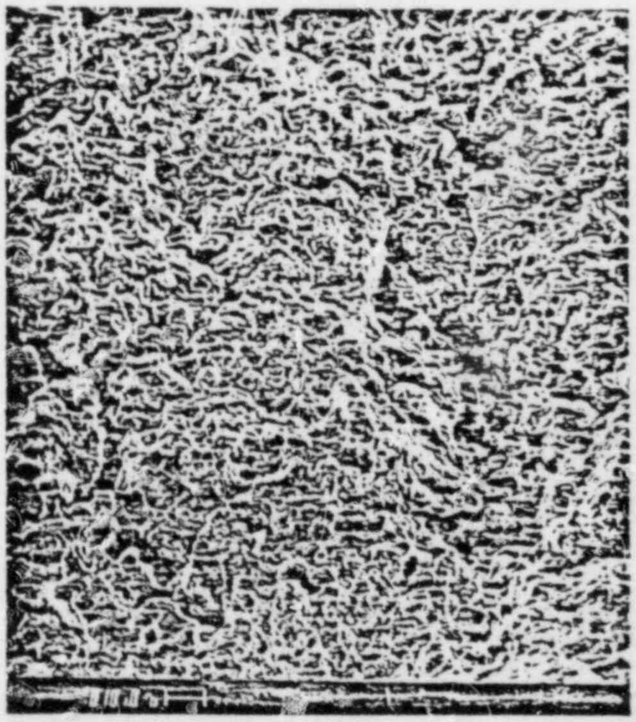


500X

Figure 6-12. Scanning Electron Micrographs of Area 1 of Figure 6-10. (Negatives 53307, 53308)

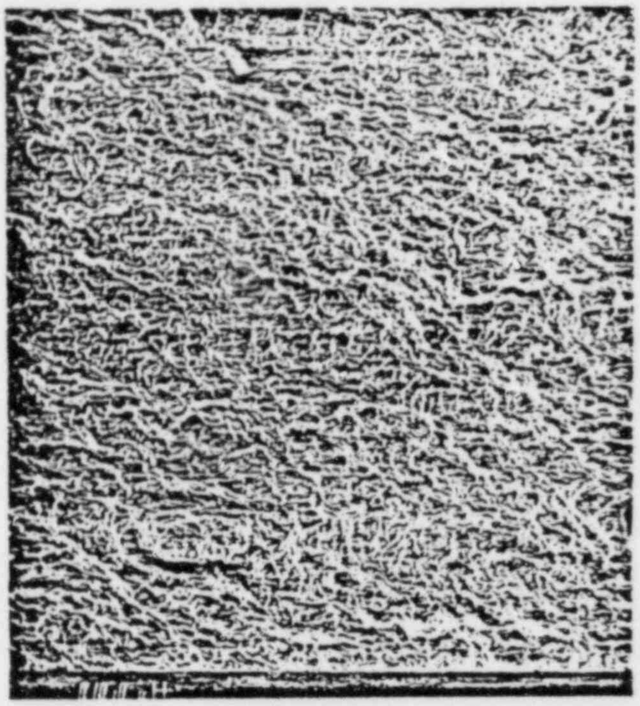


100X

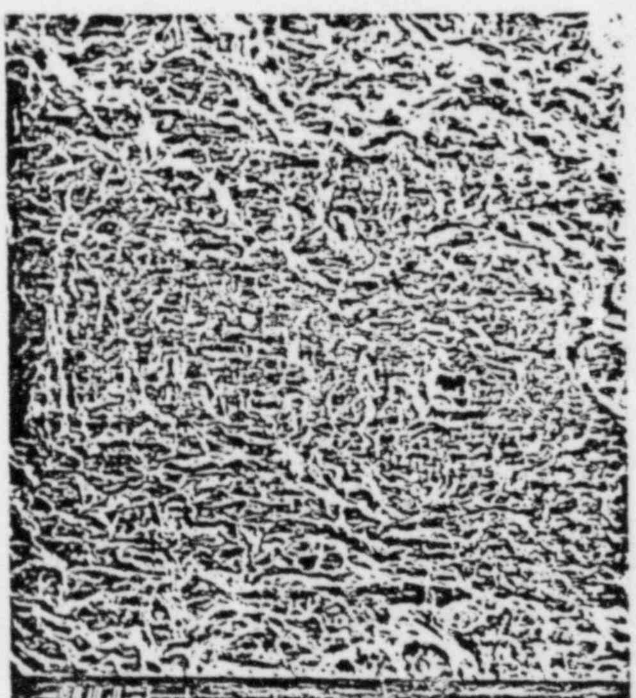


500X

Figure 6-13. Scanning Electron Micrographs of Area 2 of Figure 6-10.
(Negatives 53309, 53310)



100X



500X

Figure 6-14. Scanning Electron Micrographs of Area 3 of Figure 6-10. (Negatives 53311, 53312)

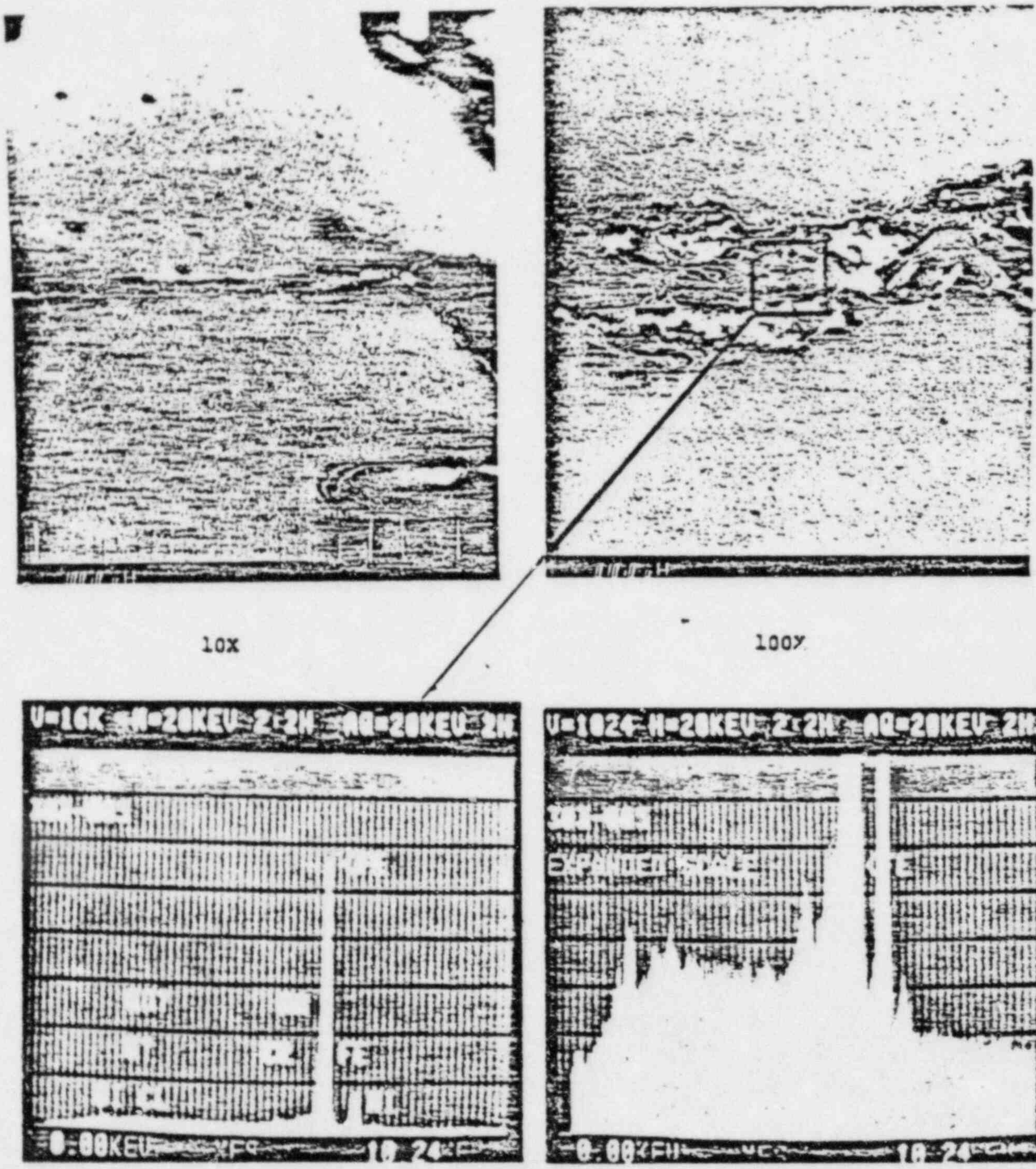
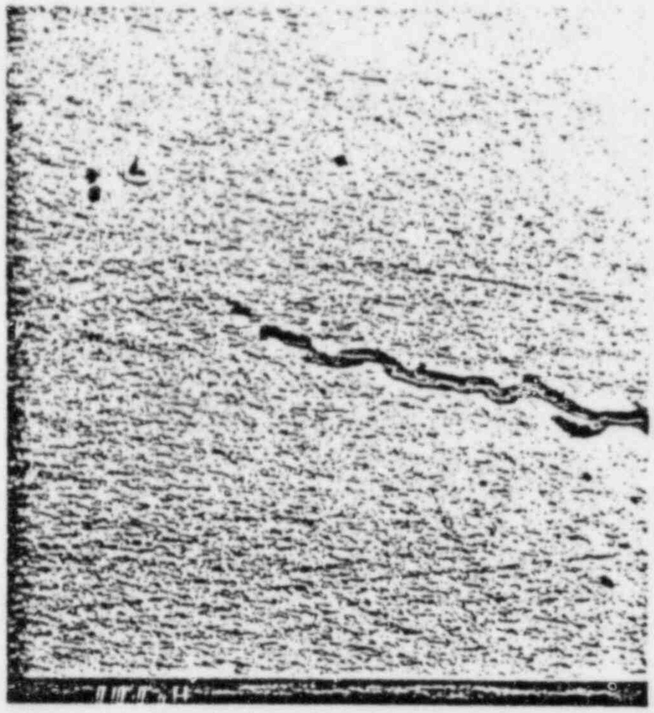
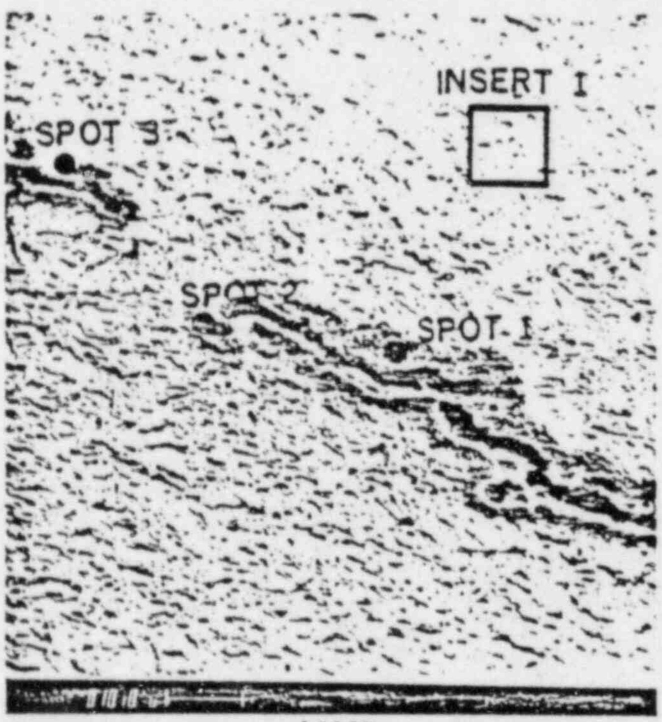


Figure 6-15. Scanning Electron Micrographs and EDS Analysis Results from a Crack in Failed Stud 11. (Negatives #53344, 53346)



100X



1000X

Figure 6-16. Scanning Electron Micrographs of the Tip of the Crack Shown in Figure 6-15 Showing Four Areas Analyzed by EDS. (Negatives #43341, 53342)

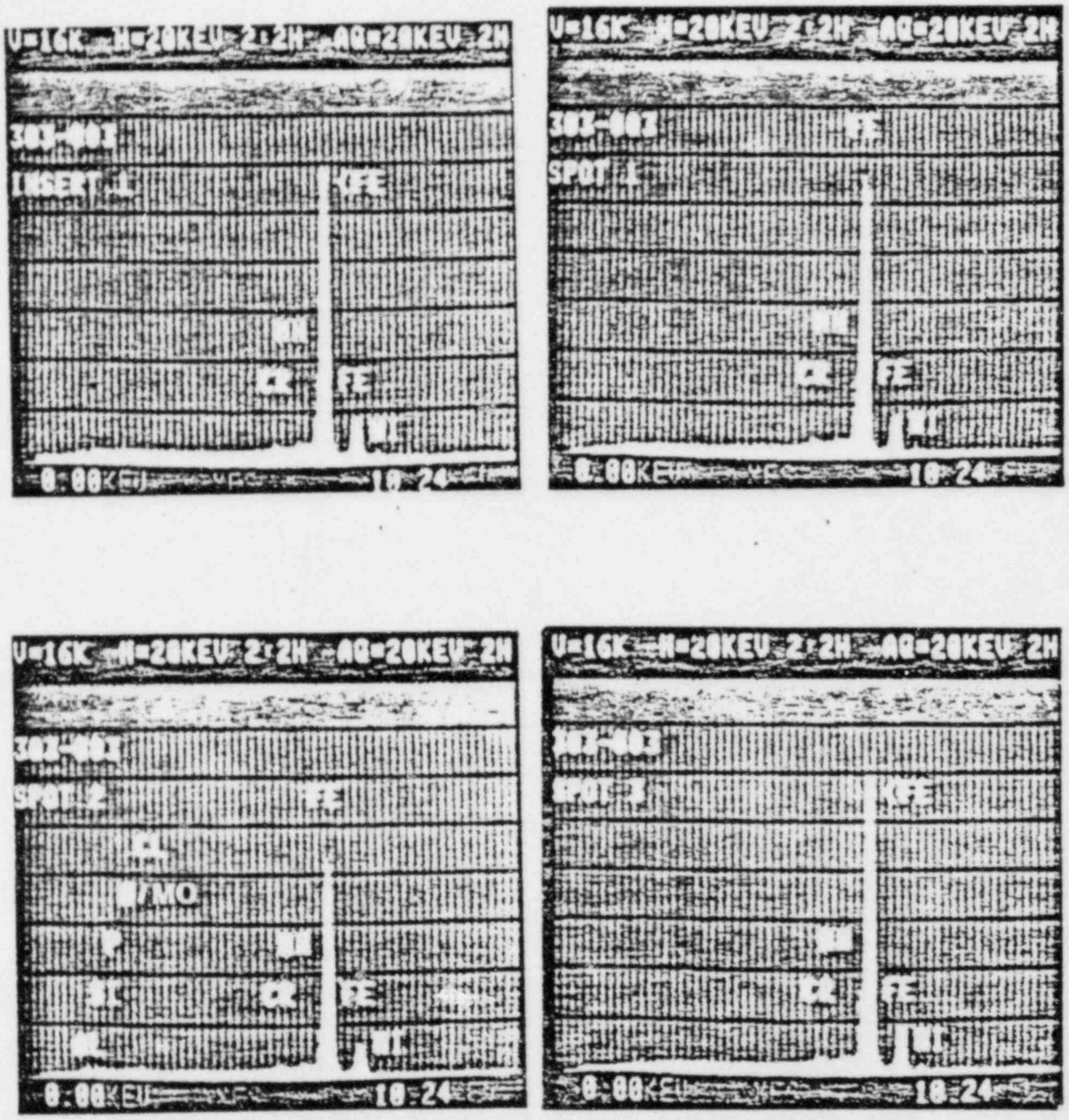
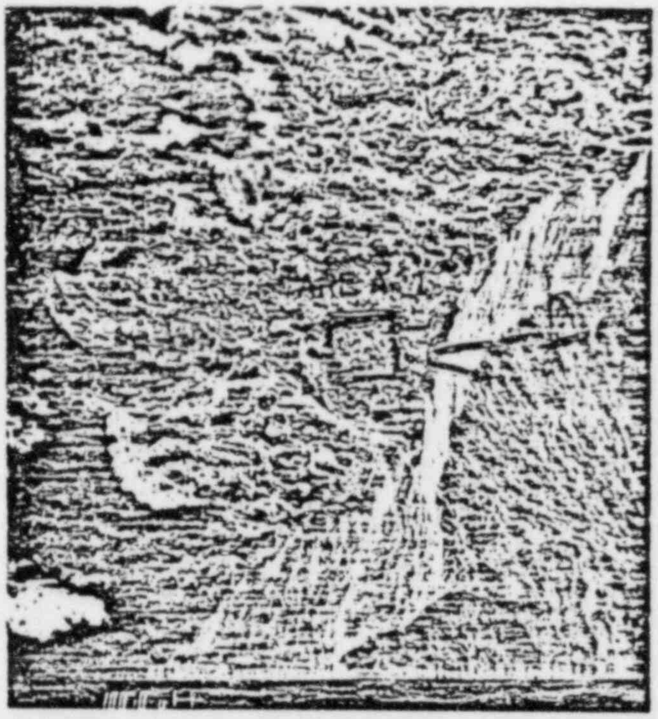


Figure 6-17. EDS Analysis Results from the Four Areas Shown in Figure 6-16.



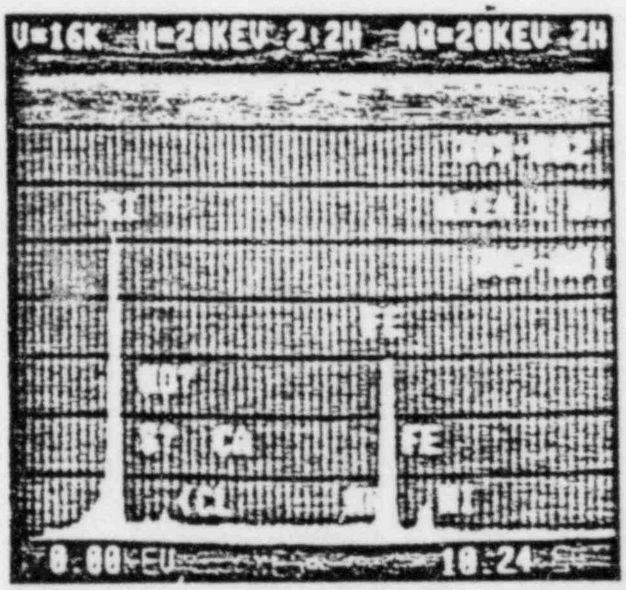
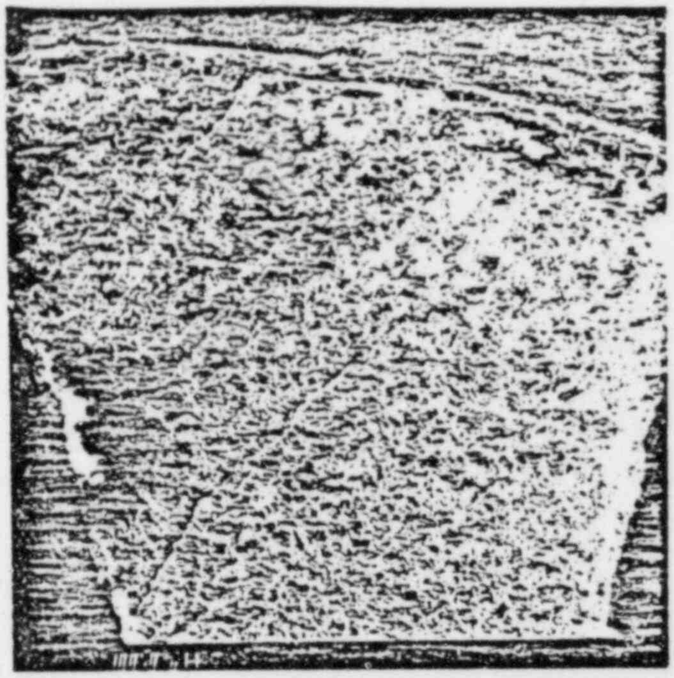


Figure 6-18. Scanning Electron Micrograph and EDS Analysis Results from the Area Shown Near the Ductile Fracture Area in Stud B. (20X)



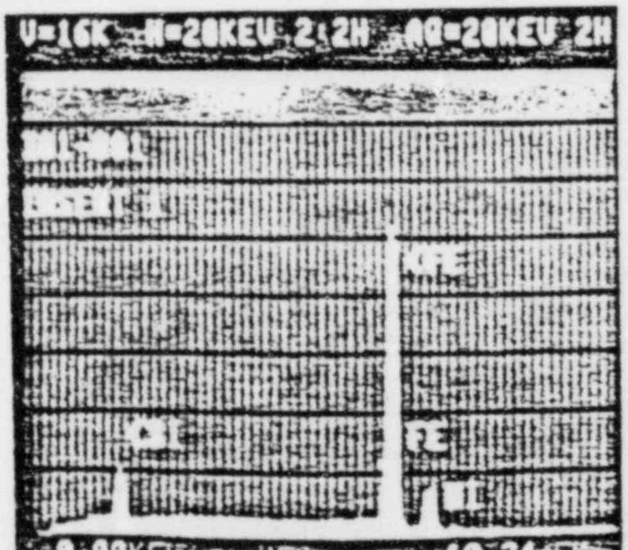


Figure 6-19. Scanning Electron Micrograph and EDS Analysis Results from the Surface of the Crack Shown in Figure 6-15. (10X, Negative #53333)

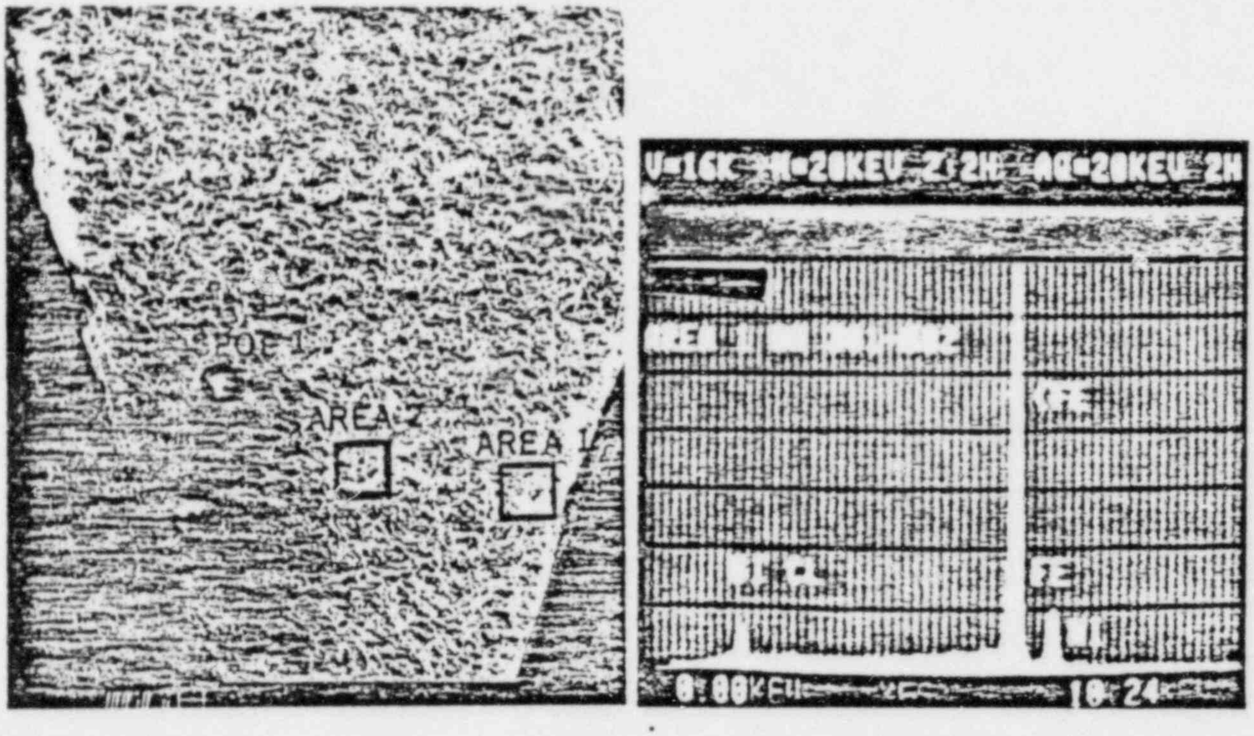


Figure 6-20. Scanning Electron Micrograph and EDS Analysis Results from Three Areas on the Crack Surface Shown in Figure 6-15.

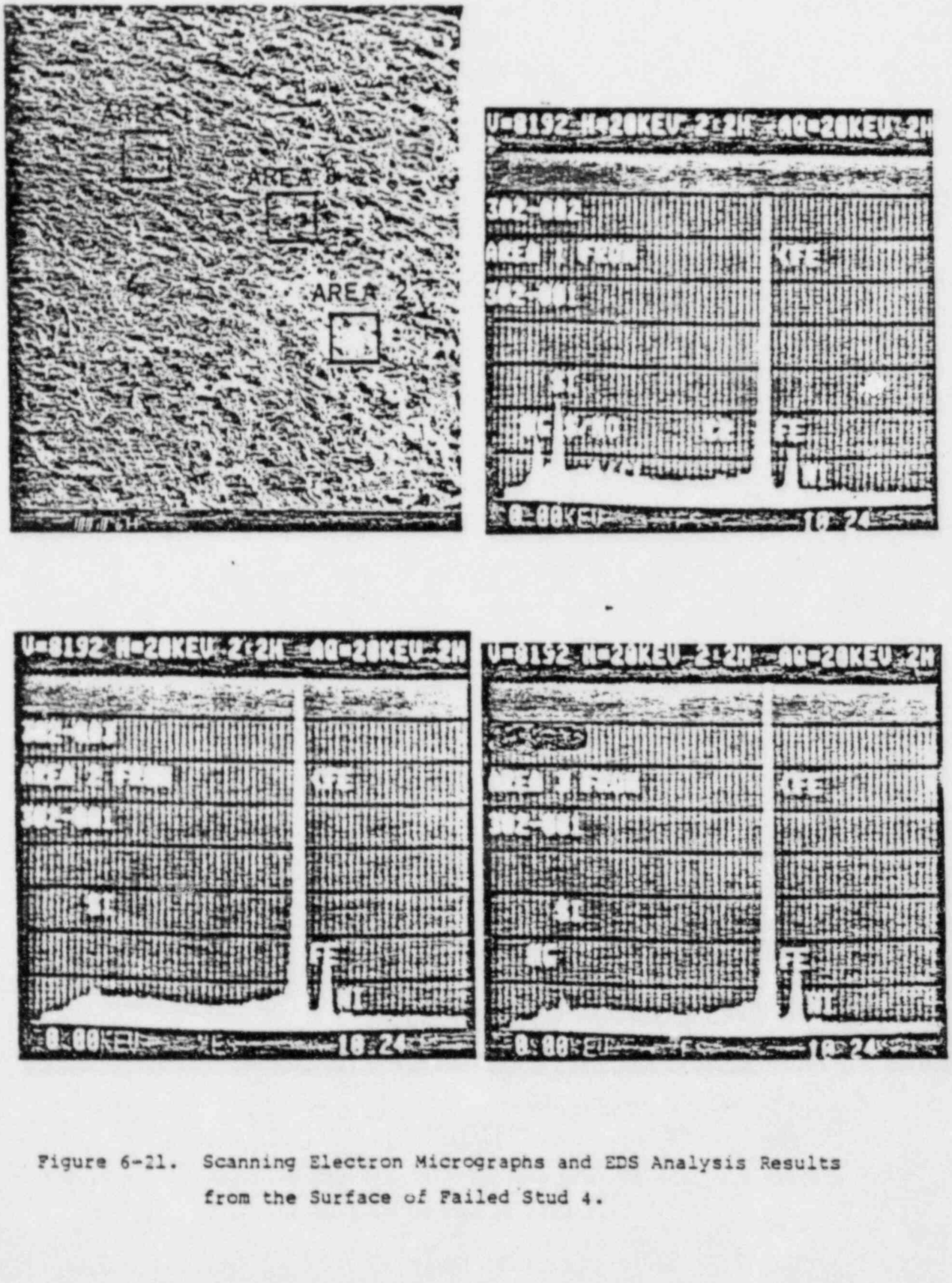


Figure 6-21. Scanning Electron Micrographs and EDS Analysis Results from the Surface of Failed Stud 4.

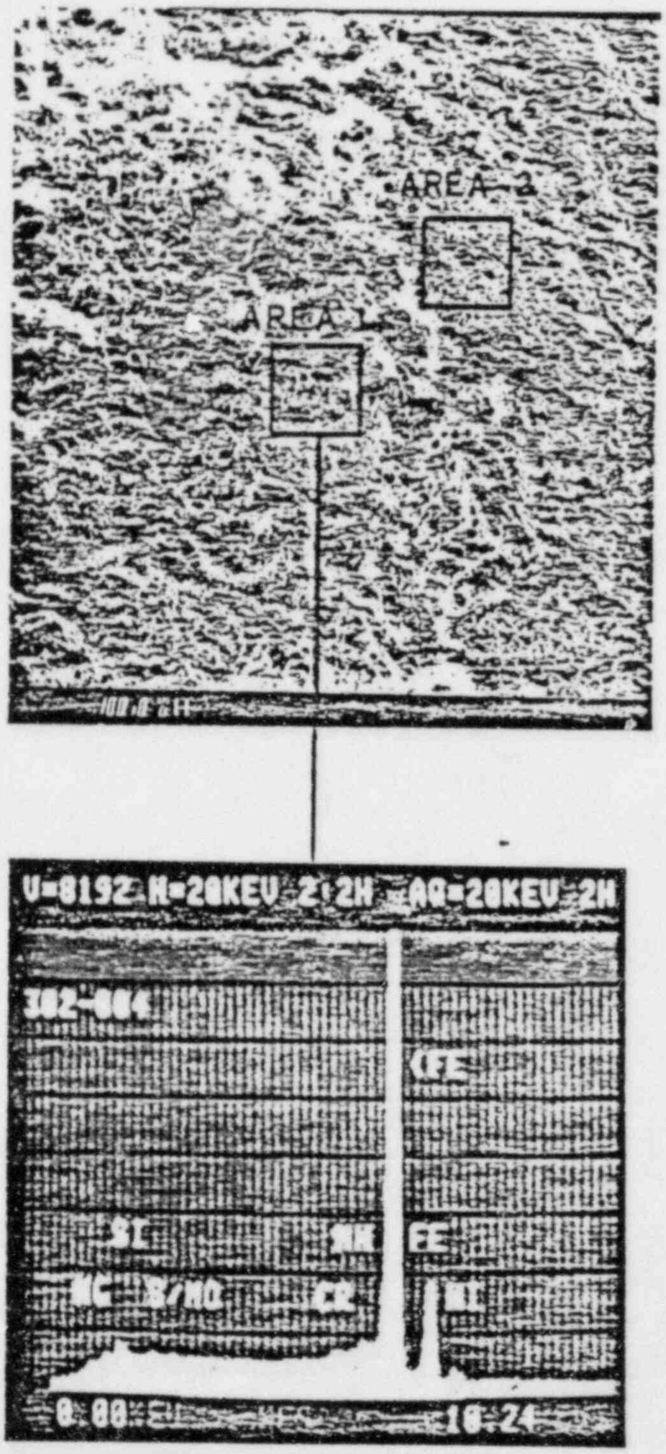
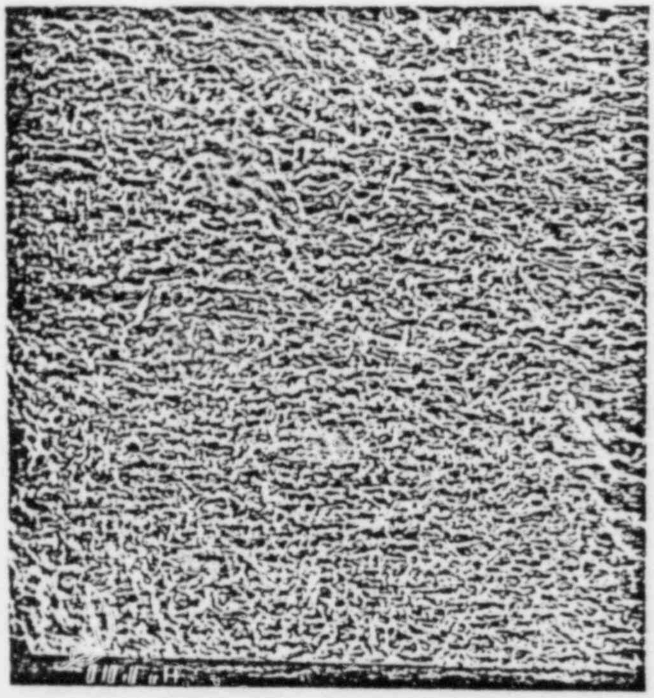


Figure 6-22. Scanning Electron Micrograph and EDS Analysis Results from the Area Shown on Failed Stud 4. (10X, Negative #53339)



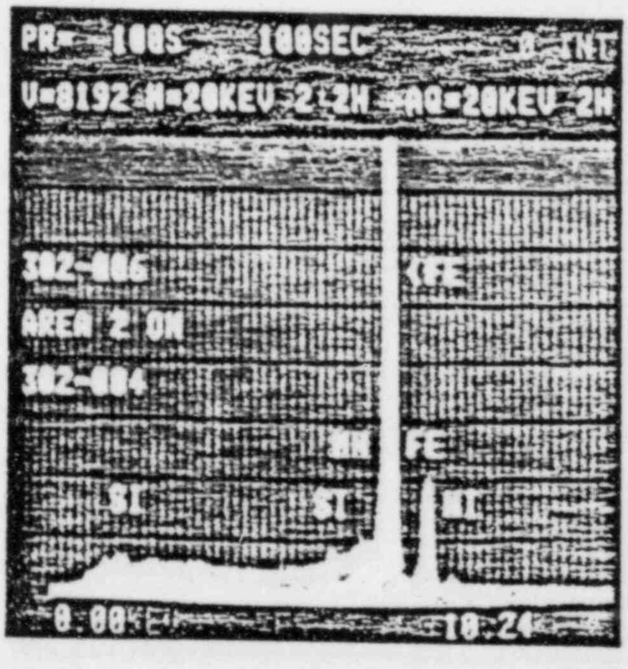
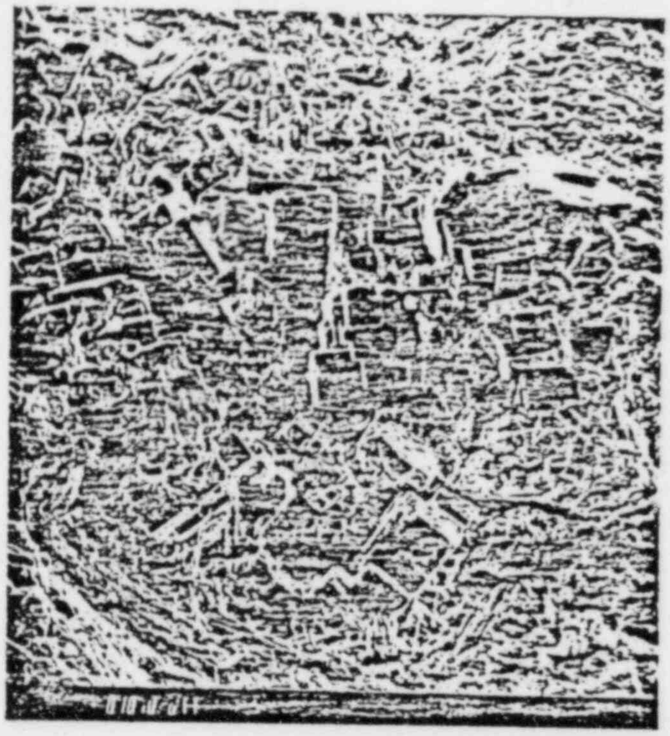


Figure 6-23. Higner Magnification SEM and EDS Analysis Results from Area 2 of Figure 6-22. (100X, Negative #53335)



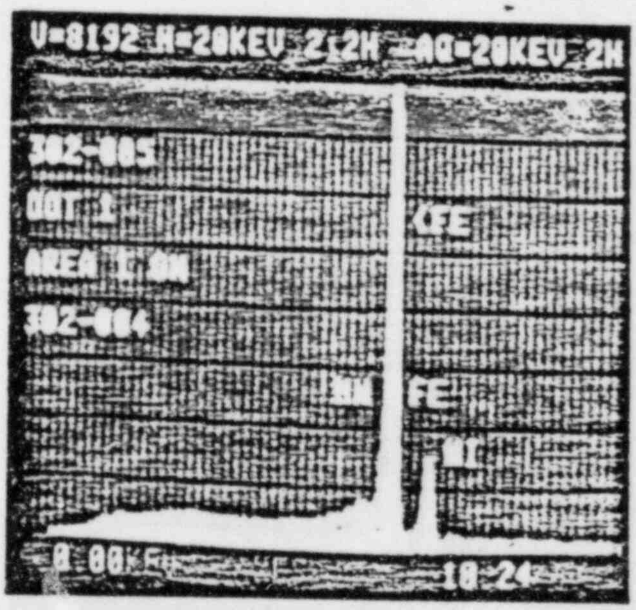


Figure 6-24. Scanning Electron Micrograph and EDS Analysis Results from the Crystalline-like Deposits on the Surface of Stud 4 (100X, Negative #53340)

7.0 HARDNESS MEASUREMENTS

Two to four Rockwell C hardness determinations were made on the shank region of each of the 19 manway cover studs. Average HRC values of these studs are listed in Table 7-1. Values range from a low of HRC 28.5 to a high of HRC 38. Conversion to Brinell hardness indicates HBN values of 282 to 365. These values are typical of SA 540 grade B24 class 3 (heat treated to a minimum specified yield strength of 130,000 psi)

Comparison of the hardness values of the fractured or cracked studs with values from the sound studs does not suggest a correlation between high hardness and cracking.

Table 7-1

HARDNESS RESULTS

Stud ID	HRC, Averages	Failed
Failed Stud 4	28.5	yes
Failed Stud 11	29	yes
A	33.5	yes
B	33.5	yes
C	34.5	yes
D	33	yes
2C8 (small end)	32	yes
2C8 (large end)	36.5	yes
2C9 (large end)	38	yes
2C9 (small end)	31.5	yes
2C10	36	no
2C11	36	no
2C12	36.5	no
2C13	34	no
2C14	30.5	no
2C15	36	no
2C16	33.5	no
2C17	*	no
2C18	36	yes
2C19 (large end)	34	yes
2C19 (small end)	32	yes
2C20	34.5	no

* Hardness not determined because of excessive pitting in shank section.

8.0 FINDINGS/CONCLUSIONS

The examinations of steam generator primary manway cover studs from Maine Yankee resulted in the folowing findings and/or conclusions:

- o Failure of the manway cover studs was the result of environmentally assisted cracking. The crack morphology (mixed mode) and the presence of numerous secondary cracks was typical for stress corrosion cracking or hydrogen embrittlement in alloy steels used for fastener applications.

- o General corrosion, such as that induced by boric acid attack, was mild and did not contribute to the failures. Other than the observed cracking, only limited pitting type corrosion occurred in either the shank or thread regions of the studs.

- o Rockwell "C" hardnesses of 19 studs tested range from 28.5 to 38 covering the range expected for SA 540 grade B24 alloy steel heat treated to a minimum yield strength of 130,000 psi. There was no apparent correlation between high hardness and occurrence of cracking.

- o Microstructures in the studs examined consisted of tempered martensite, which is typical for the materials used in the studs. No abnormalities in the microstructure were observed.

- o Qualitative analysis of lubricant samples indicated the presence of Ni, Cu, Mo and S. Ni is present in the lubricant currently used on manway cover studs at Maine Yankee. Cu, Mo and S are all used in other lubricants which may have been used in the past. Traces of Mo and/or S were detected on crack surfaces.

- o No contaminant elements known to be aggressive toward alloy steels were detected in the examination of the Furmanite sealant used on the Maine Yankee studs.

UNITED STATES
NUCLEAR REGULATORY COMMISSION
WASHINGTON, D. C. 20555

Enclosure 1

AUG 3 0 1982

MEMORANDUM FOR: Gus C. Lainas, Assistant Director for Operating Reactors
Division of Licensing

FROM: William V. Johnston, Assistant Director for Materials & Qualifications
Division of Engineering

SUBJECT: MAINE YANKEE STEAM GENERATOR MANWAY STUDS (TAC # 48059)

REFERENCES: (a) Combustion Engineering Report, "Examination of No. 2 Steam Generator Primary Manway Cover Studs from Maine Yankee" by J. F. Hall & G. C. Fisk, April 1982.

(b) Brookhaven National Laboratory Report, BNL NUREG-31742 "Examination of Failed Studs From No. 2 Steam Generator of the Maine Yankee Nuclear Power Station" by C. Czajkowski, July 1982.

(c) IE Bulletin 82-02, dtd June 2, 1982.

On March 3, 1982, the Maine Yankee steam generator number two cold leg manway cover was removed for tube inspection. During removal, five studs failed. (A sixth stud was broken a week later during cleaning.) Two of the broken studs and 18 others were sent to Combustion Engineering for failure analysis. At the request of the NRC staff, three other studs were sent to Brookhaven National Laboratory for independent analysis. Of these three, one was broken, one was cracked, and the third was intact.

At a meeting with the licensee prior to any of the results of the failure analyses, much was made of the use of a sealant material, Furmanite, in the period from October 1981 to failure in an effort to stop the joint from leaking. Prior to the installation of the Furmanite sealant, an attempt to stop the leak was made by increasing the installation torque from 900 ft-lbs to the hydrotest value of 1100 ft-lbs. Upon disassembly, it was found that the cause of the leak was that the channel head clad had not been machined to permit the proper fit up of the stainless steel gasket retainer.

Contact: C. D. Sellers
X-28049

8209210239 XA

The conclusions of the CE and Brookhaven reports are essentially similar. Both state that failure was environmentally assisted. The Brookhaven report states "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."

There appears to be contradictory information related to the ease of removal of the studs from the steam generator. In the meeting with the licensee on this subject in March they stated that all studs were removed without difficulty. This is in conflict with these observations:

1. One of the studs delivered to Brookhaven had a nut that had to be cut off.

2. Another stud also exhibited damage to the wrenching hex and the threads, indicating something less than ease of removal.

3. A Region I inspector, H. Gray, in a note to D. Sellers dated 5/21/82, stated that one broken stud exhibited evidence of shear failure indicating that it had been twisted off. He further stated verbally that he had witnessed other studs in other steam generator manways at Maine Yankee that had failed during disassembly.

Although these observations have no bearing on the stated cause of failure, they are indicative of other problems with thread lubricants.

In summary, the CE and BNL reports agree that the stud failures were environmentally assisted, and that the thread lubricants were the major source of deleterious contamination. The role of the Furmanite sealant was considered to be primarily to seal water in contact with the studs, thus creating an "autoclave" type of environment. The use of sealants in circumstances that can create such conditions should be avoided. Responses to Bulletin 82-02 should be evaluated to assess the frequency of occurrences of this situation.

William V. Johnston

William V. Johnston, Assistant Director
Materials & Qualifications Engineering
Division of Engineering

cc: See Page 3

AUG 30 1982

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C. Nelson
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C. D. Sellers

NUREG/CR-2993
BNL-NUREG-51594

DRAFT

EXAMINATION OF FAILED STUDS FROM NO. 2 STEAM GENERATOR AT THE MAINE YANKEE NUCLEAR POWER STATION

Carl Czajkowski

July 1982

Brookhaven National Laboratory
Department of Nuclear Energy
Corrosion Science Group
Upton, New York 11973

Prepared for the United States Nuclear Regulatory Commission
Division of Engineering, Office of Nuclear Reactor Regulation
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ABSTRACT

Three studs removed from service on the primary manway cover from steam generator #2 of the Main 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 lubricants 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 PHYSICAL PROPERTIES OF
ASTM A540-B24 STEEL

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

FURMANITE
INCORPORATED

579 VIKING DRIVE • VIRGINIA BEACH, VIRGINIA 23452 • PHONE (804) 480-8182 • 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 [signature]

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INCORPORATED

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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	[illegible]
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 [signature]

FURMANITE
INCORPORATED

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TABLE 4

TYPICAL CERTIFICATION FOR SEA' ANT USED (LOT 702)

MATERIAL CERTIFICATION

December 4, 1981

Material: ~~Furmanite Nuclear F-700-N, Lot 4702~~

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~~	~~[illegible]~~	~~[illegible]~~
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	[illegible].5 ppm	7.5 ppm	[illegible].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:

17 Aug 1981

Notary Public

PUBLIC
COOK COUNTY

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

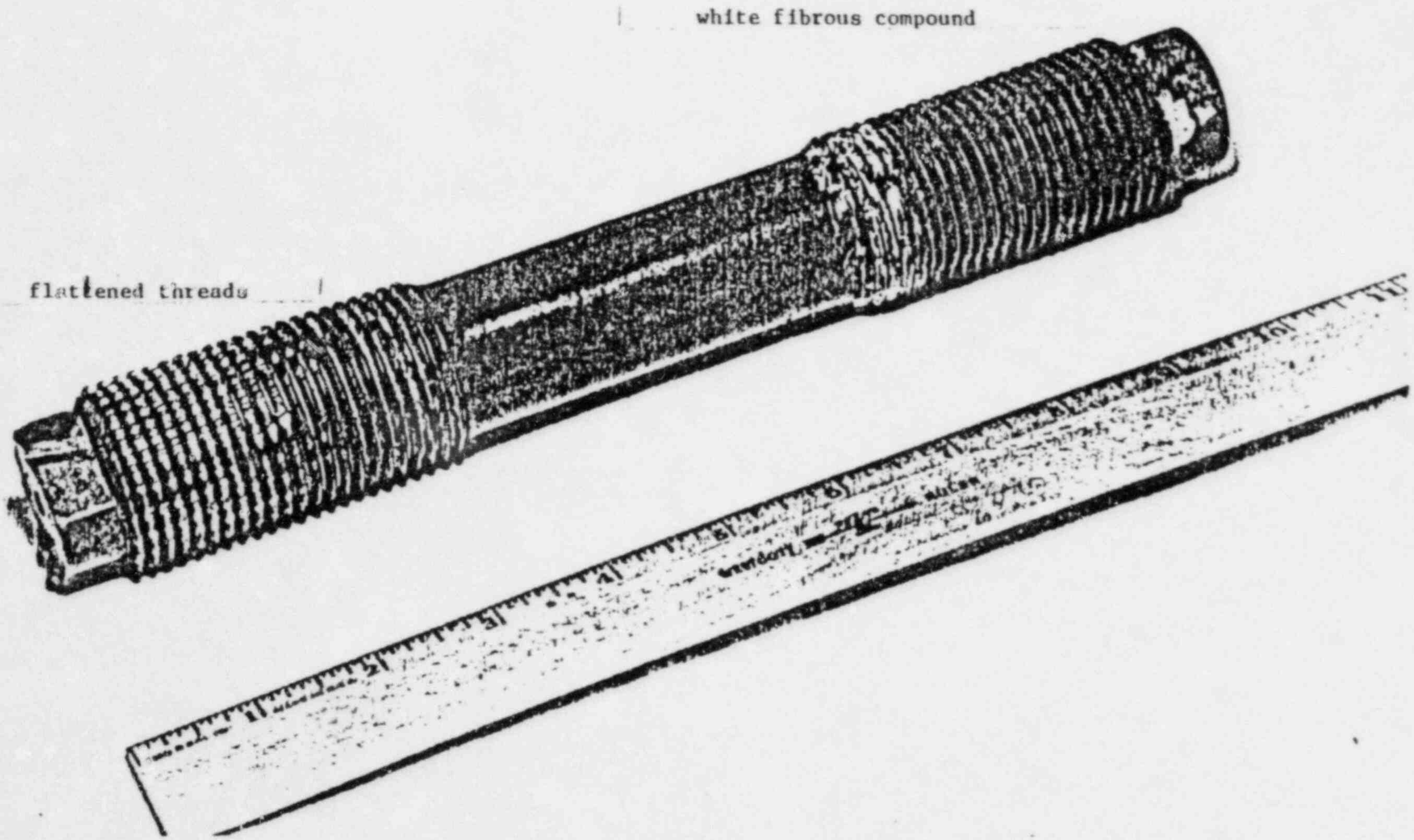


Figure 1. Photograph of "as received" stud from Maine Yankee. (Bolt had the following numbers stamped on the ends - 2C6, #46500, 6410).

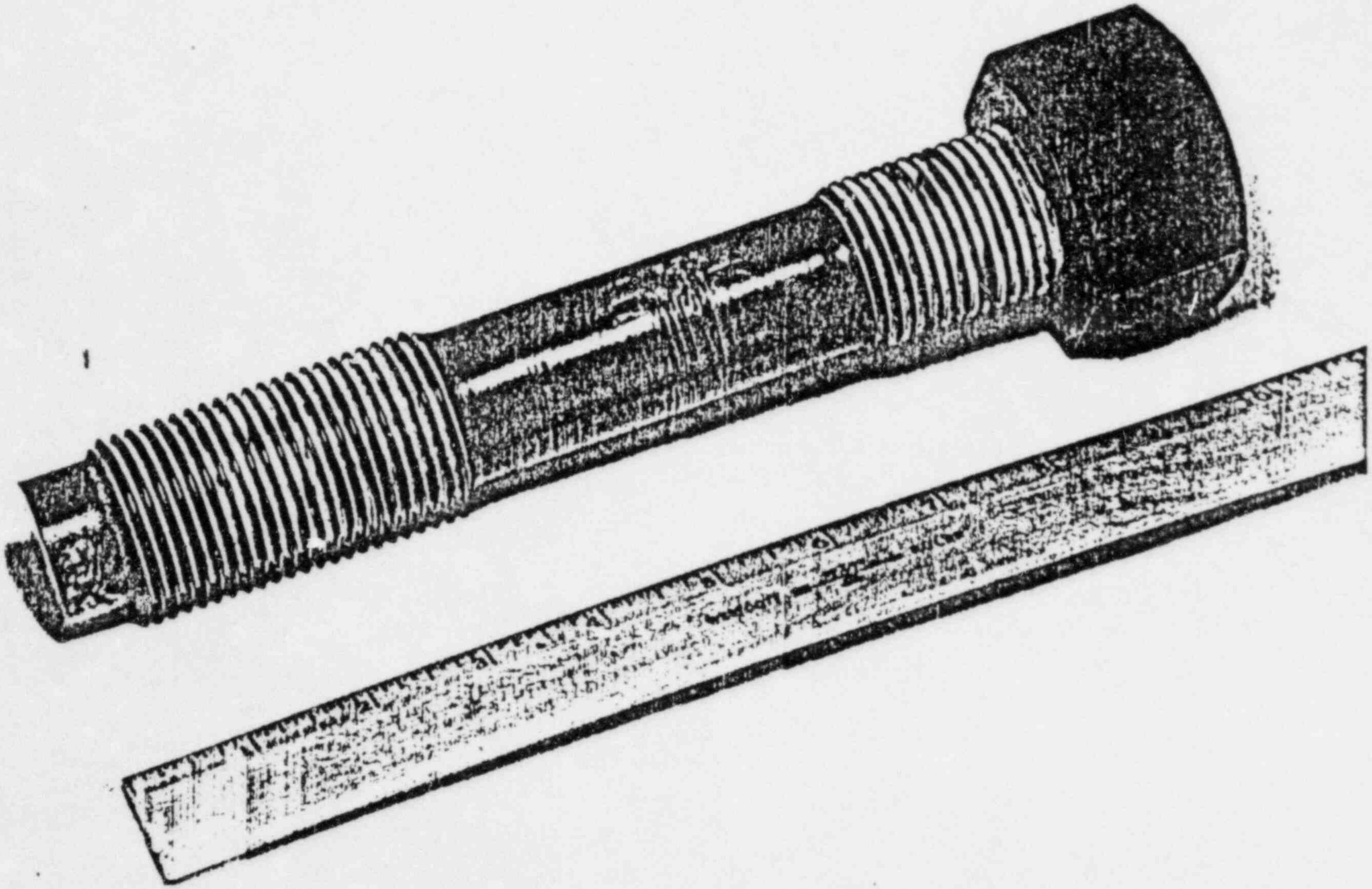


Figure 2. Photograph of second stud received from Maine Yankee. Photo depicts remnants of Dye Penetrant testing. (Bolt had the following numbers stamped on the ends (2C20, #64034)).

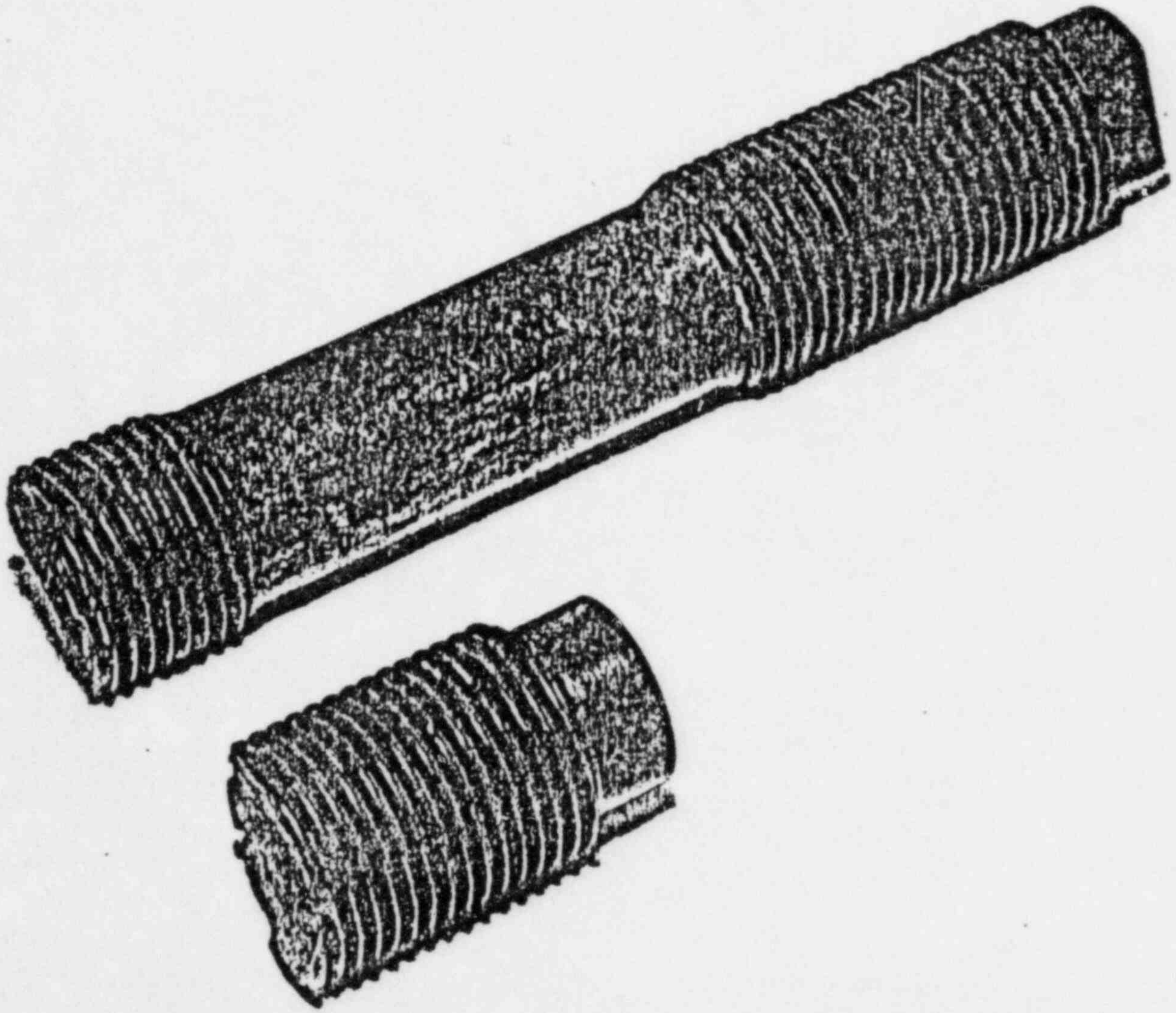


Figure 3. Photograph of "cracked" stud from Maine Yankee (Bolt had the following numbers stamped on the ends-2C8, 6418, #64034).

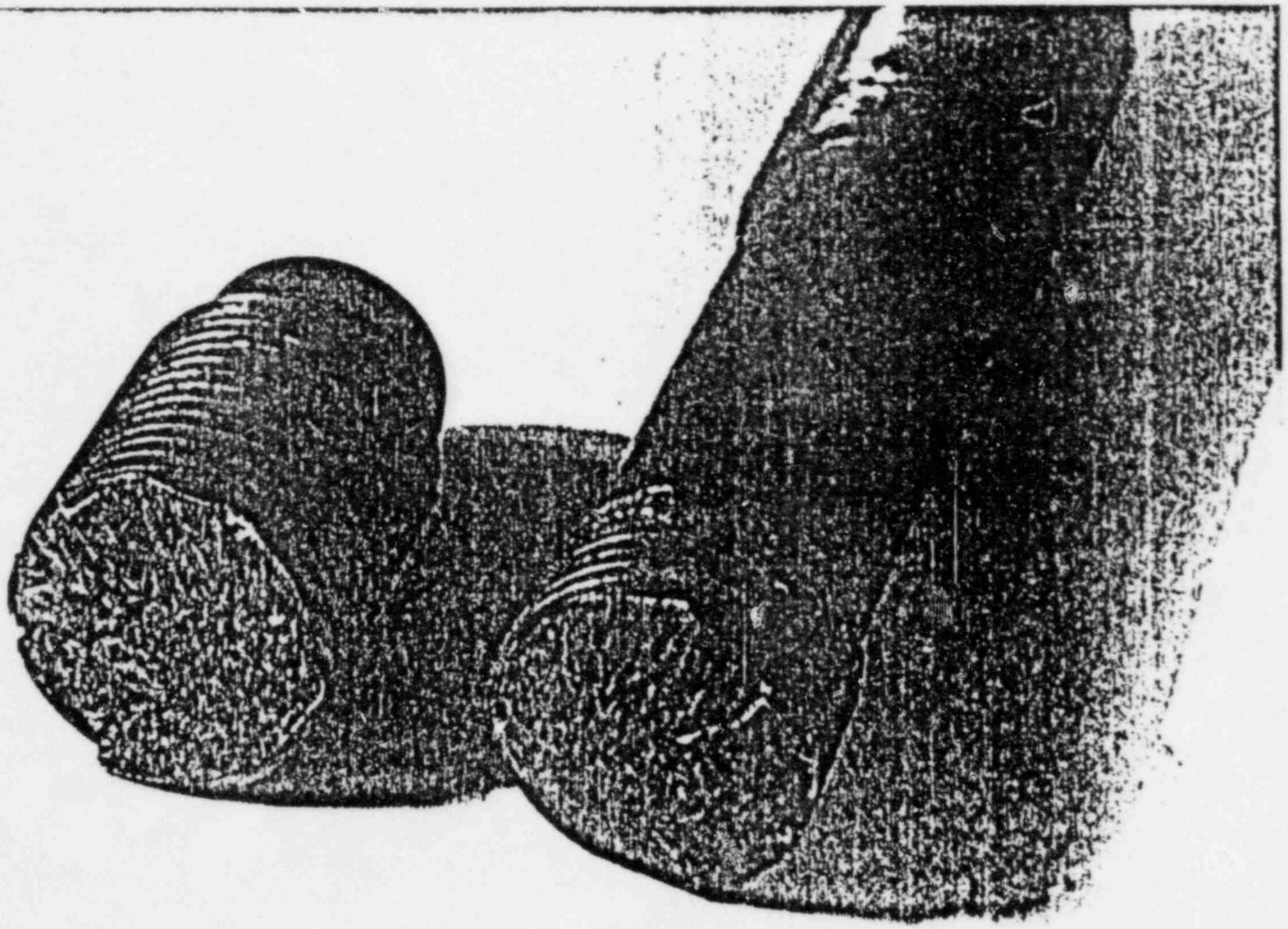


Figure 4. Frontal photograph of the fracture faces of the cracked bolt.

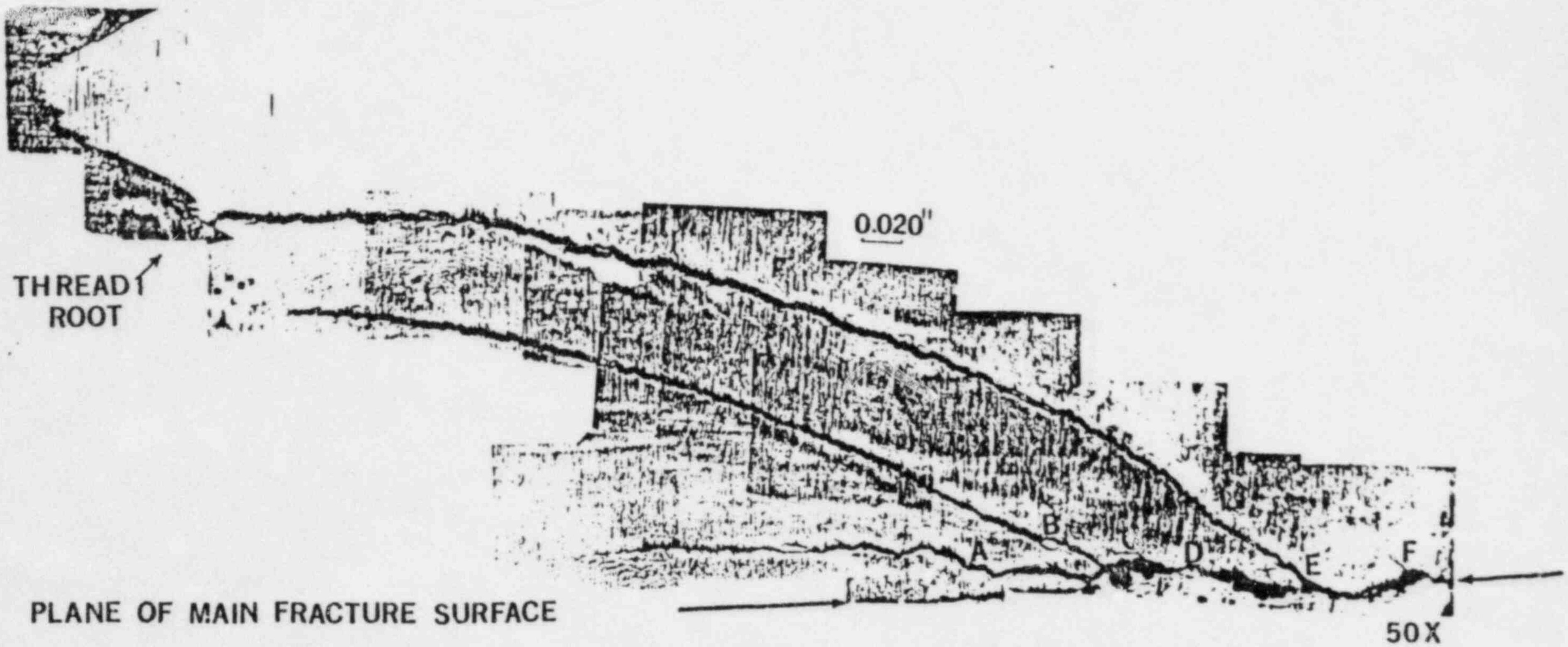
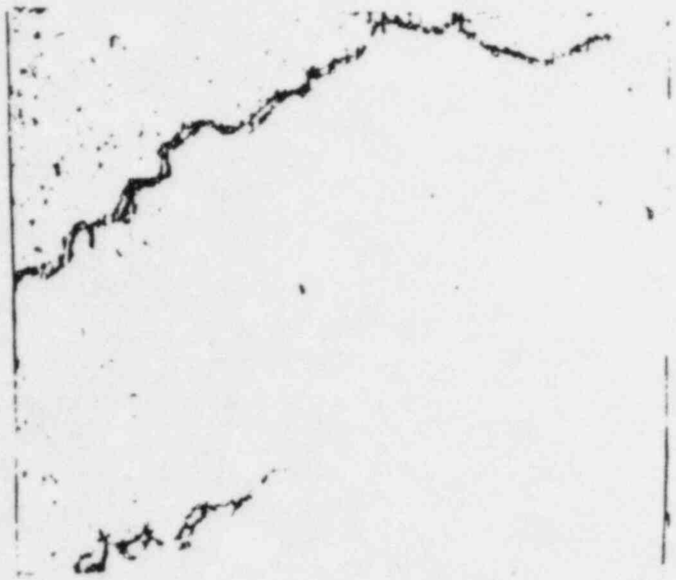
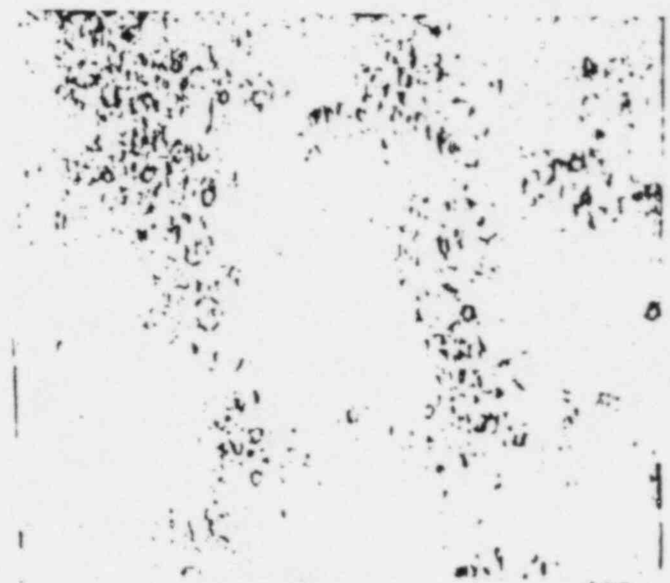


Figure 5. Optical photomicrograph of bolt section depicting extent of secondary cracking (areas A-F). Note fern like branching on larger secondary cracks.



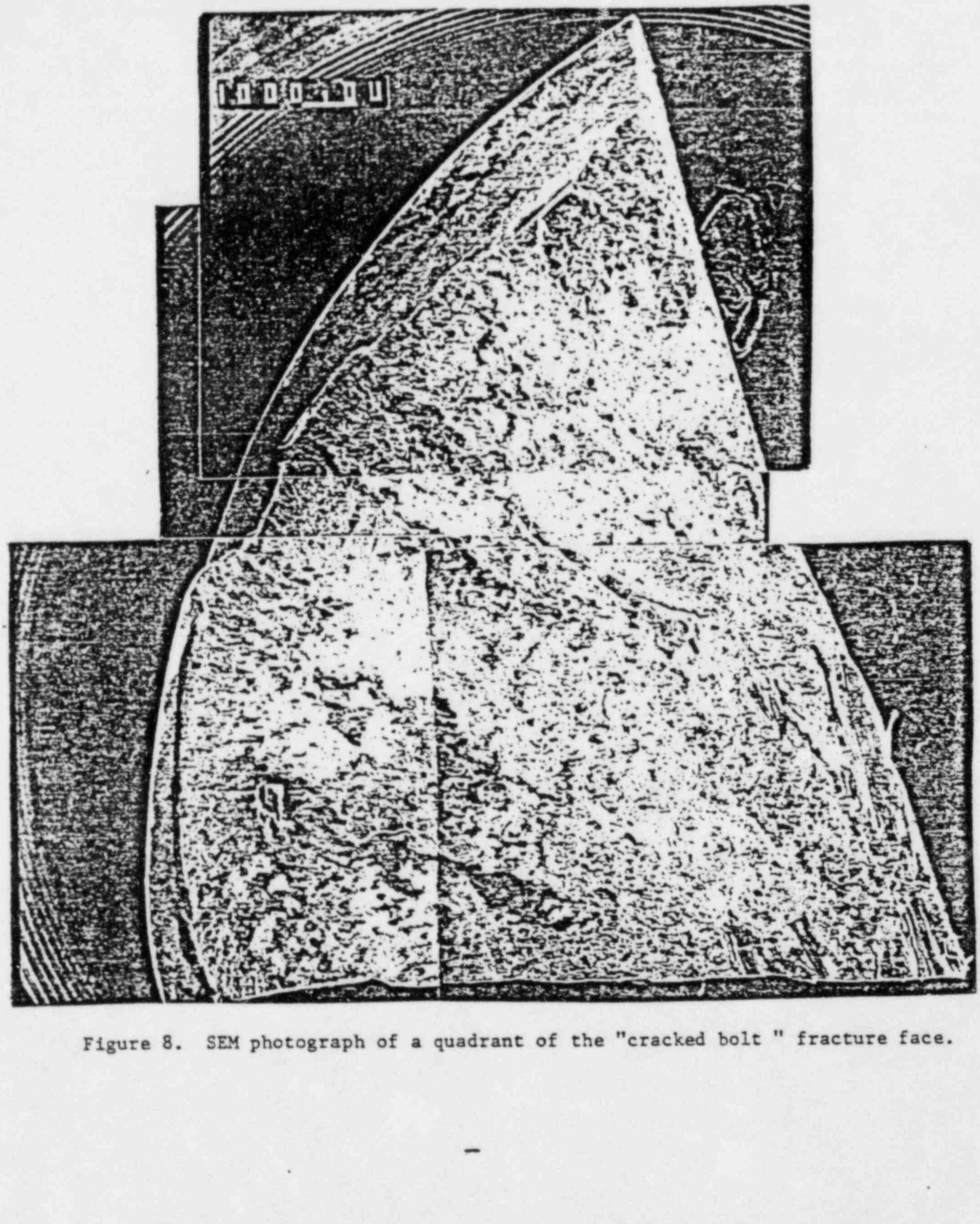
500x

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



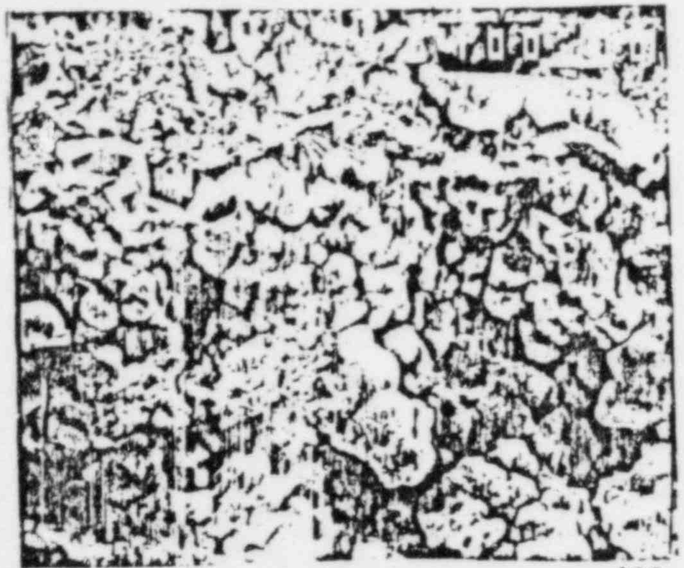
400x

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



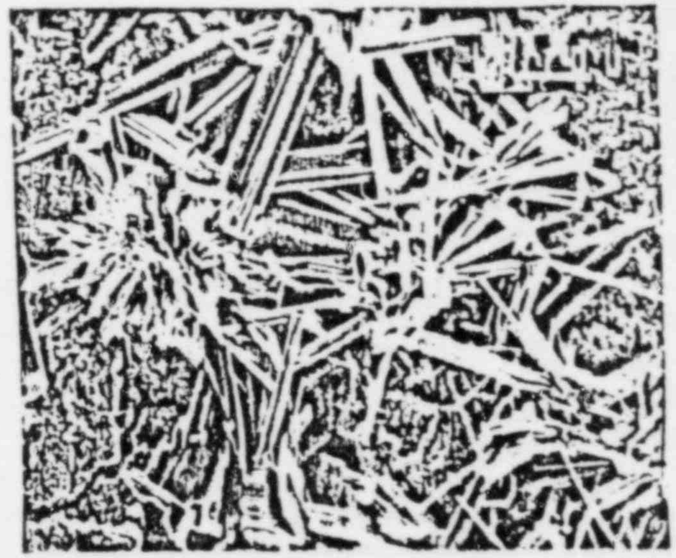
10X

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



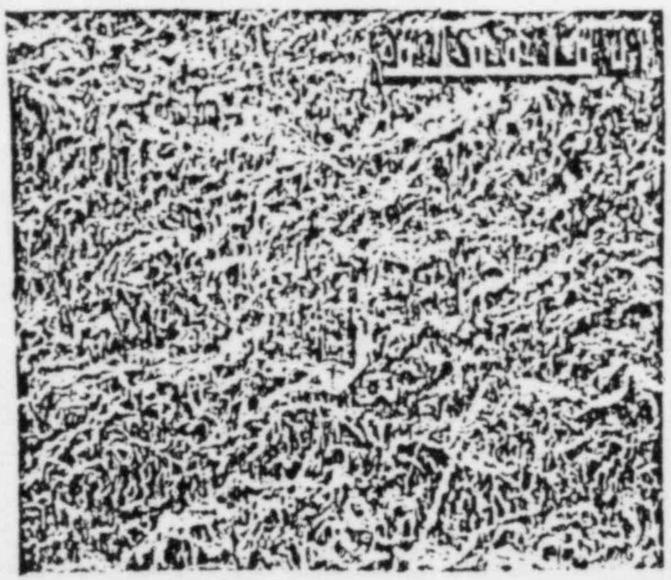
100x

Figure 9. SEM photograph of "nodular" oxide.



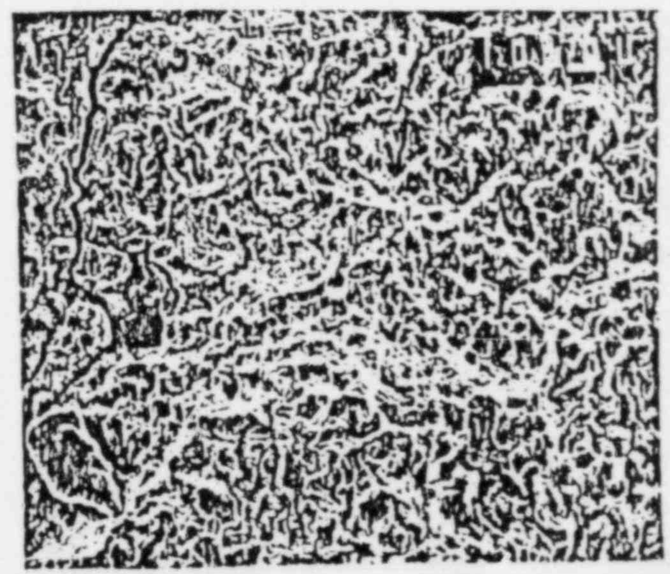
1000x

Figure 10. SEM photograph of "acicular" oxide.



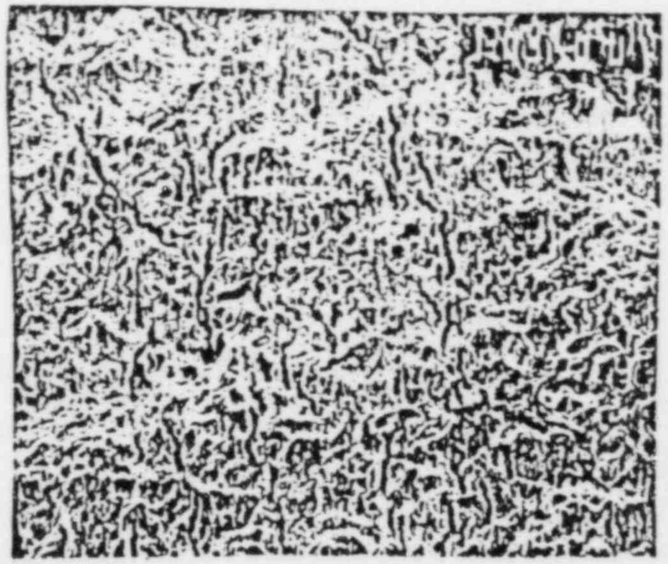
100x

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



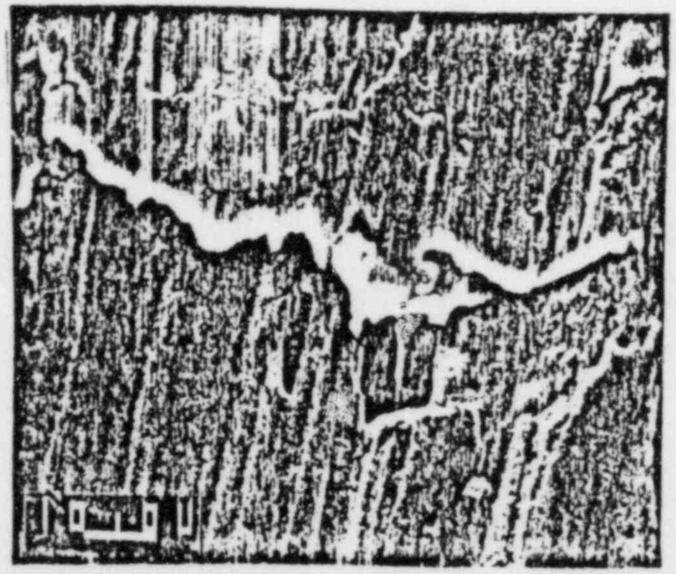
500x

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.

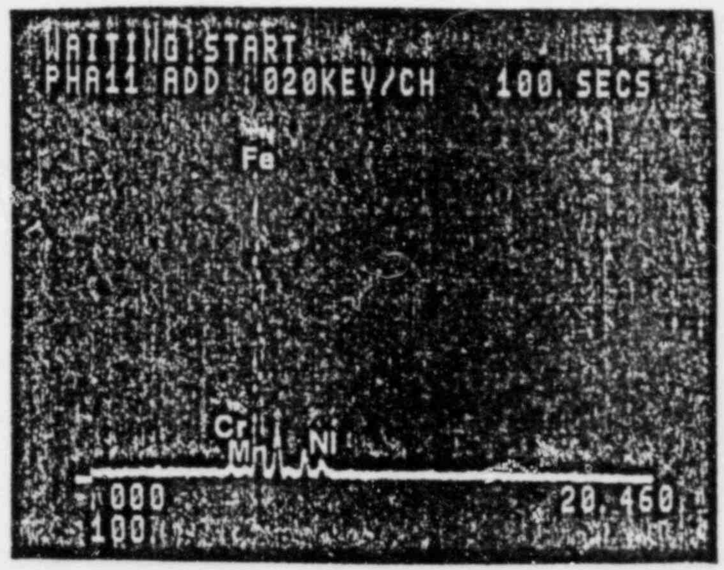
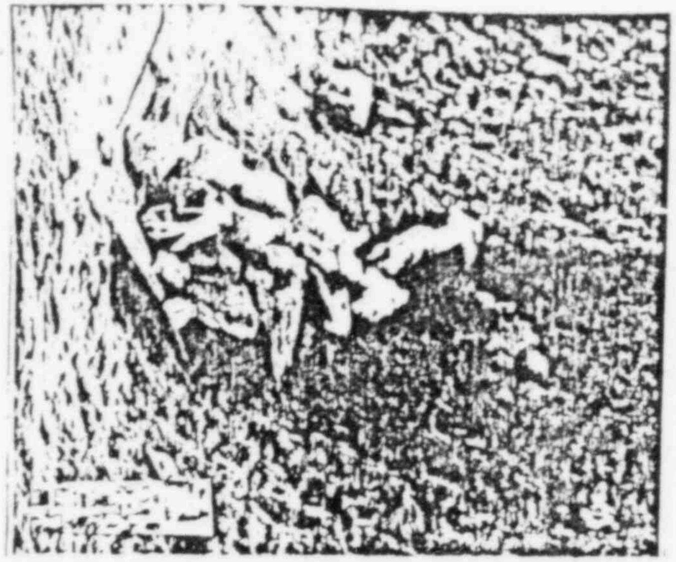


Figure 15. EDS scan of base material for constituents.



500x

Figure 16. SEM photo of fibrous particulate.

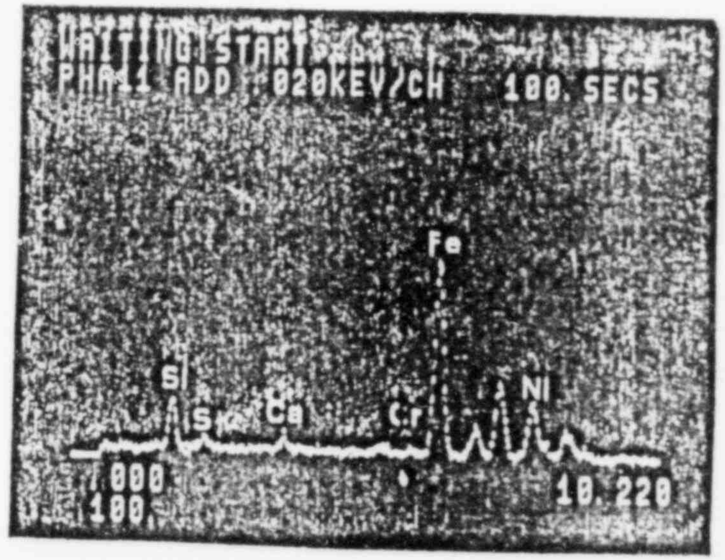
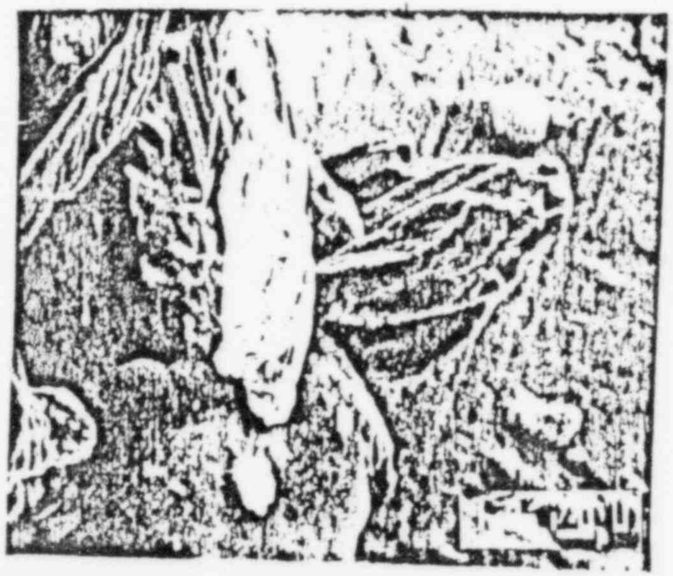


Figure 17. EDS scan of fibrous particle.



500x

Figure 18. SEM photo of second fibrous particle.

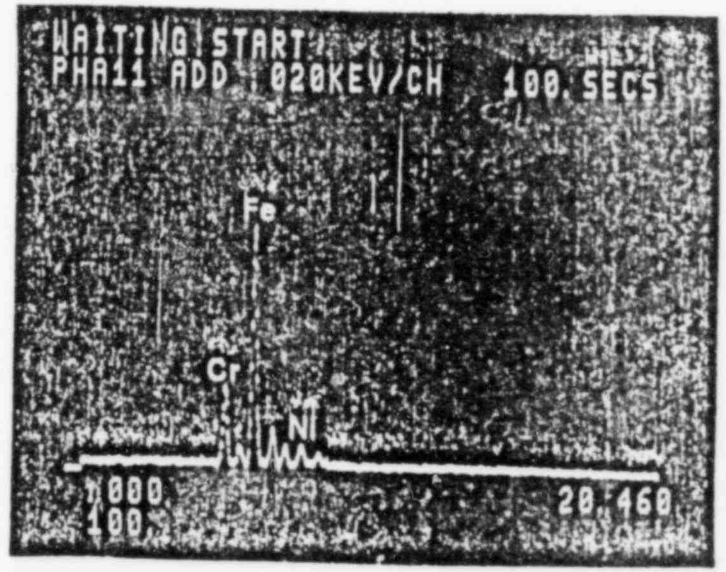
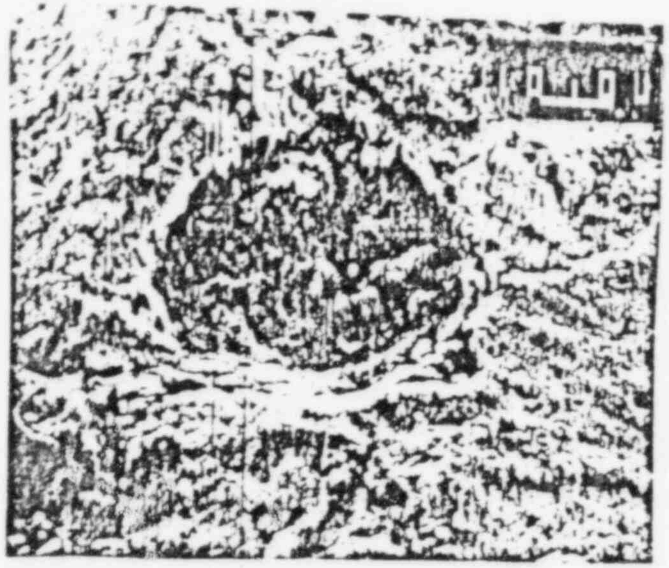


Figure 19. Constituents of second fibrous particle scanned by EDS.



1000x

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

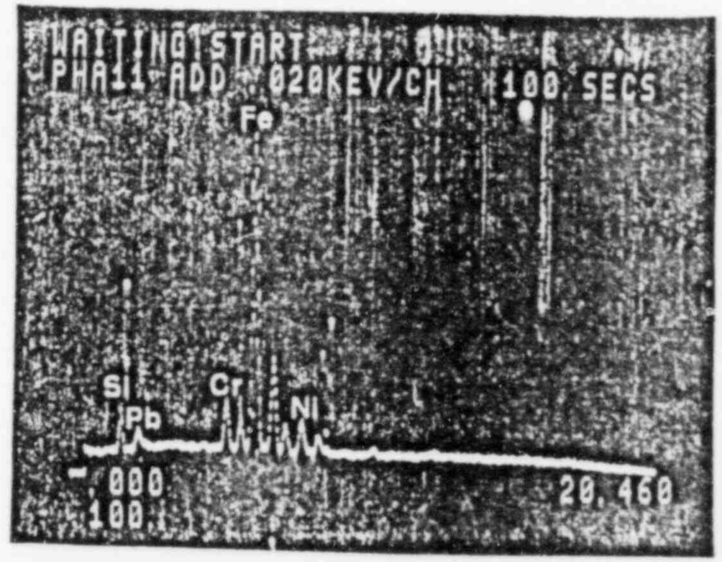
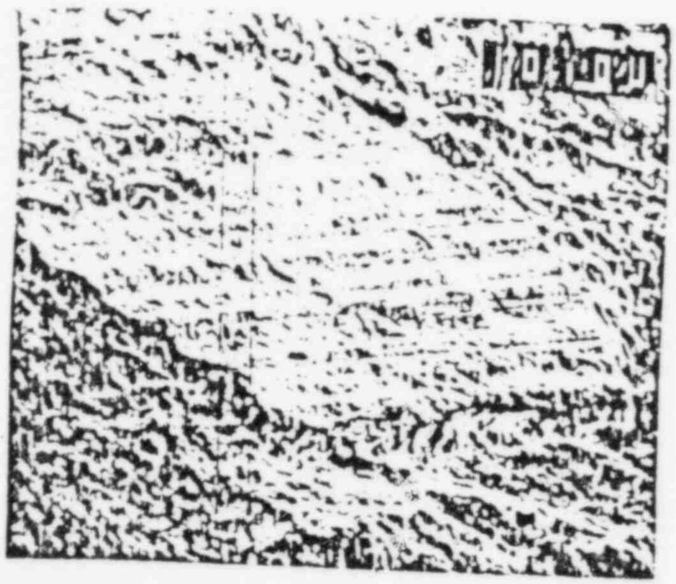


Figure 21. EDS scan of pit for chemical constituents.



500x

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

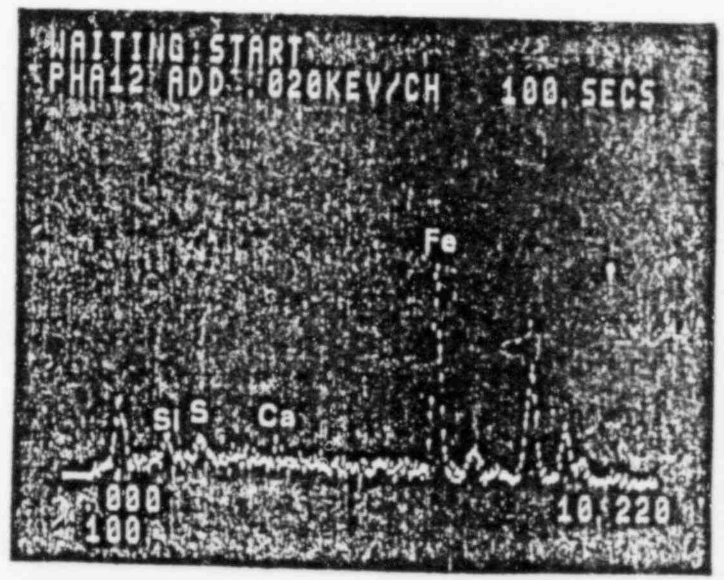


Figure 23. EDS scan of smeared material area.

1000x

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

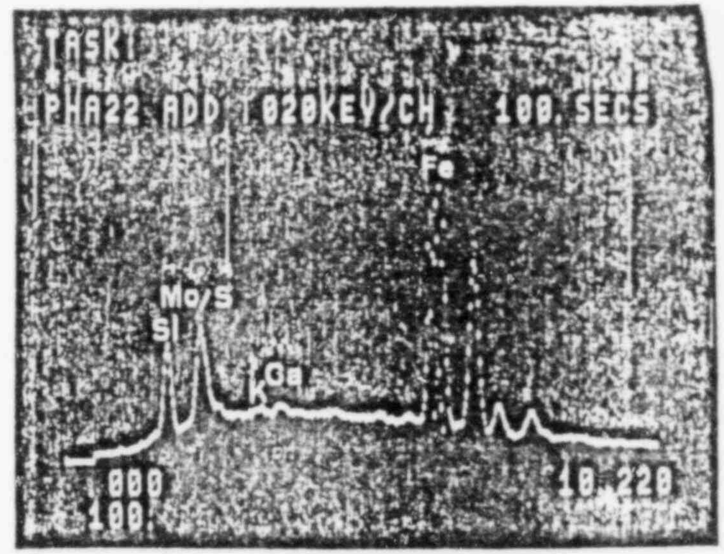


Figure 25. EDS scan of "smeared" area in Figure 25.

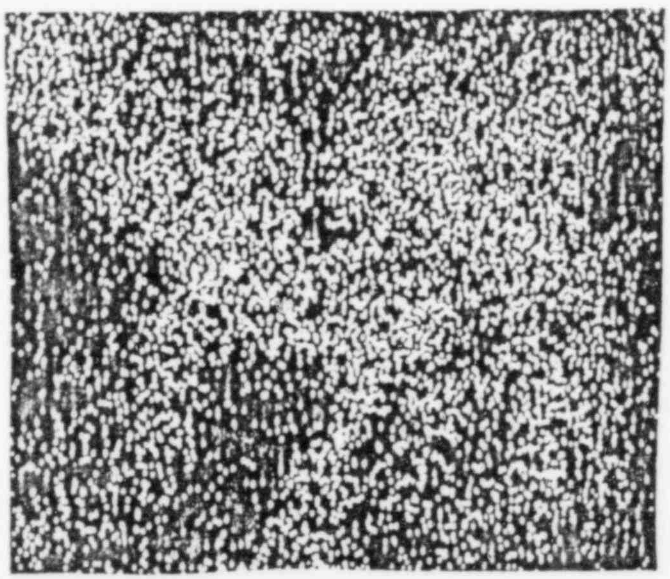


Figure 26. WDS scan of "smeared" area for molybdenum.

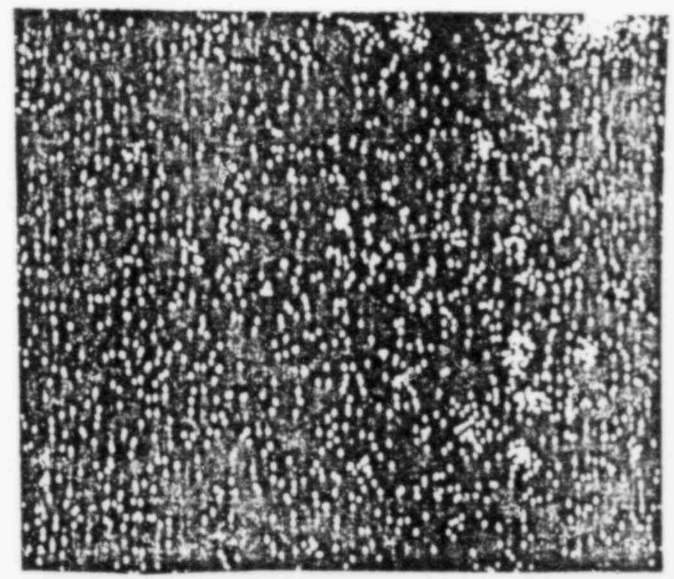


Figure 27. WDS scan of smeared area for sulfur.

NUREG/CR-2467
UCRL-53035

Lower-Bound K_{Iscc} Values for Bolting Materials - A Literature Study

Alfred Goldberg and Mary C. Juhas

Feb '82

Prepared for
U.S. Nuclear Regulatory Commission

Lawrence Livermore National Laboratory

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ACKNOWLEDGMENTS

We wish to acknowledge the support of the U.S. NRC in performing this study. Special thanks are due to R. R. Vandervoort of LLNL for reviewing the original manuscript and to C. D. Sellers of the U.S. NRC for his patience and for his valuable comments.

ABSTRACT

An extensive literature survey on stress-corrosion cracking of a variety of steels was made in response to the need by the U.S. Nuclear Regulatory Commission (NRC) to establish lower-bound K_{Iscc} values for bolting type materials. Materials evaluated include the heat-treatable plain-carbon and Cr-Mo and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 precipitation-hardening stainless steels, and the 18Ni-maraging steels. An exhaustive survey was made for water, aqueous chloride, and aqueous sulfide environments. We also report limited data on H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric environments. The data are presented in the form of K_{Iscc} versus yield strength. The proposed NRC lower-bound K_{Iscc} curve for the low-alloy steels is consistent with the data reported for the various aqueous environments, but excluding the sulfides. The corresponding lower-bound curve based on reported data for 18Ni-maraging steels falls below that proposed by the NRC, especially at the high strength levels. The lower-bound curves for the precipitation-hardening stainless steels fall below the lower-bound curve for the maraging steels at the lower strength levels, but they merge at a yield strength of approximately 220 ksi. Reference is also made to crack-growth rate (CGR) and time to failure (t_f). These two parameters frequently were found to vary differently from the corresponding K_{Iscc} values when examined as a function of material or environmental variables. The influence of various material and environmental factors on K_{Iscc}, CGR, and t_f are discussed.

EXECUTIVE SUMMARY

The U.S. Nuclear Regulatory Commission (NRC) staff, in charge of Generic Activity A-12, is establishing acceptance criteria for the structural integrity of support members for a number of nuclear-reactor plant components. Included in this task is the evaluation of high-strength bolting materials using a fracture mechanics approach. Because of a history of bolting failures by stress corrosion cracking (SCC) reliable lower-bound K_{Iscc} (threshold stress intensity for SCC) values had to be established. To obtain the necessary data we made a literature survey of SCC of bolting-type materials.

Materials evaluated include the heat-treatable plain-carbon and Cr-Mo, and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 stainless steels, and the 18Ni-maraging steels. The environments for which an exhaustive study was made are water, aqueous chlorides, aqueous sulfides, and to a limited extent are H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric. The data are presented in the form of K_{Iscc} versus σ_y (yield strength).

Considerable scatter in the plotted data is evident for virtually all the alloys examined. This is attributed to a combination of factors possibly involving variations in melting practice, heat treatment, microstructure, and composition. Differences in testing procedures, especially exposure periods, may also have contributed to such scatter. Little or no difference exists in K_{Iscc} between distilled H_2O and various aqueous chlorides (primarily NaCl); the K_{Iscc} is essentially constant over a pH range from 2 to 9. The pH as well as large differences in H_2S content will affect the K_{Iscc} values in aqueous sulfides. The effects of material variables and environmental variables on K_{Iscc}, crack-growth rate (CGR) and time to failure (t_f) are discussed. The effects of material variables are often controversial, while the effects of environmental variables are relatively self-consistent. A number of examples are presented where some variable may have little or no effect on K_{Iscc} but may result in a large increase in CGR and decrease in t_f. We would therefore caution the use of K_{Iscc} exclusive

to CGR and t_f; for example, K_{Iscc} may be based on tests with insufficient exposure time; the material may have been improperly heat treated; stray voltages may result in polarization potentials; transients may result in stress excursions developing stress intensities exceeding K_{Iscc}.

The lower-bound curves for water and the various aqueous chloride environments exhibit a sharp drop in K_{Iscc} with an increase in σ_y. For the low-alloy steels above about 190 ksi the lower-bound values of K_{Iscc} become σ_y-insensitive at about 10 ksi·in.$^{1/2}$. For the highly alloyed steels the σ_y at which K_{Iscc} becomes insensitive is increased to higher values. For maraging steels this occurs at about 240 ksi σ_y with the lower-bound K_{Iscc} close to 7 ksi·in.$^{1/2}$.

The lower-bound K_{Iscc} curve proposed by the NRC staff for low-alloy steels is consistent with the data obtained for water and various aqueous chloride environments. That proposed by the NRC for the maraging steel is non-conservative, especially at the high σ_y levels; at 240 ksi it is higher by about 25 ksi·in.$^{1/2}$ than that based on the surveyed data. At the lower σ_y levels, below about 220 ksi, the lower-bound curve for maraging steels lies significantly above that for the low-alloy steels. The curves obtained for two precipitation-hardening stainless steels fall just below the curve for the maraging steels up to σ_y of about 220 ksi; the three curves appear to merge at stresses above this value.

The presence of environments, other than aqueous chlorides and water, may require a significant drop in the proposed lower-bound K_{Iscc} values, as manifested by the plots for the sulfide environments. At σ_y of 120 ksi, a reference value in the NRC evaluation, the presence of aqueous H_2S can cause a drop in K_{Iscc} by some 50 ksi·in.$^{1/2}$ relative to K_{Iscc} for an aqueous chloride environment. Thus, a record of the environmental history during the operation of the nuclear-reactor plant could be a critical component in the final assessment of the bolting problem. We did not evaluate data on corrosion-assisted fatigue, and this failure mechanism, where operative, must also be factored into the analysis.

CONTENTS

LIST OF ILLUSTRATIONS

LOWER BOUND K_{Iscc} VALUES FOR BOLTING MATERIALS — A LITERATURE SURVEY

Alfred Goldberg and Mary C. Juhas

1. INTRODUCTION

A literature survey was made for the U.S. Nuclear Regulatory Commission (NRC) to determine lower-bound values of K_{Iscc} (threshold stress intensity for stress-corrosion cracking below which crack growth does not occur). The materials evaluated were bolting-type steels exposed to environments that might be related to operating conditions in nuclear-reactor plants. At the time this study was initiated, the NRC staff was concerned with formulating the final guidelines of acceptance criteria that would assure the structural integrity of support members for a number of nuclear-reactor plant components. The concerns and preliminary guidelines were documented in detail by NRC staff responsible for Generic Activity A-12.[1-3] One of these guidelines addressed the problem related to evaluating the integrity of high-strength bolting materials using a fracture mechanics approach. In particular, there was a need to develop a reliable lower-bound K_{Iscc} base that would permit establishing conservative and meaningful allowable loading limits for stressed bolts.

The bolting materials used in the reactor plants,[1,2] include moderate-, high-, and ultra-high-strength steels consisting of plain-carbon steels, Cr-Mo and Cr-Mo-Ni low-alloy steels, 17-4 PH and Custom 455 stainless steels, and 18Ni-maraging steels. In many cases, the ASTM specifications allow a wide range of compositions, with usually only the lower limit of tensile properties being designated. Thus, some of these bolts may have much higher yield strengths (σ_y) than are normally expected. It is well known that these steels, when heat-treated to high strength levels, are likely to be highly susceptible to stress-corrosion cracking (SCC). A sharp drop in K_{Iscc} occurs at some moderately high stress level depending on the class of alloys and the environment.[4-6] Thus, the use of steels at excessively high-strength levels may lead to early failure.

There is also a significant decrease in K_{Ic} with increasing σ_y; * however, it is not as dramatic as the corresponding decrease in K_{Iscc}. To determine a safe upper-limit for σ_y where SCC might occur, it is therefore especially important to establish the position of the K_{Iscc} lower-bound values in the region of rapid drop-off. Although there is a general decrease in K_{Iscc} with a decrease in K_{Ic}, correlations between these two parameters for a given alloy appear to be poor when related to variations in alloy composition and thermomechanical history. Evidence of opposing trends will be presented later in the report. Thus, it is important to recognize the danger of using estimated K_{Iscc} values based on combining limited K_{Iscc} with K_{Ic} data. The need for actual K_{Iscc} data for various alloy histories and compositions is also addressed in this report.

Documentation exists of a number of bolting failures in nuclear reactor plants. Although no special effort was made to review the history of such failures, some of the nuclear-related papers on bolting failures are referenced here.[7-10]

2. SCOPE

The scope of this study consisted of the following tasks:

1. To perform a literature survey and analysis on SCC of bolting-type materials in various environments that possibly reflect conditions at support structures in reactor plants (e.g., PWR steam generator and coolant pump supports).[1,2]

2. To establish lower-bound K_{Iscc} versus σ_y limits for different classes of materials.

3. To examine the effects of material variables, such as impurities, alloying composition, heat treatment, microstructure, etc., on K_{Iscc}.

4. To examine the effects of environmental variables such as relative humidity (RH), pH, concentration, temperature, polarization potential, etc, on K_{Iscc}.

2.1 DATA ACCEPTANCE CRITERIA

The survey was restricted largely to σ_y versus K_{Iscc} information. Where σ_y was not given, it was estimated from the reported hardness or tensile strength; these data are identified as such. The validity of K_{Ic} data are met when the following equations are simultaneously satisfied:

$$a_{min} = B_{min} = (W-a)_{min} = \frac{W_{min}}{2} = 2.5 \left(\frac{K_{Iscc}}{\sigma_y}\right)^2 \tag{1}$$

where a_{min}, B_{min}, and W_{min} are the minimum crack length, minimum specimen thickness, and minimum specimen width, respectively. As yet, there are no established ASTM requirements for K_{Iscc}. The practice has been to follow the requirements for K_{Ic}, however, the coefficient 2.5 may be too conservative.

A number of different specimen configurations and test procedures were reported. Constant-load specimens lead to increasing K_I values with crack growth. Here, the determination of K_{Iscc} generally involved either continuous or, more frequently, incremental load increases until crack growth was observed. This point was usually, but not always, verified by loading a new specimen just below this threshold stress intensity for some extended length of time. Constant deflection tests lead to decreasing K_I values as the crack propagates. Typically, a number of specimens were tested with different initial stress intensities (K_{Ii}); K_{Iscc} corresponds then to the value of K_{Ii} that would not lead to any crack growth in some specified time. Frequently, specimen dimensions were not indicated; however, the claim was usually made that these were within ASTM specifications for valid K_{Ic} determinations.

A possible equal or even more serious consideration than the presence of plane-strain conditions is the extensive range in testing times reported by different investigators; in a number of cases these times were not clearly indicated. Examples of the importance of having exposure times ranging into many thousands of hours to provide reliable K_{Iscc} values will be given later. Crack blunting caused by excessive K values in fatigue precracking (stress ranges often are not given) and during actual testing, especially in the variable constant-load tests, may affect the observed K_{Iscc} value. Because of these problems, all of which will usually lead to non-conservative rather than conservative K_{Iscc} values, we disregarded validating the reported data by use of Eq. 1. Departure from plane-strain conditions should also lead to non-conservative results. Although the use of invalid data would tend to increase scatter, such data should not fall below the lower-bound curves established for "valid" data.

2.2 MATERIALS

Bolting materials used in nuclear-reactor plants are mostly of compositions comparable to AISI 4140 and 4340.[1,2] Fortunately, much of the work surveyed covered these two classes of alloys and they were evaluated as separate groups. All remaining low-alloy and plain-carbon steels were combined as a third group. While H-11, 17-4 PH, Custom 455, and 18Ni-maraging steels were treated as four distinct groups. Data from experimental alloys with compositions departing from commercial alloys are identified as such.

2.3 MATERIAL AND PROCESSING VARIABLES

Alloy content, impurity content, melting and processing variables, heat-treatment, and microstructure all affect K_{Ic} values. Much work has been reported on attempts to determine the effect of these parameters on K_{Iscc}. While large improvements in K_{Ic} over a wide range of σ_y have resulted from microstructural refinement, melting practice, and impurity control, the effect of such material variables on K_{Iscc} usually appear to be minor, especially in the higher σ_y range. In the lower σ_y range (>150 ksi) however, the quenched-and-tempered structures are generally more resistant to SCC than are the normalized-and-tempered or bainitic-and-tempered structures of equal strengths. Because of a primary interest in the high strength range (≥150 ksi) the steels were not separated according to melting practice, minor differences in composition, or heat treatment. However, examples will be presented where material parameters do or do not show any effects. Finally, it was noted that differences in results reported by different investigators on apparently the same materials were usually greater than variations in results reported for a specific study concerned with the effects of material parameters.

2.4 ENVIRONMENTAL VARIABLES

There is considerable evidence in the literature that supports the contention that hydrogen attack,[11-13] as well as anodic dissolution, contributes to SCC in aqueous solutions and, in fact, these two mechanisms may act conjointly or successively in a given environment.[14] Although the rate-controlling process may be different in various hydrogen media (molecular, atomic, precharged, aqueous), thus affecting the crack-growth rate (CGR) in stage II, the K_{Iscc} or K_{IH_2}* does not vary significantly with the source of hydrogen.

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* The term K_{IH_2} is used here for the threshold stress in hydrogen gas or hydrogen-charged environments.

Therefore, data on hydrogen studies have been included in the analysis. Some recent work in France[15] on several low-alloy steels tested in gaseous hydrogen and in aqueous NaCl suggests that $K_{IH_2} > K_{Iscc}$; however, the time to failure (t_f) above the threshold value is considerably less in H_2 than it is in the aqueous environment. The ranking of the various steels examined were the same for the two environments.[15] The data plotted by Sandoz for 4340 steels are in general agreement with these results in that $K_{IH_2} > K_{Iscc}$ (salt water) $> K_{IH}$ (cathodic charge).[12]

Since exposures to coastal and industrial environments are likely to have occurred, data has been included for salt water (NaCl solutions, sea water, sea coasts, and artificial sea water) and sulfide (H_2S aqueous and H_2S gas) environments. The data show that K_{Iscc} decreases with increase in humidity and that there is little difference between distilled water and NaCl solutions. However, sea-water environments are generally more damaging than are the NaCl solutions. The rapid drop in K_{Iscc} with an increase in σ_y occurs at much lower stresses in sulfide than in salt solutions. Thus, a lower-bound curve based on sulfide solutions is probably over-conservative for reactor plant environments; however, these data do point to the dangers of industrial sulfide-bearing environments. Exact compositions of the various environments are not specifically identified in our paper. The NaCl solutions range from approximately 2 to 5 wt. %; the sulfide solutions range from H_2S in distilled water to the NACE solution (5% NaCl + 0.5% acetic acid, saturated with H_2S).

For water and aqueous chlorides there is little, if any, effect of pH on K_{Iscc} over the range of pH about 2 to 9.[4] This is consistent with the observation that the pH and solution potential at the crack tip are virtually independent of the conditions external to the crack tip.[13,16] Thus, the pH was not considered as a variable for these environments. For aqueous sulfides the susceptibility to cracking does increase with pH over this same range. Nevertheless, the possible effects of pH were not isolated.

There is a minor effect of polarization potential on K_{Iscc}. However, the corresponding effect on CGR can be significant; the minimum rate occurs near the open-circuit potential. Either high cathodic (atomic hydrogen) or anodic (molecular hydrogen and/or anodic dissolution) reactions can result in an increase of several orders of magnitude in CGR. The selection of data was restricted to freely corroding potential tests. If applied potentials are used in the reactor plants to reduce corrosion (or if stray voltages exist), the lower-bound values for freely corroding conditions are likely to be too conservative. Some examples of the influence of polarization will be presented.

Data of K_{Iscc} versus σ_y are presented to provide a guide for establishing lower-bound curves for the alloys and environments outlined in the above. The lower-bound curves originally proposed by the NRC are included in all figures. The presentation of data will be followed with a detailed discussion on the effects of various material and environmental variables.

3. K_{Iscc} VERSUS σ_y PLOTS

The data are categorized according to type of steel and further subdivided according to test environment. Some of the data were extracted from review articles where different environments having similar effects had been grouped together. Identification of the specific environment for each of these data points was not always possible as some of the original references were not available. Therefore, those environments that include various purities of H_2O, aqueous NaCl, simulated sea water, sea water, and coastal environments were plotted together as "combined." Variations in K_{Iscc} resulting from testing of high-strength steels in these different aqueous environments usually can be neglected.[4,6,17] Some of the data in the combined plots are likely to be repeated in plots for the individual environments. Duplicate values were omitted in plots that contain a high density of data. All available SCC data for the aqueous (H_2O, NaCl, sea waters, and H_2S) environments were included. Data for gaseous (H_2 and H_2S) environments were obtained only incidental to the search for the aqueous environments. The role of gaseous-induced cracking is likely to be secondary to that caused by the aqueous environments.

3.1 SAE 4340 STEELS:

Figure 1 contains the "combined" aqueous data for SAE 4340 steels.[4,5,17-46] The solid line, A-A, in all figures refers to the lower-bound K_{Iscc} curve for low-alloy steels originally specified by Task Group A-12.[1-3] Figure 2 contains 4340 data separated according to specific aqueous environments.[4,5,12,18,21-23,25,27-29,31,38,40,41,43,44,46-60] Data for 4340 exposed to H_2S environments are plotted in Fig. 3 for dry gas,[4,49,55,61-63] wet gas,[64,65] and aqueous solutions.[4,39,66-68] For the aqueous H_2S solutions, no distinction was made between those containing only H_2S and those containing additions of chlorides and acetic acid. There does not appear to be any significant effect of chloride additions on the K_{Iscc}, although the acidified solutions accelerate the cracking process.[66] Too low an H_2S concentration in aqueous solutions may result in excessive K_{Iscc} values.[66] The H_2S gas was assumed to be dry unless specified otherwise.

Based on the water and aqueous chlorides data, in Figs. 1 and 2, the proposed NRC lower-bound curve, A-A, is reasonable and not over-conservative. We believe that the large scatter in data for a given σ_y is most likely due to variations in material composition, manufacturing history, and heat treatment, although the role of these variables is not too clear. It is very likely that some of the scatter is also due to variations in testing methods, exposure times, and interpretation of test results. Above about 190 ksi, σ_y has little affect on K_{Iscc}. Below this value K_{Iscc} rises rapidly with a decrease in σ_y.

As can be seen from Fig. 3 the presence of H_2S, especially when associated with H_2O has a large effect on lowering K_{Iscc} over a wide range of σ_y. The lower-bound curve for the plotted data is given by curve B-B.

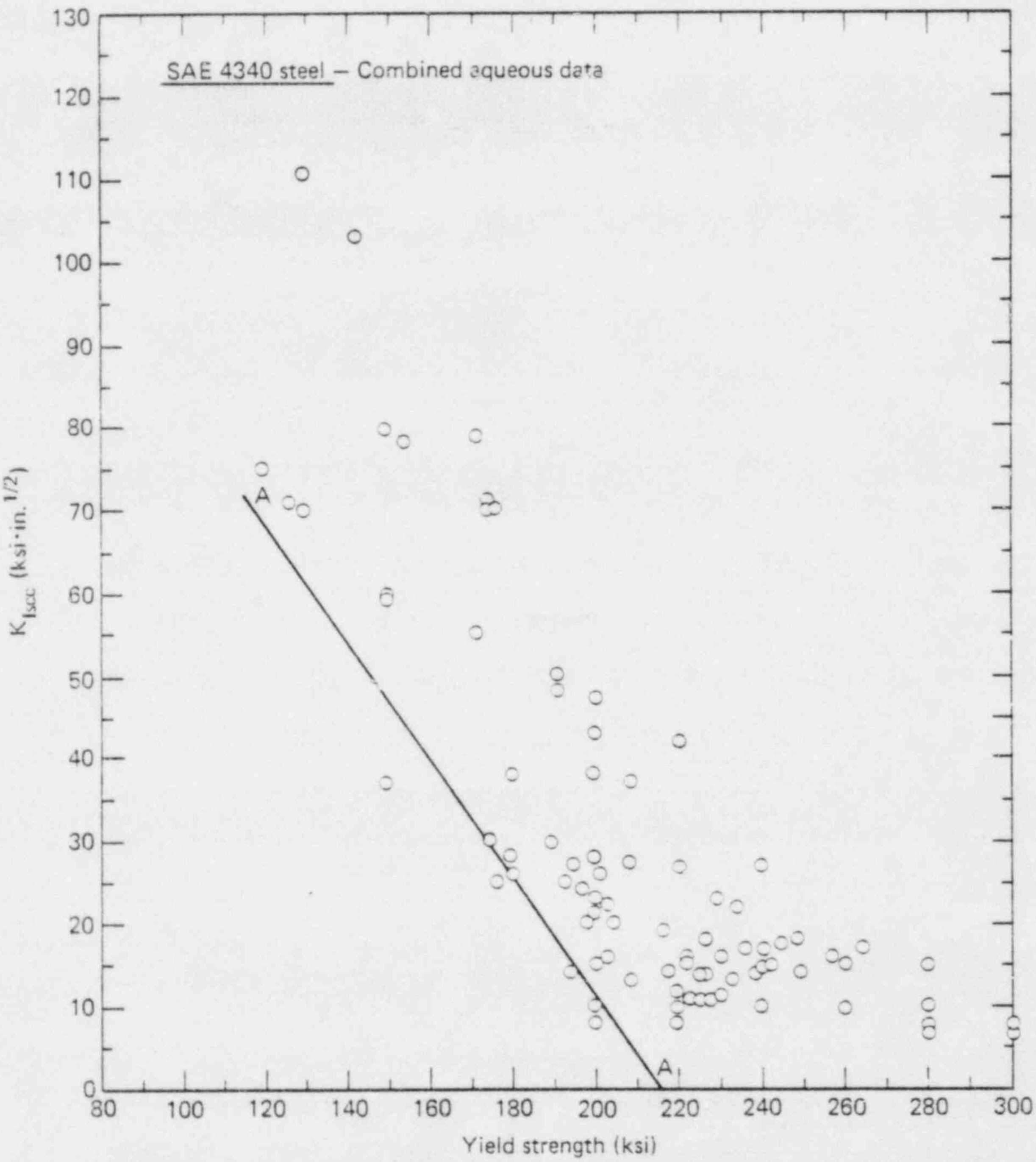

Figure 1. $K_{I\,scc}$ versus yield strength for SAE 4340 steel reported for "combined" aqueous data.

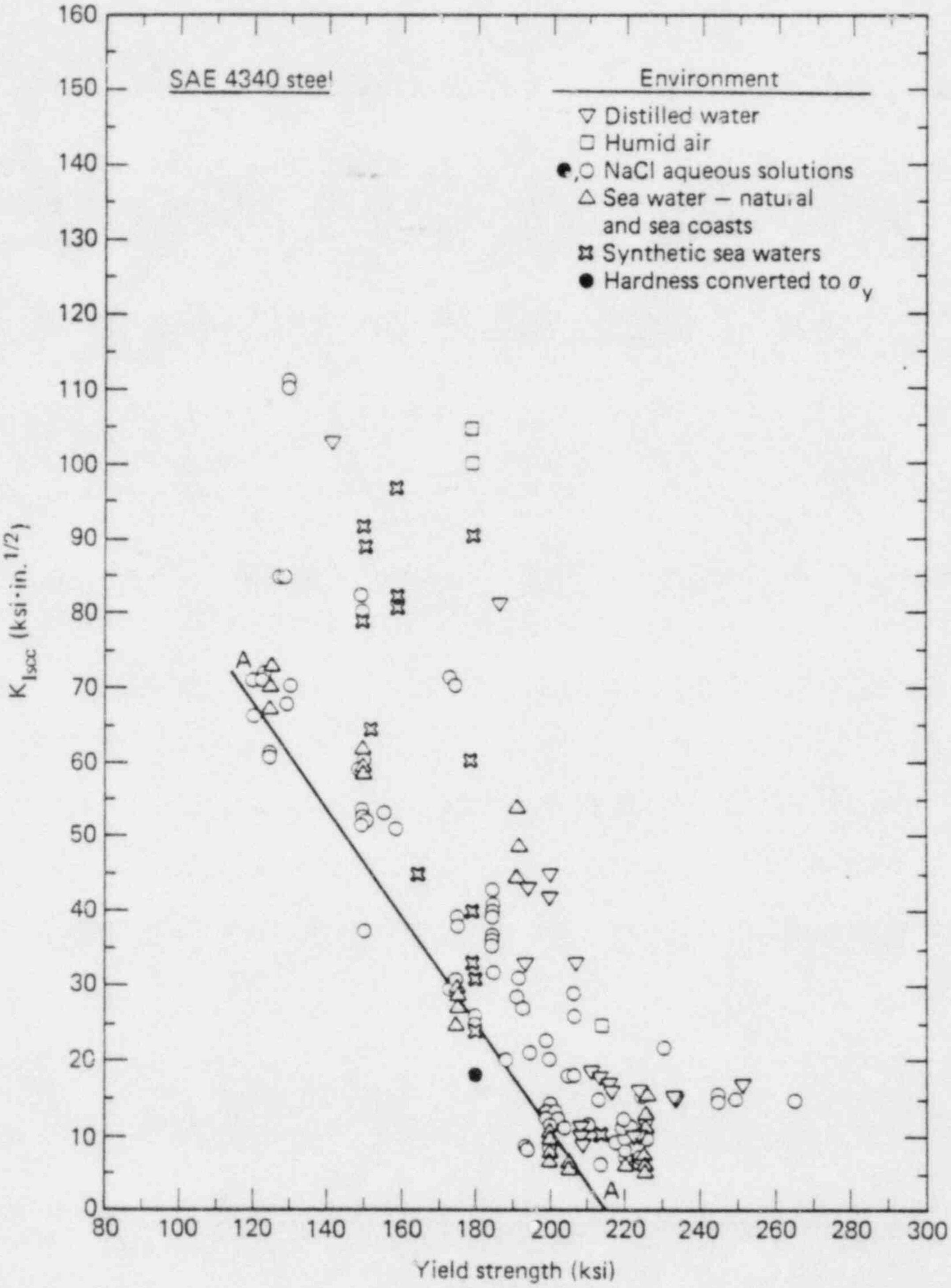

Figure 2. K_{Iscc} versus yield strength for SAE 4340 steel reported for various environments containing water or aqueous chlorides.

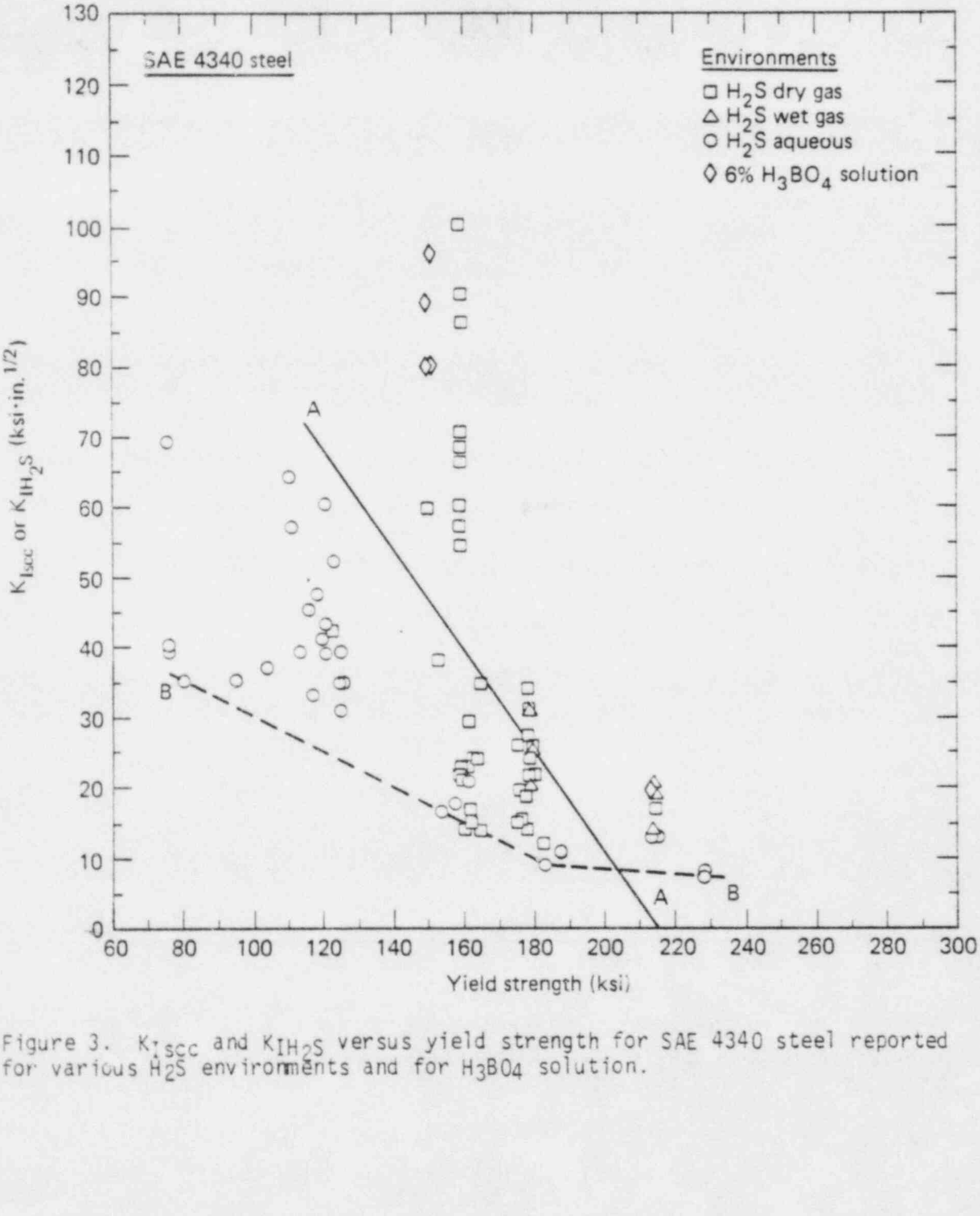

Figure 3. K_{Iscc} and K_{IH_2S} versus yield strength for SAE 4340 steel reported for various H_2S environments and for H_3BO_4 solution.

In both aqueous chlorides and H_2S environments K_{Iscc} appears to be relatively insensitive to σ_y above about 190 ksi, with the lower-bound values falling close to 10 ksi·in.$^{1/2}$. The large scatter reported for the dry gaseous data is primarily related to the range of H_2S partial pressures. For moist H_2S gas, no effect of pressure was obtained over the range from 3 to 115 psi.[65] Also included are data on compact bolt-loaded tension specimens tested in 6% H_3BO_4 aqueous solutions reported by Landerman.[49] Data on 4340 steel exposed to H_2 environments are presented below.

3.2: SAE 43XX-TYPE OF STEELS:

Figure 4 contains data for several different 43XX-type of steels heat treated to ultra-high strengths and exposed to various environments, but excluding sulfides and H_2. The data all fall within the range where K_{Iscc} is insensitive to σ_y. The lower-bound curve, C-C, for these specialty steels at $\sigma_y \geq 190$ ksi lies at about 10 ksi·in.$^{1/2}$. This is higher than the lower-bound values of 5 to 8 plotted for the 4340 steels over the same σ_y range in Figs. 1 and 2. The references for Fig. 4 are 4330M,[4,69] 4330V[4,5,47,48,57] 4335,[53] 4340M and 300M,[4,28,41,47,54,57,59,71] and experimental 4340-(1)[72] and 4340-(2)[4,5,41]. Figure 5 contains limited data for 4335V,[4,73] 4340,[4,12,21,65,74-77] and 300M[78] in H_2 gas. The large range in K_{IH_2} reported for a given σ_y is primarily related to a corresponding variation in the partial pressure of H_2.

3.3: SAE 41XX-TYPE OF STEELS

Figure 6 shows data reported for 4130[5,24,53] and 4140[47,79] tested in H_2O and aqueous chlorides. The data are very limited; nevertheless, the results confirm the conclusion drawn from the corresponding 4340 data that the proposed NRC lower-bound curve is reasonable and that the K_{Iscc} value is insensitive to variations in σ_y above about 190 ksi. Data on the effect of H_2S environments for 4130,[48,81-83] 4130 modified,[83] 4135 modified,[68,80,82-84] and 4140[4,5,61,66,67] are shown in Fig. 7. Figure 8 contains some data on hydrogen gas effects.[4,85,86] As in the previous section, the large spread in K_{IH_2} is due to a corresponding spread in hydrogen pressure,

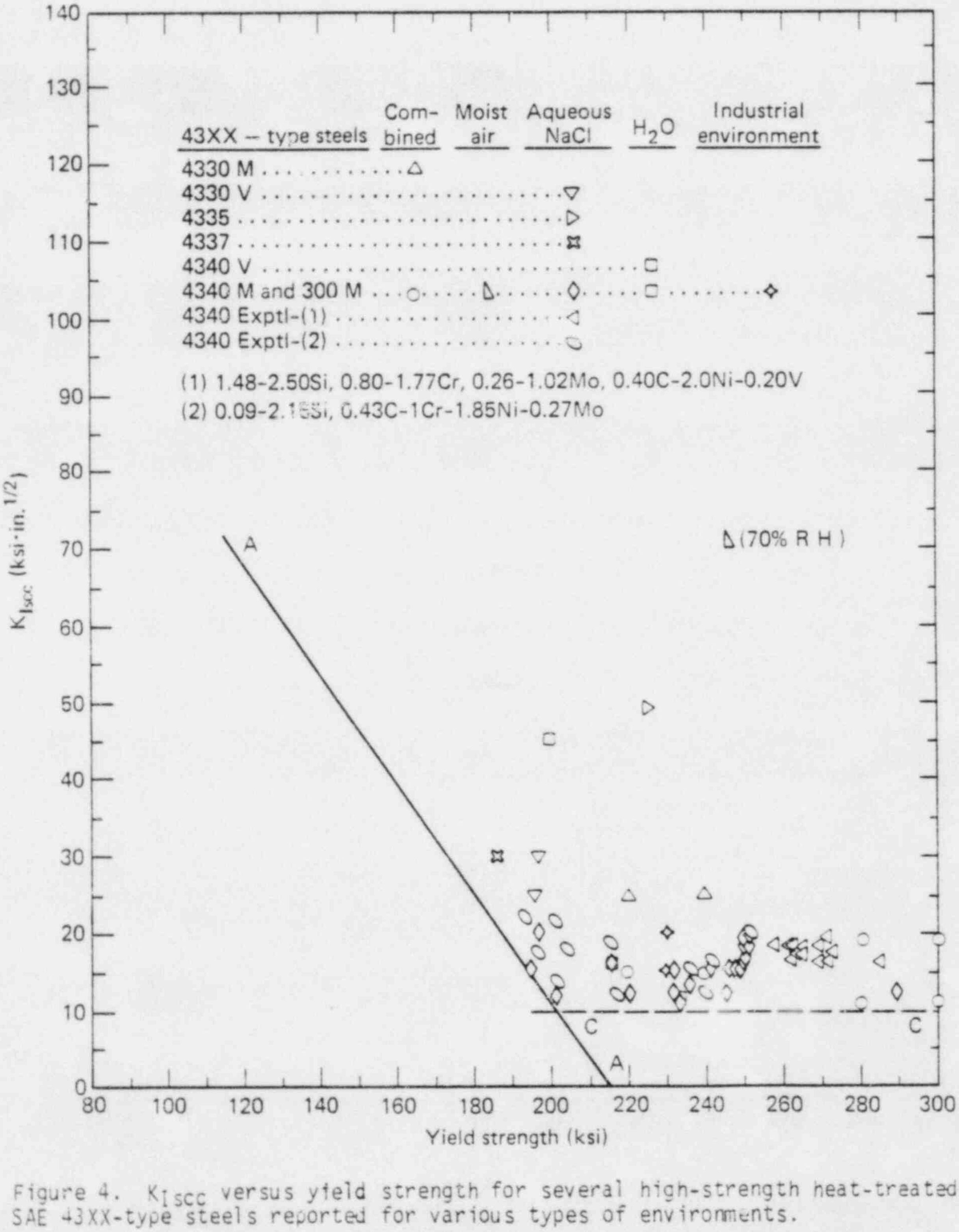

Figure 4. K_{Iscc} versus yield strength for several high-strength heat-treated SAE 43XX-type steels reported for various types of environments.

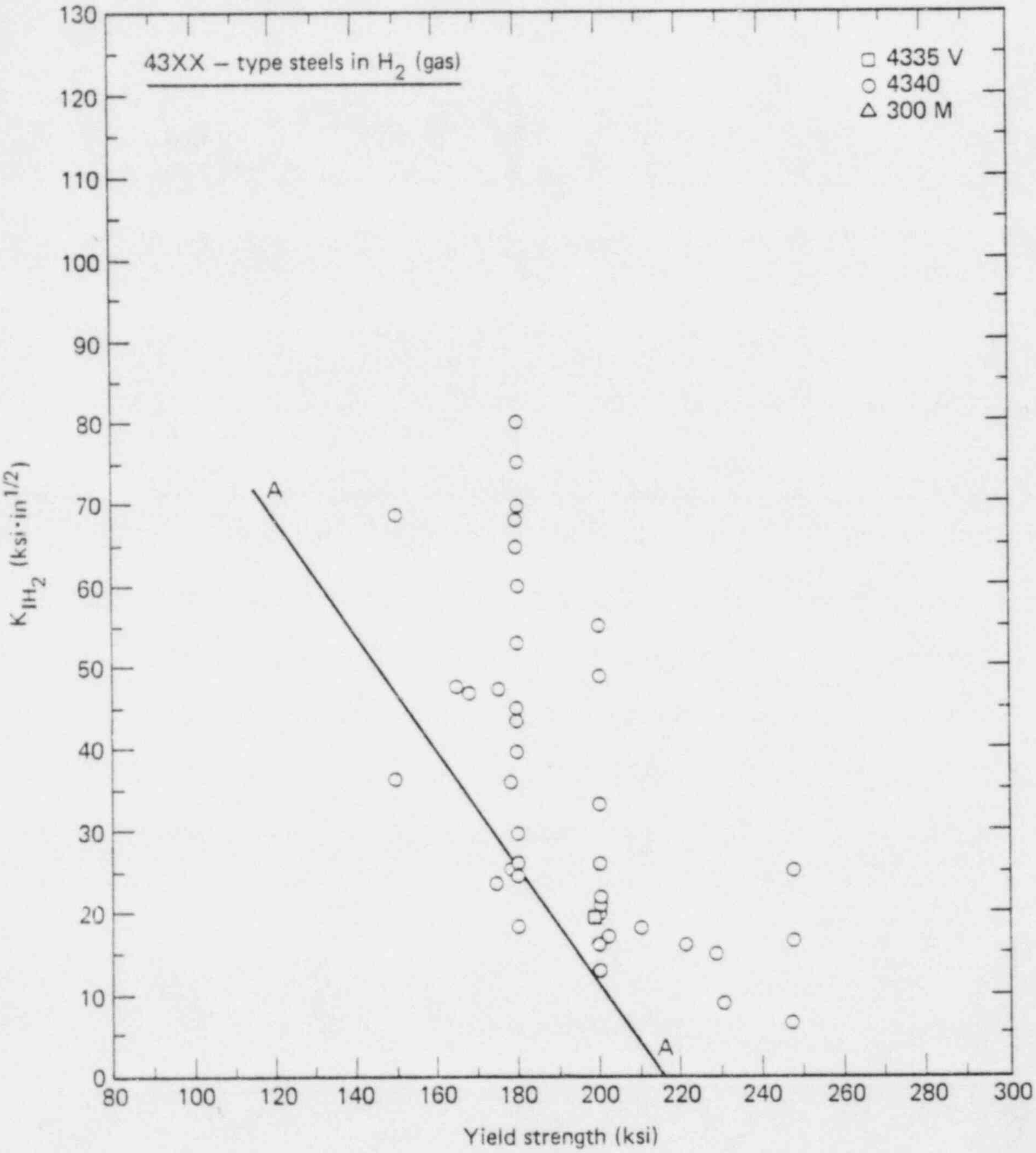

Figure 5. K_{IH_2} versus yield strength for SAE 4335V, SAE 4340, and 300M steels reported for H_2 gas.

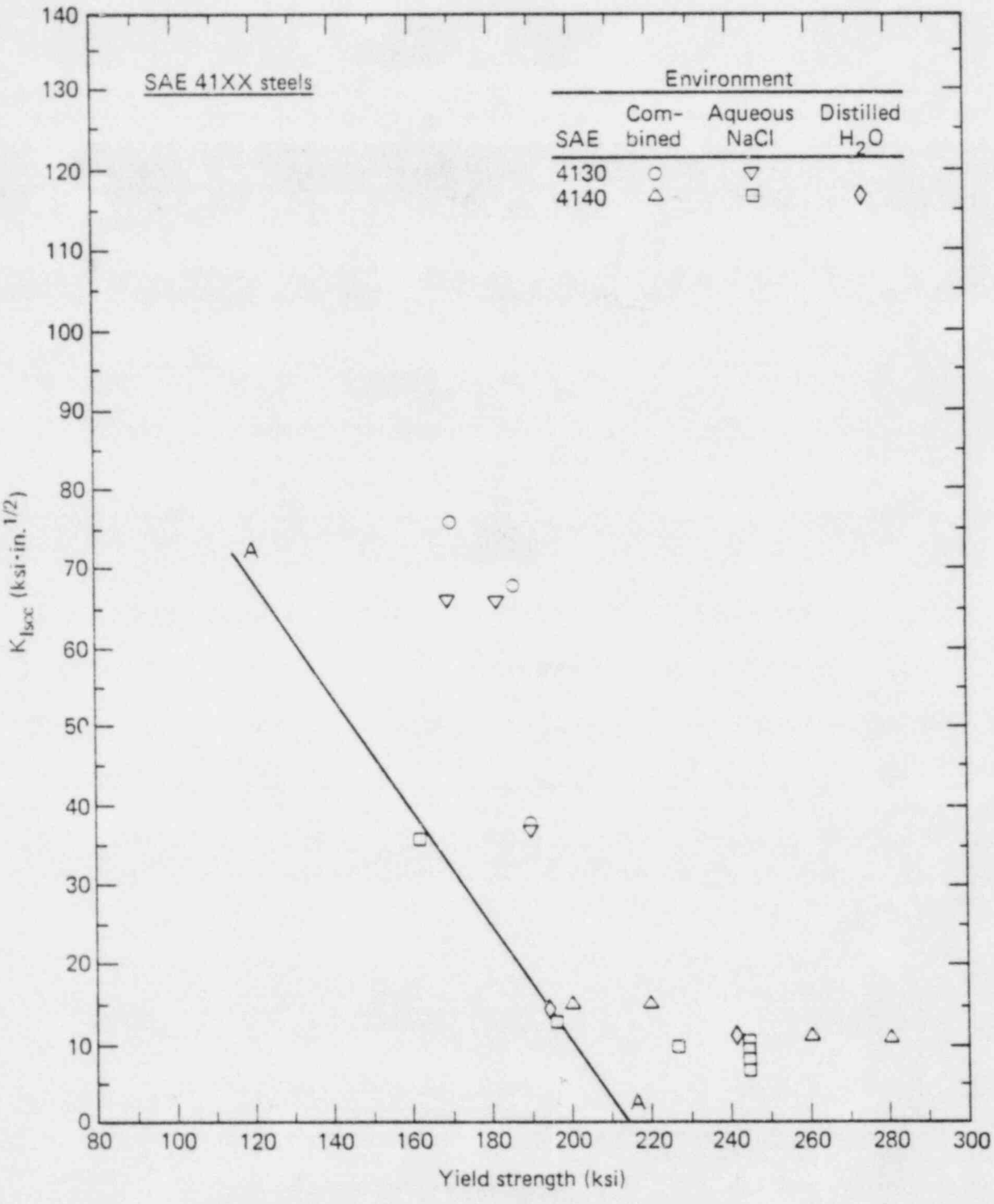

Figure 6. K_{Iscc} versus yield strength for SAE 4130 and SAE 4140 steels reported for "combined," aqueous NaCl, and distilled H_2O environments.

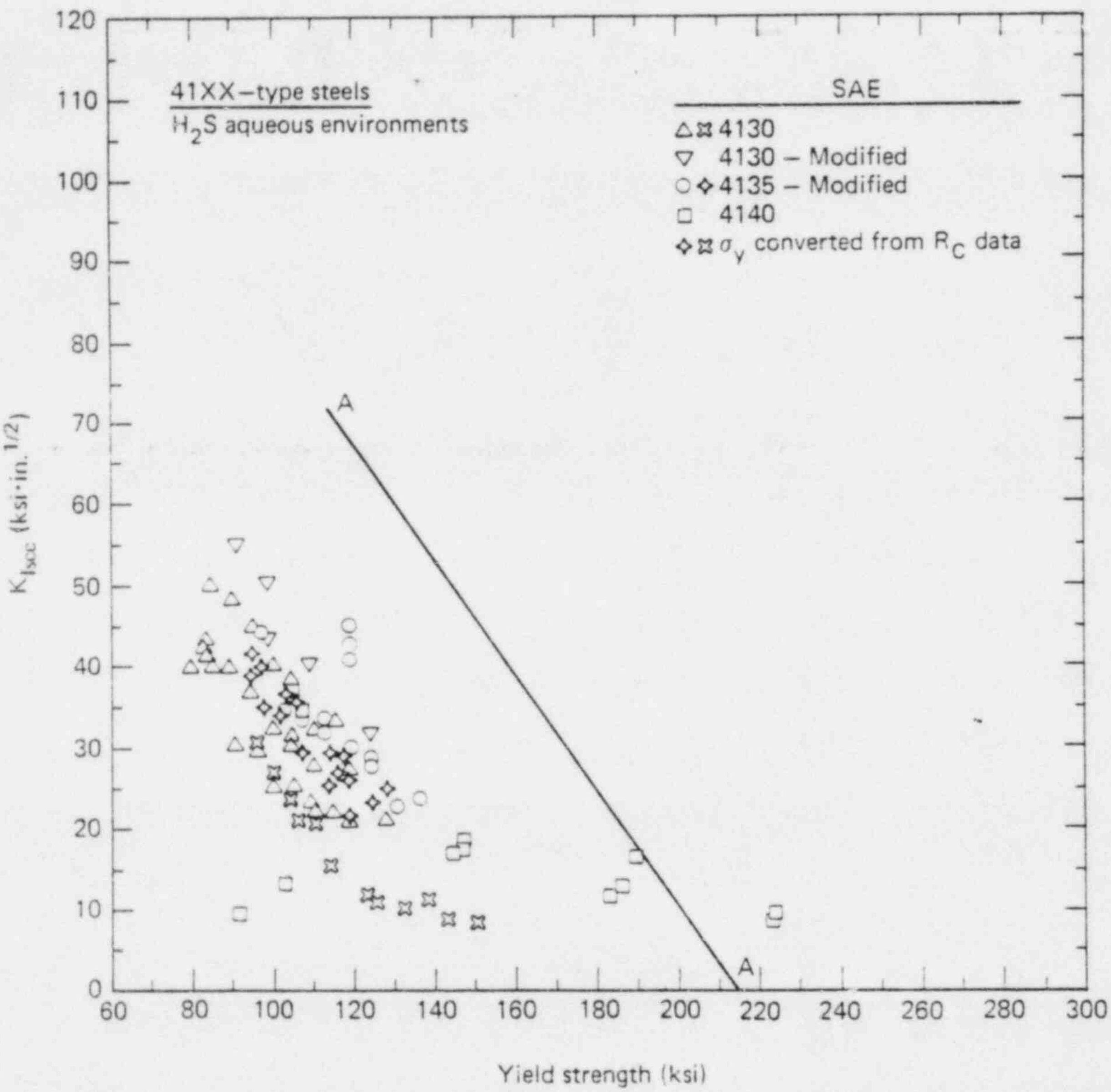

Figure 7. K_{Iscc} versus yield strength for several SAE 41XX-type steels reported for H_2S aqueous environments.

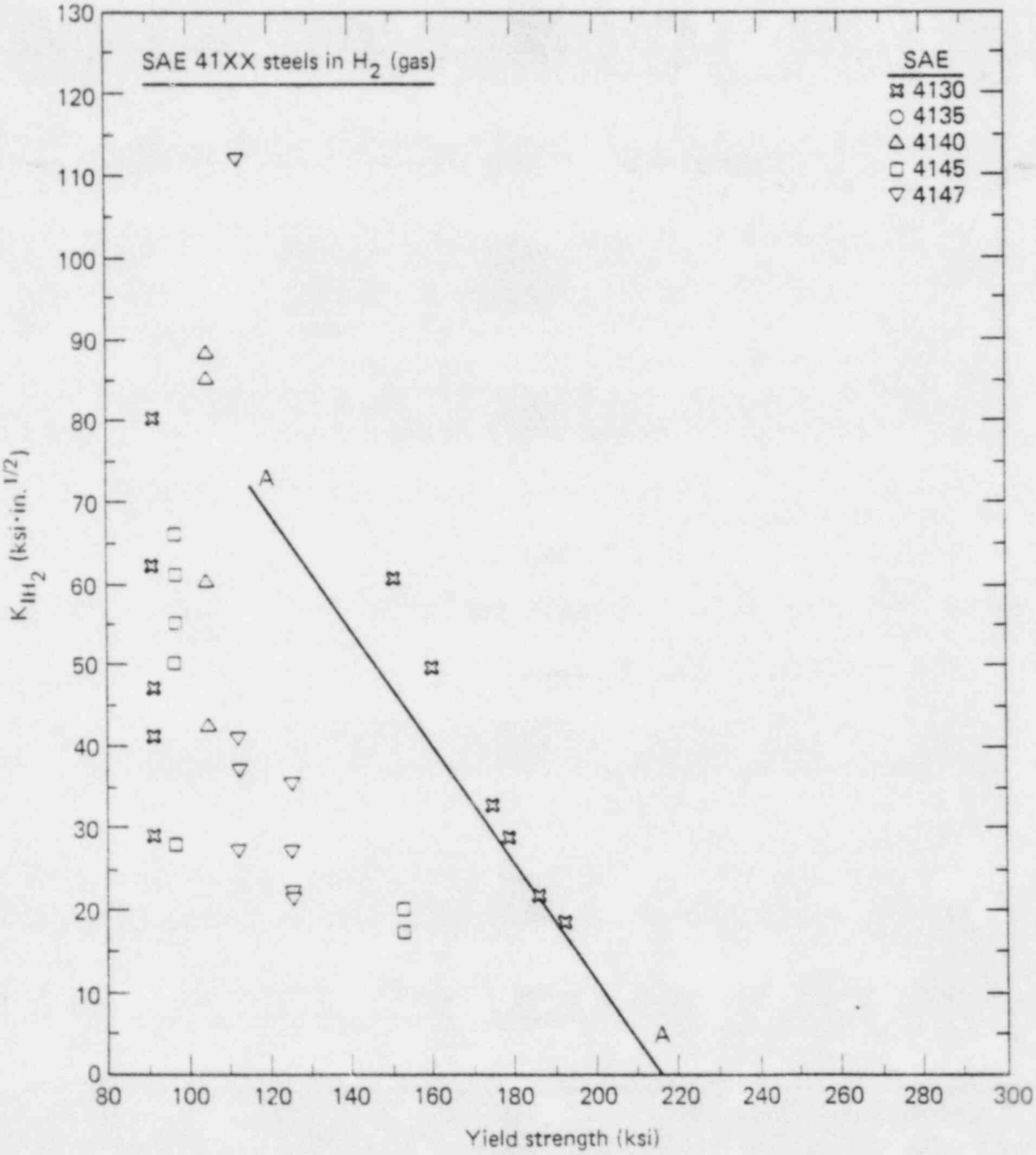

Figure 8. K_{IH_2} versus yield strength for several SAE 41XX steels reported for H_2 gas.

which ranges from about 11 to 14,000 psi. The data for 4130 that lie just above the proposed NRC lower-bound curve, A-A, were obtained at 11.2 psi.[86] Those data falling significantly below this curve generally were obtained from tests at above 3000 psi H_2 pressure.[85]

3.4: D6AC STEEL

Figure 9 contains available data for D6AC exposed to "combined," distilled water, and aqueous chloride environments,[4,5,17-20,28,29,34,37-39,42,45,47,53,56,59,87-90]. All data clearly lie above the proposed NRC lower-bound curve, A-A. However, at the higher stress levels, above about 220 ksi, the spread in K_{Iscc} as well as the lower-bound values appear to be independent of σ_y. The data for D6AC are not too dissimilar from those obtained for the 43XX and 41XX series of steels.

3.5: H-11 DIE STEEL

Figure 10 shows data plotted for H-11 die steel tested in humid air and the various aqueous environments of water, sea water, coastal, and NaCl.[4,5,29,35,37,38,56,57,59,91,92] The comments made for the D6AC steel data are also applicable here. The data indicated by the diamond symbols, from low to high K_{Iscc} values, correspond to variations in humidity from 100 to 0.1%, respectively.

3.6: HY STEELS

Figure 11 contains the results reported for HY steels (HY80 to HY150) in air, aqueous chloride, synthetic and natural sea-water environments,[93-104] and in combined environments.[104-110] The arrows on four of the data points indicate "invalid" data due to insufficient section size. With reference to the proposed NRC lower-bound curve, A-A, these steels are superior to the 43XX and 41XX steels at least for $\sigma_y \leq 170$. Bolts, however, are usually not made of HY-type steels. Some data are included for H_2S environments[4,67] which fall below the proposed NRC lower-bound curve for non-H_2S environments.

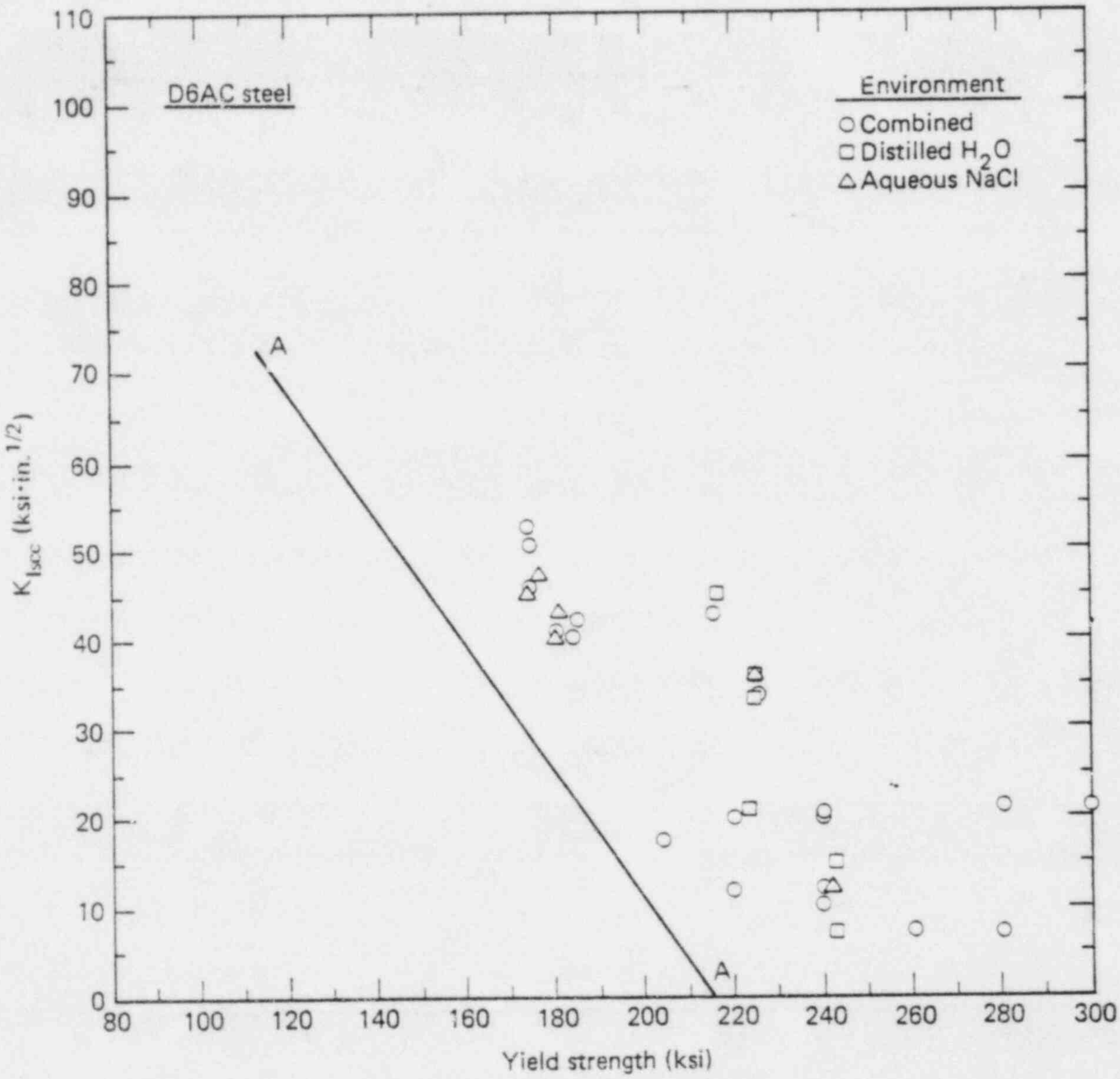

Figure 9. K_{Iscc} versus yield strength for D6AC steel reported for "combined," aqueous NaCl, and distilled H_2O environments.

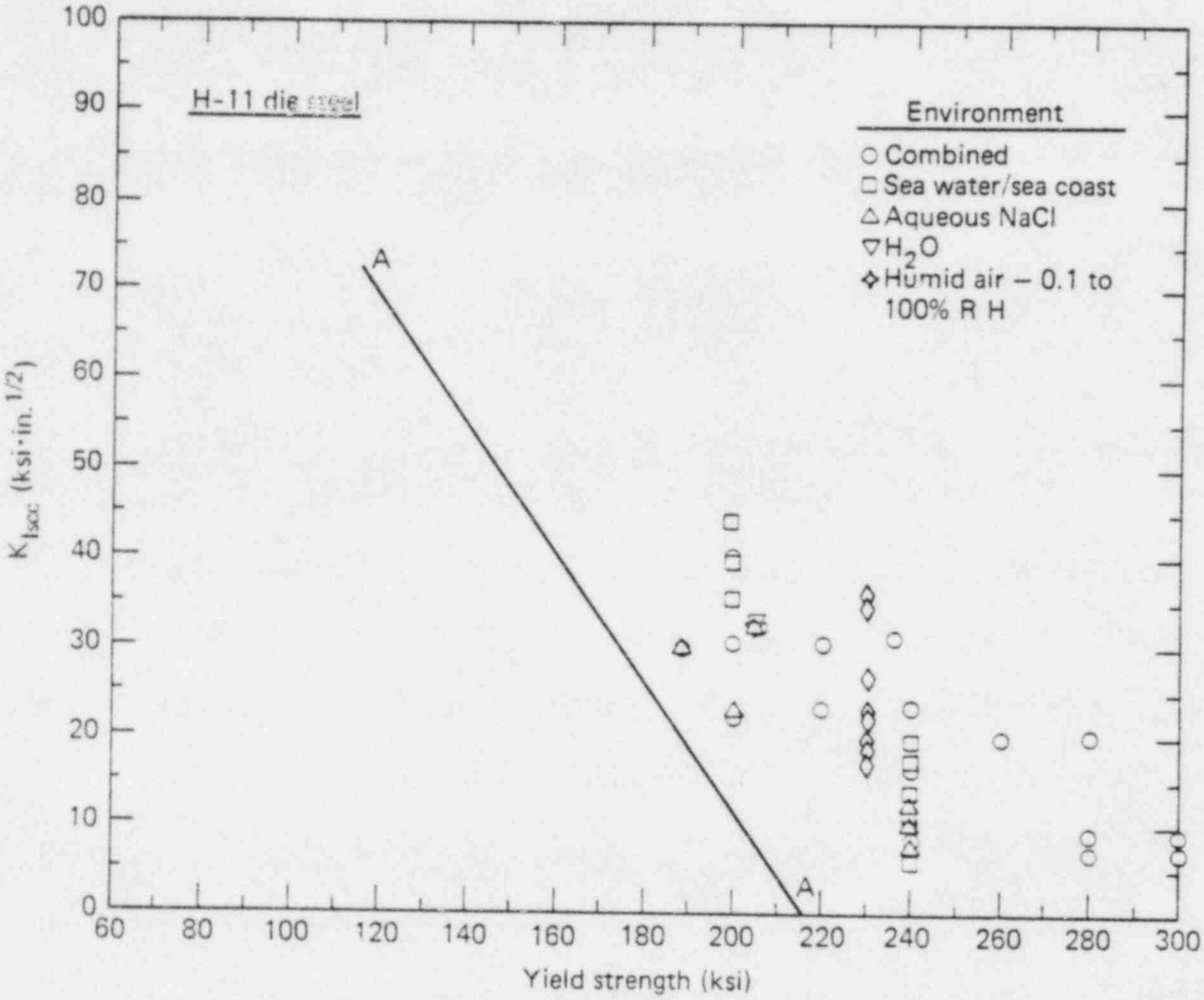

Figure 10. K_{Iscc} versus yield strength for H-11 die steel reported for various environments containing water or aqueous chlorides.

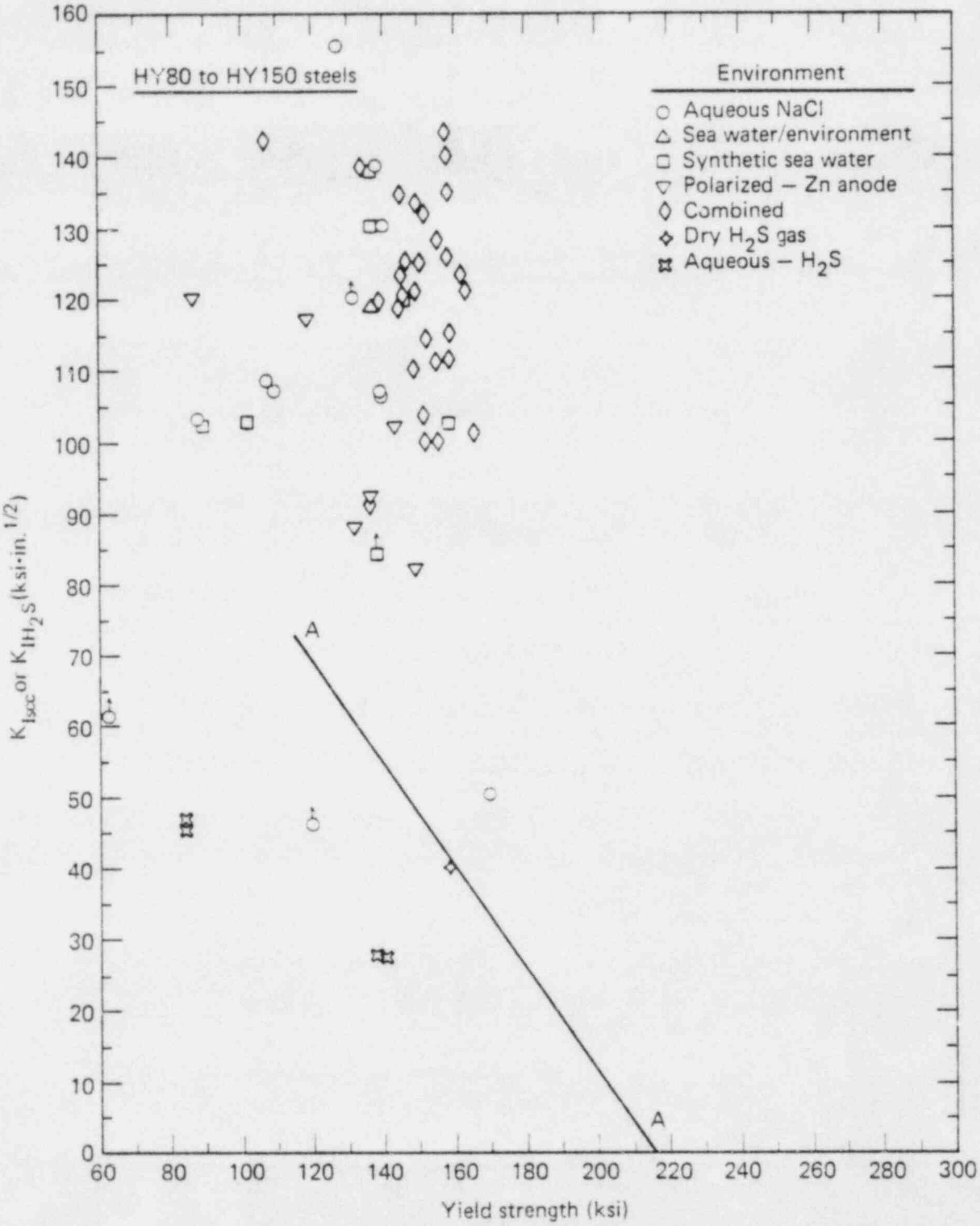

Figure 11. K_{Iscc} versus yield strength for HY steels reported for various environments containing water, aqueous chlorides, and/or H_2S.

3.7: MISCELLANEOUS LOW-ALLOY STEELS

Limited data were obtained for various miscellaneous low-alloy steels tested in water and various chloride-containing environments. These data are primarily for foreign steels. The data are plotted in Fig. 12.[4,15,28,95,102,111-117] With the exception of two questionable data points indicated by arrows the data either fall close to or above the proposed NRC lower-bound curve. Again, the lower-bound data appear to be independent of σ_y above a value of about 190 ksi.

Figure 13 contains data available for miscellaneous low-alloy steels tested in dry H_2S gas[4,63,115,118,119] and in aqueous H_2S solutions,[4,67,68,82,101,119-124] (usually the H_2S saturated, 3.5% NaCl-0.5% acetic acid NACE solution). The lower-bound curve for H_2S environments would fall considerably below the proposed NRC curve, A-A.

Threshold data of several low-alloy steels exposed to H_2 gas[4,85,115,125-128] are plotted in Fig. 14. Also included are some results for HY80, HY100, and HY130[4,85,125] and H-11 die steel.[129] As in previous such plots the range in K_{IH_2} for a given stress level is usually related to the P_{H_2}. The P_{H_2} for the plotted data ranges from 14.7 to 14,000 psi. It would be more informative if the data for all the low-alloy steels were analyzed as a function of P_{H_2} and then cross-plotted with K_{IH_2} versus σ_y for a constant P_{H_2}. However, since the H_2 and H_2S results were peripheral to the main objective of this review, no further effort was undertaken in this area.

3.8: PRECIPITATION HARDENING STAINLESS STEELS

Bolting materials of Custom 455 and 17-4 PH are being used in the reactor plants supports.[1,2] Data obtained for these two materials are plotted in Figs. 15[4,49,60,130,131] and 16,[4,5,17,19,47,52,53,130,132-137] respectively. The proposed NRC lower-bound curves for both the low-alloy steels (A-A) and maraging steels (D-D) are superimposed on these data. The lower-bound curve, E-E, in Fig. 15 corresponds to the limited data for H_2S. Excluding the H_2S data, the lower-bound curve for the remaining environments

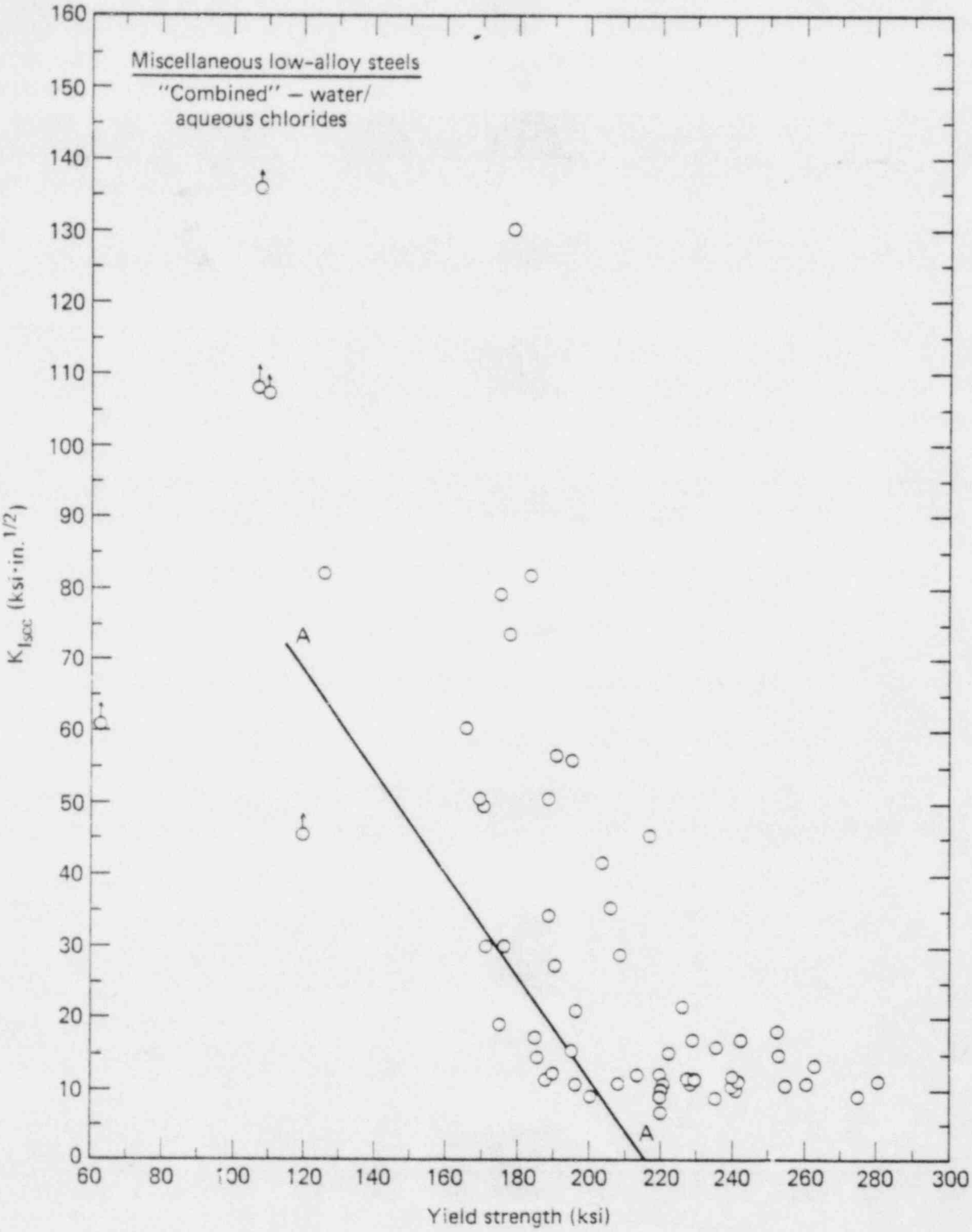

Figure 12. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for "combined" environments.

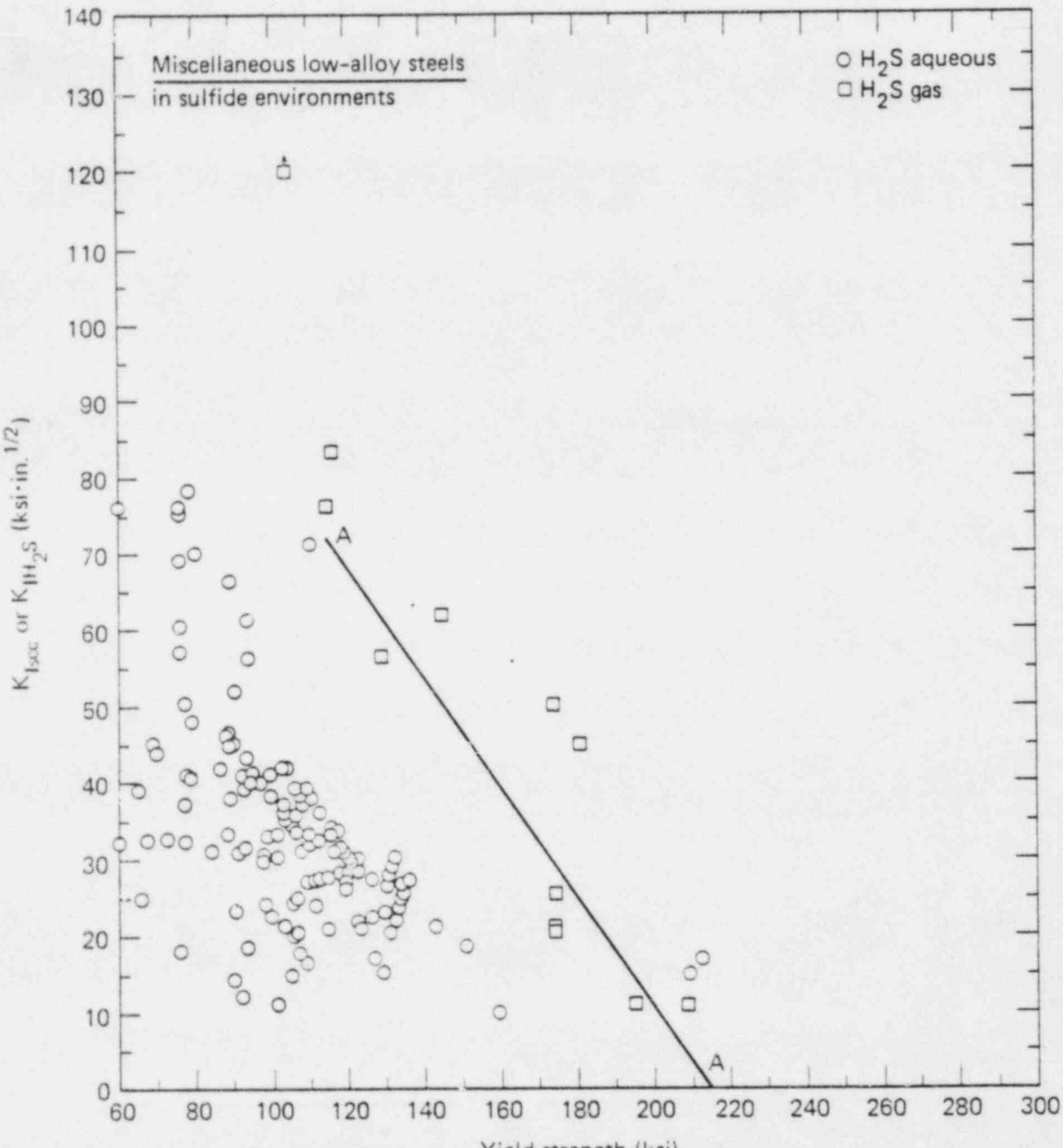

Figure 13. K_{Iscc} versus yield strength for miscellaneous low-alloy steels reported for H_2S environments.

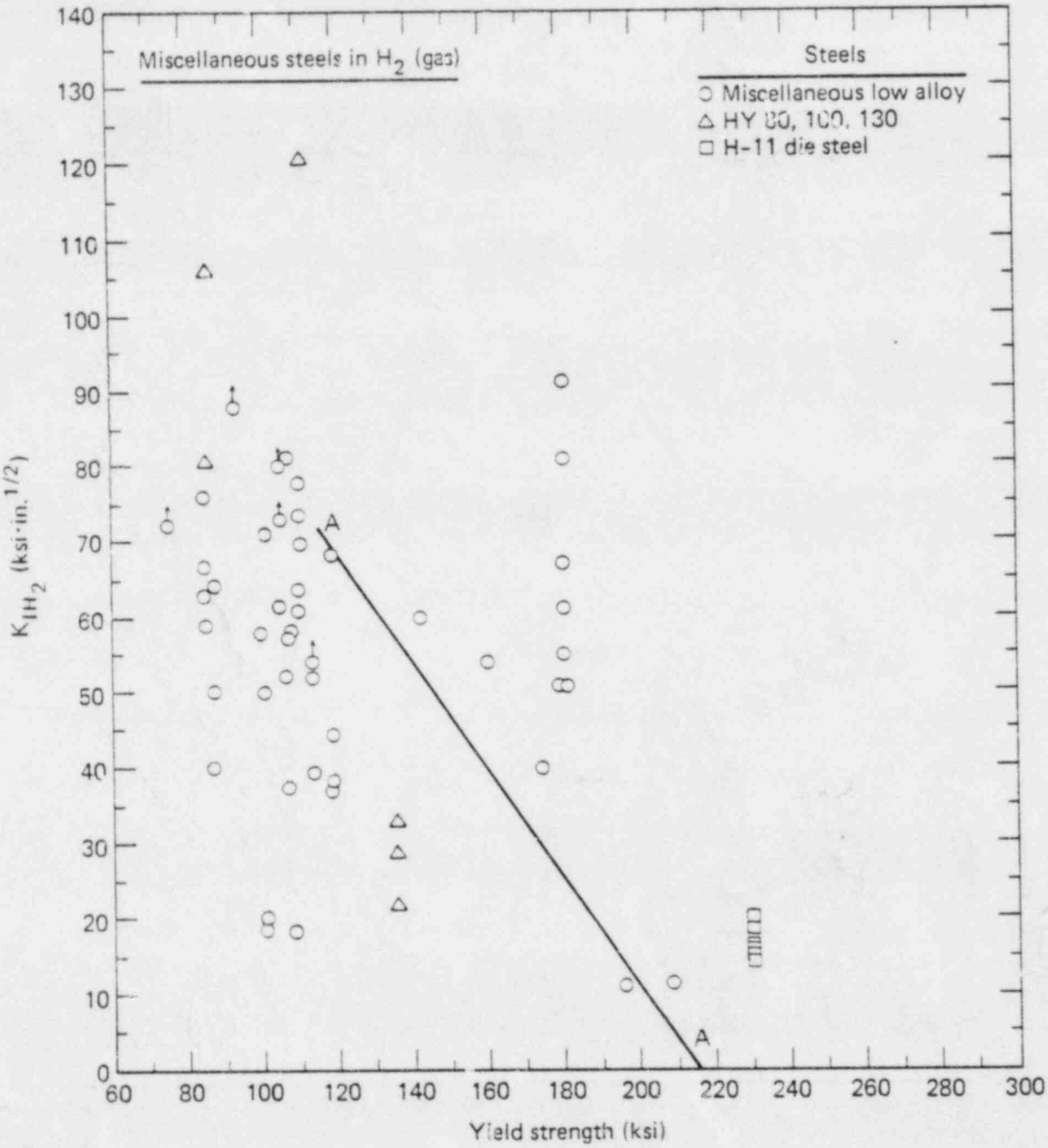

Figure 14. K_{IH2} versus yield strength for several different types of steels reported for H_2 gas.

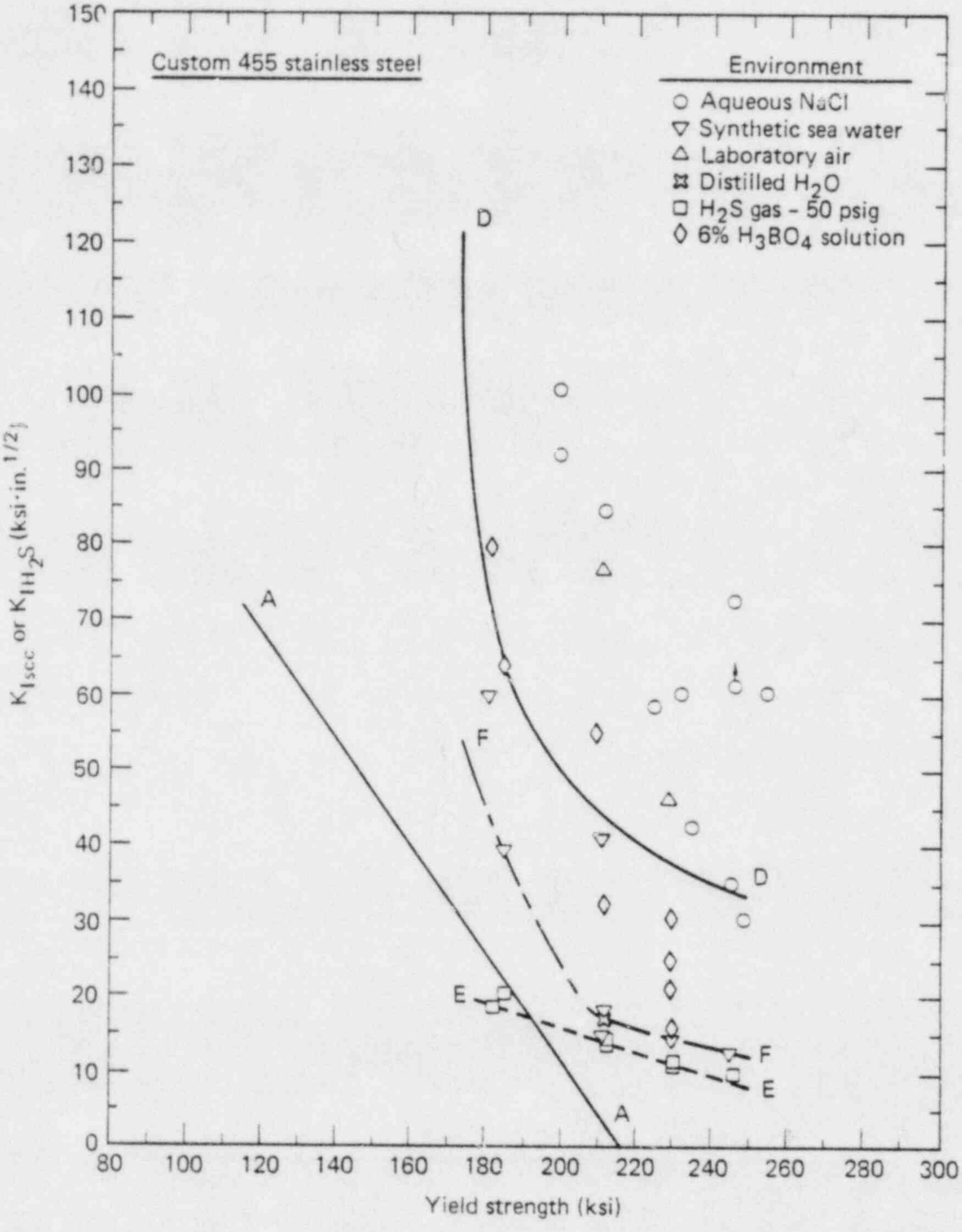

Figure 15. K_{Iscc} or K_{IH_2S} versus yield strength for Custom 455 stainless steel reported for various environments containing water, aqueous chlorides, air, H_2S gas, or H_3BO_4 solution.

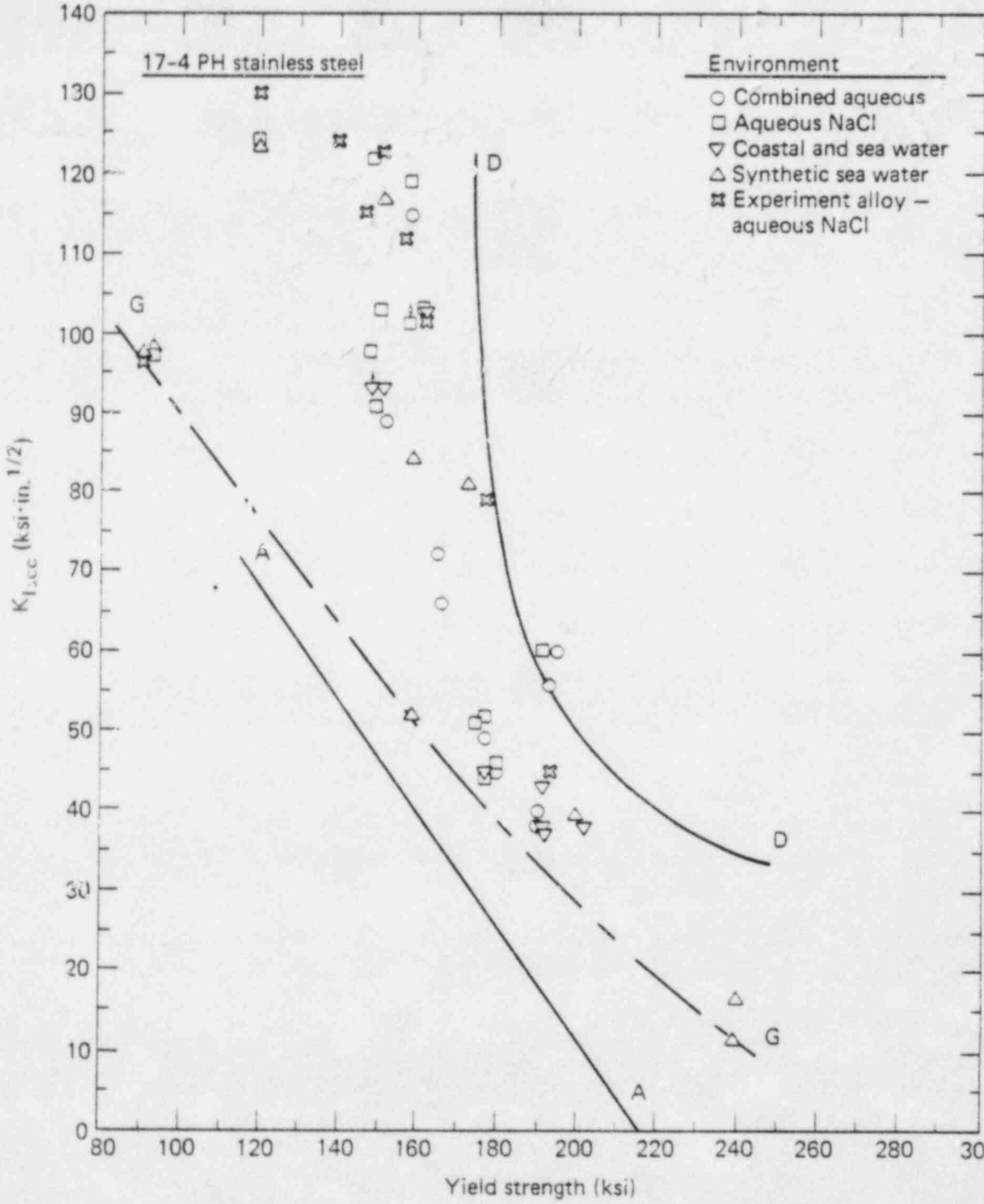

Figure 16. K_{Iscc} versus yield strength for 17-4 PH stainless steel reported for "combined" or specific aqueous chloride environments.

falls between the curves for the low-alloy and maraging steels as suggested by curve F-F. With the exception of the aqueous NaCl data, most of the data used for Fig. 15 were obtained from reference 49. Consistent with the data for Custom 455, the data obtained for 17-4 PH suggest that the lower-bound curve for this alloy also falls between the curves for the low-alloy and maraging steels. This is also depicted by curve G-G in Fig. 16. The stress level at which K_{Iscc} becomes insensitive to σ_y for these two steels is not clearly evident. Excluding the H_2S gas, it would appear to be relatively insensitive to σ_y above 210 ksi.

3.9: 18Ni-MARAGING STEELS

Considerable information is available on SCC of 18Ni-maraging steels in various aqueous environments. Figure 17 contains K_{Iscc} data obtained largely from review papers[4,5,17] for the "combined" (water and chloride-bearing) environments.[4,5,17,19,20,26,37,40,42,43,45,46,59,133,138-154] Clearly, the proposed NRC lower-bound curve, D-D, for maraging steels should be displaced to considerably lower levels at $\sigma_y \geq 190$ ksi, namely to H-H. Figure 18 contains data plotted for specific environments.[4,18,21,27-29,40,47,57,58,60,71,102,114,138,142,143,150,151,155-160] Included also in Fig. 18 are the proposed NRC lower-bound curve, D-D, the curve H-H based on the data in Fig. 17, and data on a number of experimental 18Ni-maraging steels.[29,151,153,157,161]

Because of the sharp drop in K_{Iscc} with increasing σ_y as well as the large scatter in these values, considerable care must be taken in the use of maraging steels for nuclear-reactor bolting materials. It would appear that samples from the same stock as the heat-treated material actually in use should be subjected to qualification SCC tests to evaluate its reliability in the reactor environment. In fact, the large scatter in virtually all the plotted data suggests that this procedure should be recommended for all steels.

4. DISCUSSION ON INFLUENCING FACTORS

The plotted data clearly show that a considerable spread in K_{Iscc} is obtained at any given σ_y for a given material or type of material. We will

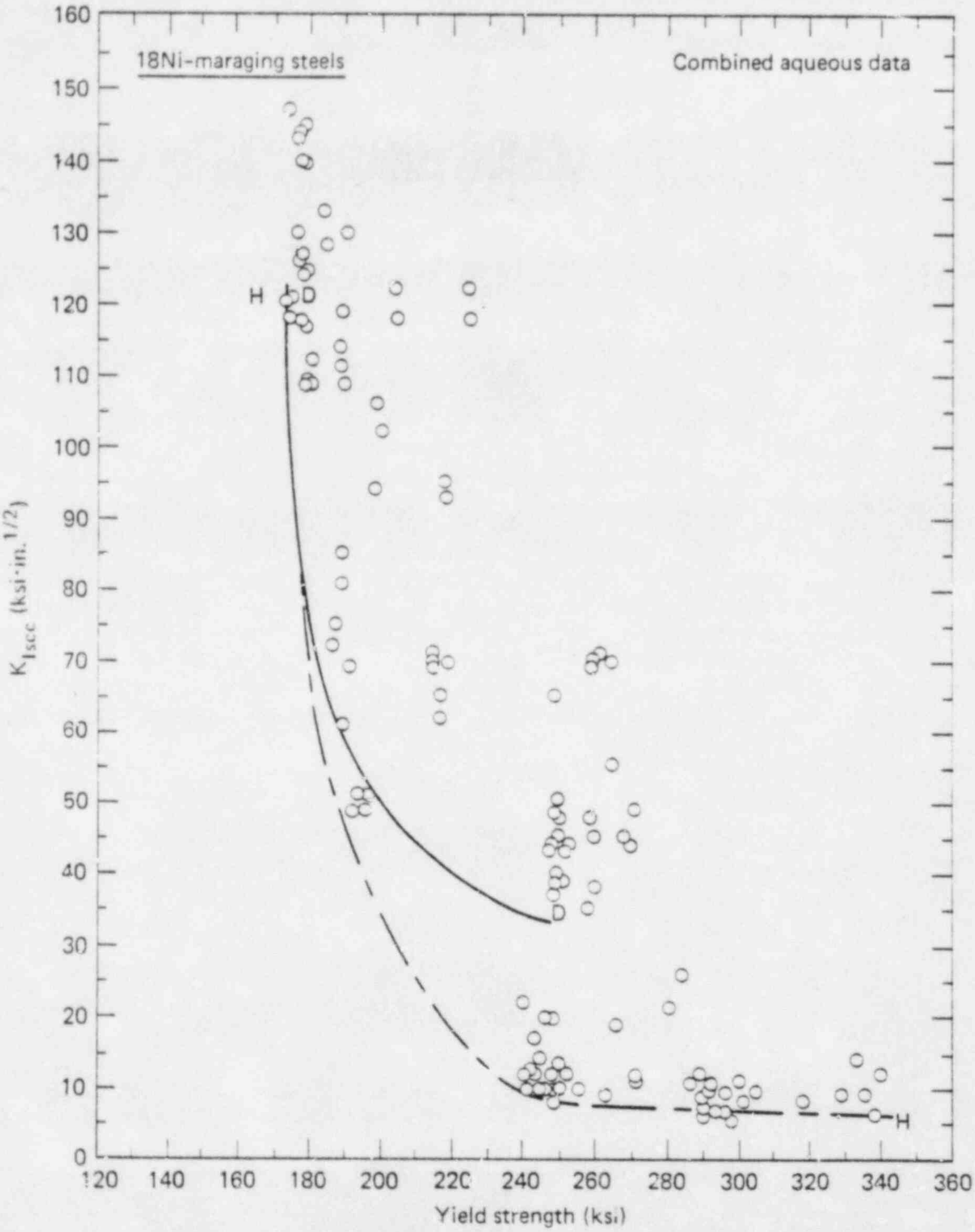

Figure 17. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for "combined" aqueous environments.

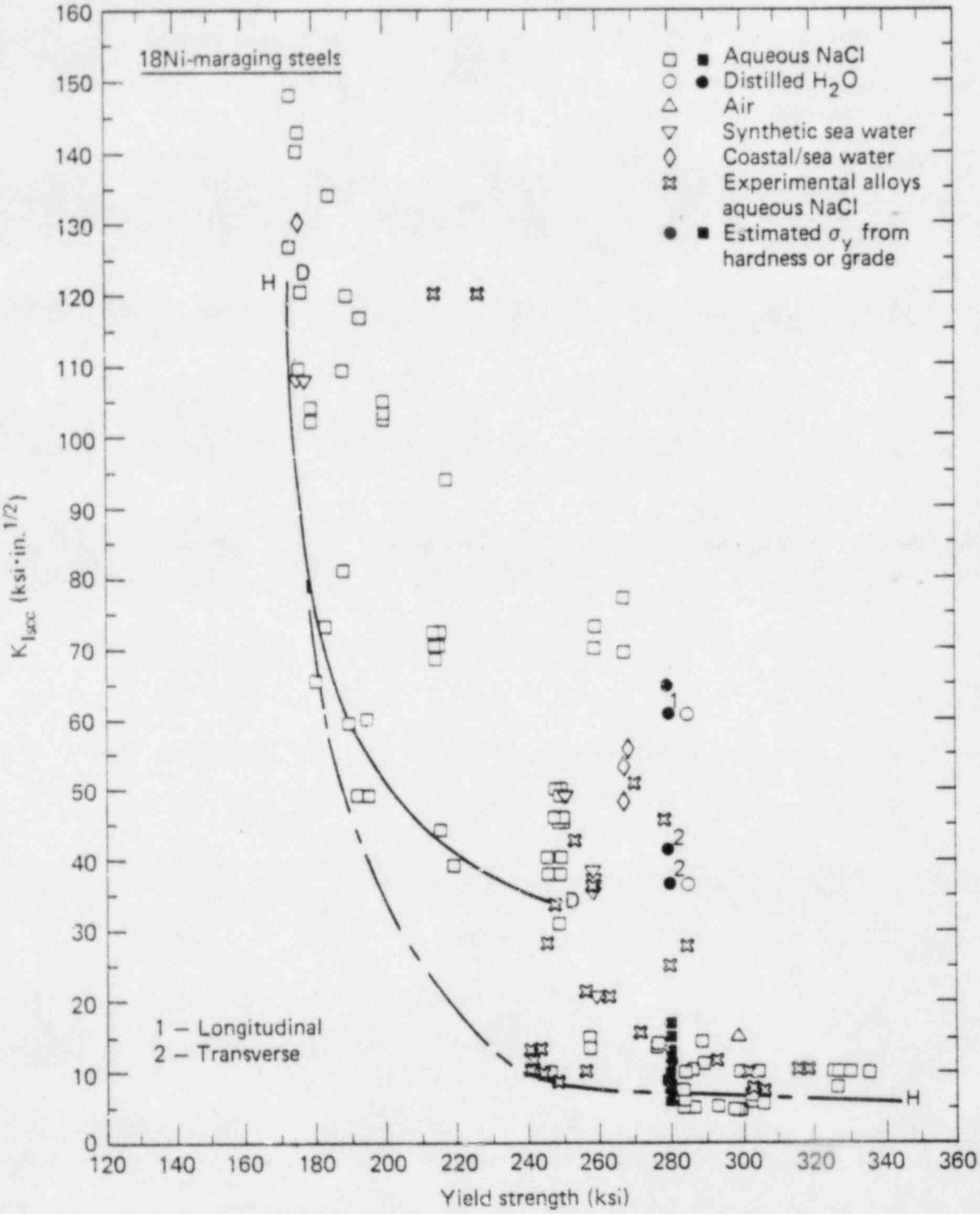

Figure 18. K_{Iscc} versus yield strength for 18Ni-maraging steels reported for water, air, and various aqueous chloride environments.

attempt here to examine the factors that may result in such large variations in the data. The effects of material variables associated with melting practices,[4,5,157,162] heat treatment and thermomechanical processing,[4-6,28,29,31,48,53,80,84,88,112,157,162-164] and composition[4-6,12,21,25,29,41,48,54,56,64,66,73,80,82,83,151,157,164], and environmental parameters involving pH,[4-6,48 88,157] polarization potential,[4-6,11,12,43,48,53,150,165-168] cathodic poisons,[4-6,53] and temperature[4-6,48,86,92,169,170] on SCC are discussed in a number of articles. (No attempt was made here to make a complete listing of such references). The effects related to material variables often are unclear and controversial; by contrast, the effects of environmental parameters are mostly well established. Several examples illustrating these comments will be given here. We will not discuss the effects of variations in specimen configurations and testing procedures. The reader is referred to several detailed papers on this subject.[47,51,93,103,171-174] In the discussions that follow, we will refer primarily to H_2O and aqueous chloride environments.

4.1: COMPOSITIONAL EFFECTS

Sandoz, in one of his review articles,[5] points out that the wide range in K_{Iscc} is a reflection of the many variables in melting practice, processing, fabrication, and heat treatment. The effect of uncertain variables in testing further widen this range. He further states[12] that there are no fundamental effects of composition on K_{Iscc}. However, above K_{Iscc} the CGR can be significantly influenced by composition. For a given K_{Iscc} and σ_y the CGR is lower[12] and time to failure longer[29] in the more highly alloyed high-strength steels. Improved melting practices, with better control of impurities, have resulted in significant increases in the K_{Ic} values, especially for the intermediate strength steels.[5] However, the corresponding effect on K_{Iscc}, although somewhat similar, is considerably less; in fact, factors such as composition and heat treatment that benefit K_{Ic} may adversely affect K_{Iscc}. Carter points out that for aqueous H_2S there is no apparent correlation between K_{Iscc} and K_{Ic}.[4] Waid and Ault studied a series of low- and medium-alloy steels and found that, although compositional variations affected K_{Ic}, the K_{Iscc} in aqueous NaCl was insensitive to these variations.[72] However, such compositional changes can have marked influences on CGR. A number of investigators have pointed out that variations in Si content do not affect K_{Iscc}; however, an increase in Si may markedly decrease the CGR.[4,41,54,72] This affect has been attributed to the retardation by Si in the replacement of ε-carbide with the high-temperature carbide during tempering.[41] The presence of retained austenite and ε-carbide in 4340 and 300 M steels was observed to reduce the CGR in aqueous NaCl without affecting K_{Iscc}.[54] For 35NCD16 (4Ni-2Cr-0.5Mo-0.35C), also tested in aqueous NaCl, retained austenite did not show any effect on K_{Iscc}, but it did reduce the CGR.[112] Carbide-forming elements have little affect, if any, on K_{Iscc}; but, their presence apparently increases the incubation time and time to fracture as evidenced by tests in distilled H_2O.[4] Reduction of P and S improves the resistance to crack growth, but have little effect on K_{Iscc}.[41,48] In maraging steels compositional effects are at most marginal.[4,151,157]

A reversal in the general observation of an increase in K_{Iscc} with alloying was attributed to the greater degree of twinned martensite in a 9Ni-4Co-0.45C steel as compared to that present in the low-alloy steels.[72] The twinned martensite is more succeptible to hydrogen attack than is the untwinned martensite.[48] Carter points out in his extensive review paper[4] that the effect of alloying elements are contradictory and the only consistant trend is that shown by carbon; i.e., an increase in C causes a decrease in the resistance to SCC. But, as pointed out below, an anomalous behavior is obtained for the highest carbon content. The trend with C content is consistent with the observation that low-carbon martensites are less susceptible to SCC and hydrogen attack than are high-carbon martensites.[5] In a plot of K_{Iscc} versus σ_y for aqueous NaCl, Carter[4] shows the data for 4130, 4330V, and 0.2C steels superimpose on each other; but, these data fall significantly above those for a 4340 steel plotted in the same figure.

In Fig. 19, the data published by Sandoz[25] show the effects of variations of several elements on the SCC of essentially 43XX-type steels in 3.5% NaCl. Carbon and Mn are the only two elements showing definite trends. There is a considerable drop in K_{Iscc} from 0.2 to 0.3C. This is inconsistant with similar results reported above by Carter[4] for of the 0.3 and 0.2 carbon contents, unless one considers the effects of alloying

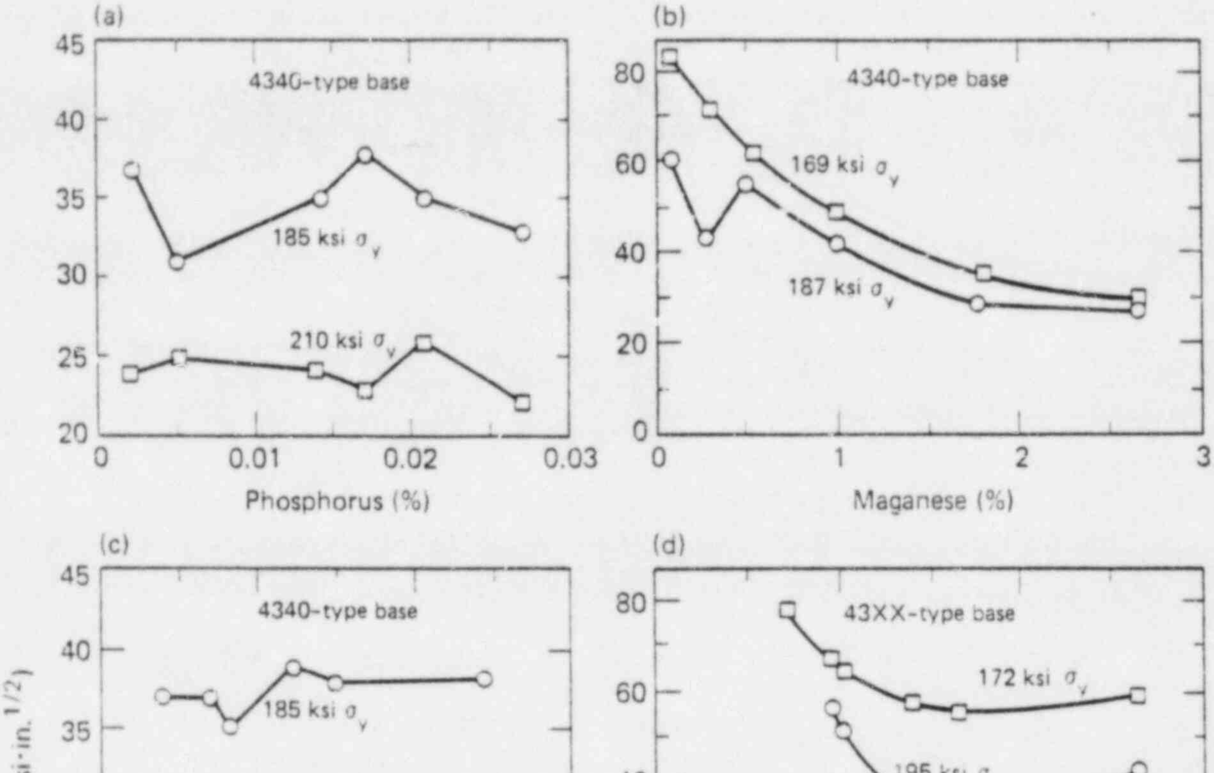

Figure 19. Effect of various impurities and alloying elements on K_{Iscc} for ASE 43XX-type steels.[25]

counteracting the carbon effect. However, the 4130 and 4330V steels also show identical results. It is suprising that K_{Iscc} should exhibit a minimum and then increase with carbon content above 0.35C. Furthermore, this carbon content corresponds approximately to where there is a transition from untwinned to the more hydrogen-susceptible twinned martensite. We believe that the two single values at 0.53C may be suspect or are affected by other compositional factors.

Carter[4] points out that the trend shown here for Mn in the 4340 steel is in conflict with the insensitivity of Mn content on SCC in the lower carbon steels. He further points out that the 4340 data may reflect a secondary effect, in that Mn lowers the $A_{1,3}$ temperature and, in tempering, this temperature may inadvertedly have been exceeded resulting in some reversion to austenite, especially if segregation is present. This would result in regions of untempered martensite causing a reduction in K_{Iscc}.[4] Alternatively, Sandoz[21] attributes the effect of C and Mn on K_{Iscc} to the crack tip changing from relatively anodic to cathodic with an increase in content in either of these two elements. Both explanations may be justified. It is clear that the effects of impurities and alloying elements on K_{Iscc} are not too well understood.

4.2: MICROSTRUCTURAL EFFECTS

Variations in heat treatment, prior deformation, and thermomechanical processing, all show effects on K_{Iscc} through the microstructures that are developed. An analysis of the review paper by Carter[4] suggests that there are fewer contradictions on the effects of heat treatment and microstructure, as well as of composition, for exposures in sulfide and hydrogen environments then there are for H_2O and aqueous chloride environments. For the low-alloy steels there appears to be little or no effect of austenite grain size on K_{Iscc}[4,5,6,29,31,48,53,88] but an increase in grain size increases CGR and decreases t_f. In maraging steels the role of grain size is not at all clear; annealing may cause other factors to enter, such as grain boundary precipitates and residual elements in solid solution. Furthermore, subsequent aging temperatures may cause opposite or dissimilar effects on K_{Ic}, K_{Iscc}, and CGR. Compared to a normal 500°C aging treatment, underaging may increase K_{Ic} and decrease K_{Iscc} with an expected corresponding increase in CGR. By contrast, overaging may increase both K_{Ic} and the resistance to SCC.[4,157] Other work has shown that aging temperature has little effect on the K_{Iscc} of maraging steels but can cause significant changes in CGR.[5,29] Excessively high annealing temperatures and poor melting practices will result in a loss in resistance to SCC, believed to be due largely to the precipitation of a Ti(C,N) grain boundary film.[157] Stavros and Paxton,[150] in comparing the properties of an 18Ni-300-grade maraging steel heat-treated to produce 2 different grain sizes (ASTM Nos. 0 and 9), found that there was no significant effect of either grain size or subsequent aging tratment on K_{Iscc}. Values all fell within the range of 10 to 15 ksi·in.$^{1/2}$. However, both the larger grain size, containing grain boundary precipitates, and the lower aging temperatures result in the shortest times to failure. All specimens were heat-treated and aged to the same hardness. It is likely that no grain size effect was seen because the data fall in the σ_y-insensitive region of the K_{Iscc} versus σ_y plots. More meaningful information on K_{Iscc} versus grain-size effects should be obtained for the lower strength maraging grades.

Large differences in K_{Iscc} between longitudinal and transverse orientations have been observed in banded structures.[28] In homogeneous structures there is usually little orientation effect.[4] In general, the proper combinations of deformation and thermal treatments should increase K_{Iscc}. Kerns, et al.[6] review work that shows improvements obtained by ausforming over conventional heat treatment in both K_{Ic} and in the resistance to SCC in distilled H_2O. Most bolting steels are used in the quenched-and-tempered condition. There is some controversy over the relative merits of tempered bainite versus tempered martensite and this may be due largely to environment. In the low-alloy steels the tempered-martensitic structures, relative to normalized-and-tempered or tempered-bainitic structures, appear to have the best resistance to SCC in aqueous sulfides.[80,162] In water and aqueous chlorides no significant effect is obtained for steels with $\sigma_y \geq 200$ ksi[48]. In a 3.5% NaCl environment a 4340 martensitic structure showed a 15-fold increase in CGR relative to that obtained for a lower-bainitic structure, both tempered to approximately 200 ksi σ_y level.[6] The difference in resistance to SCC for the two structures was attributed to more affective hydrogen trapping in the coherently twinned interfaces in the martensite as contrasted to the incoherent bainitic laths. The same argument should hold for the H_2S environment for here also the dominant mechanism of cracking is generally believed to be due to hydrogen attack.[4,6,48]

4.3: PRESTRESSING AND PRIOR DEFORMATION EFFECTS

Prestressing has been shown to increase the resistance to SCC[4,27,62,89,159,175] and this has been attributed to residual compressive stresses on unloading as well as blunting of the crack tip.[27,62,175] Prestressing has been shown to increase the incubation and failure times as well as K_{Iscc}. This points out the importance of not using excessive fatigue loads in precracking the test specimen.

By contrast to the effect of prestressing a precracked specimen, prior cold work (prestraining) before specimen preparation may reduce the resistance to SCC.[4,5,103,176] Novak[103] shows inconsistent patterns for the effect of small prestrains on K_{Iscc} for different materials tested in synthetic sea water. Inconsistant trends on prestraining to 5% were also obtained for the threshold stress in aqueous NaCl when comparing the results for HY80 and HY130 steels.[176] Cold work prior to heat treatments involving aging or ausforming results in an increase in K_{Iscc}.[5] Carter[4] states that prior cold work is clearly detrimental to SCC resistance in H_2S. The effects of prior cold work in saline solutions must be more clearly established.

4.4: ENVIRONMENTAL EFFECTS

Although testing in distilled H_2O and in aqueous NaCl may give the same K_{Iscc} values, the CGR may be significantly greater in the latter environment.[4] In comparing results from different environments used on two high-strength steels, McIntyre[115] found the same K_{Iscc} value of ~11 $ksi \cdot in.^{1/2}$ for exposures to H_2S gas, aqueous NaCl, and H_2 gas. But, the CGR decreased significantly in the same sequence. The importance of considering crack-growth rate as well as K_{Iscc} is strongly pointed out by these results.

Carter states that the CGR clearly increases with an increase in temperature, but the effect on K_{Iscc} is controversial.[4,48] Here it is probably related to the temperature sensitivity of pitting. Johnson and Willner show that K_{Iscc} for H-11 steel tested in water is independent of temperature over the range of about 5 to 60°C.[92] Charbonnier, <u>et al</u>.,[112] on testing 35NCD16 steel in aqueous NaCl, also found no effect on K_{Iscc} while the CGR increased with an increase in temperature (20 to 50°C). Simmons, <u>et al</u>.[170] studied the effect of temperature on cracking of 4340 in H_2 and distilled water. They observed that an increase in temperature increased the CGR (stage II) but that all the data merged in stage I. This suggests the independence of K_{Iscc} to both environment (H_2O and aqueous chlorides) and temperature. Carter[4] reports data for 4130 steel in H_2O over the range of 1 to 89°C that show an increase in CGR of nearly 3 orders of magnitude with an increase in temperature; again the data all merge in stage I. Landes and Wei[169] also show a strong temperature-dependence of CGR for 4340 in distilled H_2O. In contrast to these results, for H_2S solutions an increase in resistance to SCC was obtained with an increase in temperature.[4,48].

Carter and Hyatt[48] report that in the absence of complete saturation with H_2O vapor the CGR will decrease with an increase in temperature. Tests on the effect of relative humidity on H-11 steel show that there is a strong dependence of K_{Iscc} on RH. The K_{Iscc} decreases with an increase in RH up to almost 50% RH, and changes little thereafter up to 10% RH. The authors[92] propose that water vapor condenses at the crack tip for values > 50% RH. We suggest that the inverse effect of temperature on CGR is associated also with condensation, in that condensation at the crack tip will occur at higher values of RH as the temperature is raised. The CGR also increases with RH and levels off at somewhat above 50% RH.[92] Simmons, <u>et al</u>.[170] also ow a decrease in K_{Iscc} with an increase in RH. Studies on SAE 4340 w made at 0.1, 0.5, and 8 torr water-vapor pressure.

The effect of pH is reflected largely by the solution chemistry at the crack tip, where a pH of about 3 to 4 is reached early in the cracking process, relatively independent of the environmental pH.[13,16] Sandoz[5] in considering "salt waters", states that highly acidic solutions generally promote cracking and highly basic solutions generally retard cracking and usually there is little effect of pH on K_{Iscc} over the range of 3 to 10. Carter,[4,48] in reviewing the effects of pH on K_{Iscc} in water and aqueous chlorides, gives the insensitive range as pH 2 to 9. For notched specimens of 300-grade maraging steels Carter[4] lists this range as 0.5 to 11. For smooth specimens of the same steels there is an effect over this range which is associated with pitting. For aqueous sulfides the susceptibility to SCC is affected by pH over the entire acidic range, and the effect is further modified by the H_2S content.[48]

Impressed voltages appear to have little or no effect on K_{Iscc} (at least for low- and medium-alloy steels) but they have large effects on t_f and CGR,[4-6,43,53,157] especially in acidic environments.[4] Minimum in CGR usually occurs under slightly cathodic polarization potentials relative to the freely corroding potential[4-6] with the CGR increasing rapidly with further increase in negative potential. The accelerated crack growth under even moderate cathodic potentials attests to the role of hydrogen cracking in SCC. Where anodic dissolution predominates, exposure below K_{Iscc} can lead to crack blunting and therefore greatly increase crack initiation time. With insufficient test time this may lead to an apparent increase in K_{Iscc}.[167] A large drop in K_{Iscc}, from 103 to 24 ksi·in.$^{1/2}$, was reported for an 18Ni-200 grade maraging steel when cathodically polarized in a 3-1/2% NaCl solution.[168] Maximum test periods were $<$ 1000 h. Dautovich and Floreen in a review paper[157] on maraging steels also report similar examples of a large drop in K_{Iscc} for several different σ_y levels in the 200 and 250 grades. Thus, it may be that the effect of polarization on K_{Iscc} is different for maraging steels than it is for the low- and medium-alloy steels. Alternatively, the maraging steels may reflect insufficient exposure time under freely corroding potentials coupled with an increase in CGR under cathodic polarization. Novak reports that even 10,000 hours, based on his studies, may not be adequate for 250-grade maraging steel exposed to salt water.[177] In fact, the subject of adequate exposure time has received too little attention. Extrapolation of currently accepted K_{Iscc} data largely based on tests usually ranging from 100 h to about 1000 h may lead to erroneous conclusions of maximum allowable stresses for long exposure times of many years.

Kerns, et al.[6] and Carter and Hyatt[48] discuss the role of cathodic poisons in terms of group V and VI elements (P, As, Sb, S, Se, Te), hydride species, CN^-, and sulfur bearing compounds on restricting the recombination of hydrogen atoms to form H_2 gas and in promoting hydrogen entry. The effect of these poisons is to increase the CGR and possibly decrease, but to a much lesser extent, K_{Iscc}.[4-6,53]

As a final comment, on evaluating the integrity of bolting materials by the use of lower-bound K_{Iscc} curves, the many variables affecting K_{Iscc} must be included in any such analysis. Both material variables and environmental variables must be considered. Furthermore, the effects of any of these variables on CGR should be considered if stress transients occur that could result in exceeding the lower-bound K_{Iscc} values.

5. SUMMARY

The results of an analysis of an extensive literature survey on stress corrosion cracking (SCC) are presented in the form K_{Iscc} versus yield strength (σ_y) plots. The data are for a variety of steels typical of bolting materials. Environments include water, aqueous chlorides, aqueous sulfides, and H_2S and H_2 gases.

The plots of the reported data exhibit considerable scatter. This could be attributed to either material, environmental, or test variables and these possibilities are discussed. Variables such as grain size, composition, or polarization potential may affect K_{Iscc} and crack-growth rate (CGR) or time to failure (t_f) in dissimilar or even opposing ways. It is therefore suggested that an evaluation based on K_{Iscc} should include CGR and/or t_f where possibilities exist of exceeding K_{Iscc}. Uncertainties in published K_{Iscc} data based on too short exposure periods are also emphasized.

Lower-bound K_{Iscc} curves for the plain-carbon and low-alloy steels exposed to water and aqueous chloride environments are consistent with the proposed NRC lower-bound curve, while the corresponding curve for 18Ni-maraging steels falls below the curve proposed by the NRC, especially at the higher strength levels. For the precipitation-hardening stainless steels, at the lower strength levels the curves fall below the lower-bound curve suggested for the maraging steels but merge with the latter curve at stress levels above about 220 ksi σ_y. At high σ_y values it appears that K_{Iscc} becomes insensitive to variations in σ_y for all heat-treatable high-strength steels. The stress value at which this occurs is about 190 and 240 ksi for the low-alloy and high-alloy steels, respectively, with the lower-bound K_{Iscc} apparently levelling off at ~10 ksi·in.$^{1/2}$ for all the steels examined.

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16. ABSTRACT (200 words or less)
An extensive literature survey on stress-corrosion cracking of a variety of steels was made in response to the need by the U.S. Nuclear Regulatory Commission (NRC) to establish lower-bound K_{Iscc} values for bolting type materials. Materials evaluated include the heat-treatable plain-carbon and Cr-Mo and Cr-Mo-Ni low alloy steels, 17-4 PH and Custom 455 precipitation-hardening stainless steels, and the 18 Ni-maraging steels. An exhaustive survey was made for water, aqueous chloride, and aqueous sulfide environments. We also report limited data on H_2S and H_2 gases, aqueous H_3BO_4, and atmospheric environments. The data are presented in the form of K_{Iscc} versus yield strength. The proposed NRC lower-bound K_{Iscc} curve for the low-alloy steels is consistent with the data reported for the various aqueous environments, but excluding the sulfides. The corresponding lower-bound curve based on reported data for 18 Ni-maraging steels falls below that proposed by the NRC, especially at the high strength levels. The lower-bound curves for the precipitation-hardening stainless steels fall below the lower-bound curve for the maraging steels at the lower strength levels, but they merge at a yield strength of approximately 220 ksi. Reference is also made to crack-growth rate (CGR) and time to failure (t_f). These two parameters frequently were found to vary differently from the corresponding K_{Iscc} values when examined as a function of material or environmental variables. The influence of various material and environmental factors on K_{Iscc}, CGR, and t_f are discussed.

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