# Metallurgical, Stress and Fracture Mechanics Analyses

of

Cracked Steam Generator Manway Studs

from

Oconee Unit 3 of Duke Power Company

Report No. IE-123

April 17, 1981

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Prepared for: United States Nuclear Regulatory Commission Office of Inspection and E forcement

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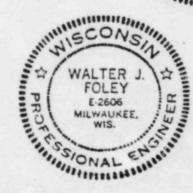
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by: L. H. Burck, PhD, P.E. Consultant to PARAMETER, Inc.

#### Exhibit A

Examination of Steam Generator Manway Studs from Oconee Unit 3 Reactor

> by: V. Pasupathi, D. R. Farmelo and E. O. Fromm, Battelle Columbus Laboratories

#### Attachment 1

Stress Analysis of Cracked Steam Generator Manway Studs from Oconee Unit 3 of Duke Power Company

> by: W. J. Foley Parameter, Inc.

#### Attachment 2

Fracture Mechanics Analysis of Manway Studs

> by: L. H. Burck, PhD, P.E. Consultant to PARAMETER, Inc.

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Metallurgical Analysis of Cracked Steam Generator Manway Studs from Oconce Unit 3 of Duke Power Company

Item 4 of

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#### I Abstract

2:1 An investigation was performed to determine the cause of cracking in two of nine cracked SA320, L-43 steam generator manway stud bolts removed from Oconee Unit 3 of Duke Power Company. In addition, one of the 55 uncracked stud bolts from the same unit was also examined. The investigation consisted of metallurgical tests conducted at Battelle Columbus Laboratories, under the direction of Parameter, Inc., and stress and fracture mechanics analyses performed by Parameter personnel. The results of the study show that cracking was a result of stress torrosion which was promoted by sulfur and chlorine contamination. The source of the sulfur is likely the partial decomposition of MoS, lubricant which was applied to the stud threads at the time of installation. The source of the chlorine contamination could not be associated with the lubricant or with any other substances known to have been applied to the stud bolts either before or after the cracking occurred. A contributing factor to the cracking was the configuration of the manway cover mounting which allows moisture to be trapped when the cover is sealed. The uncracked stud examined was found to be softer and to have a lower tensile strength than did the cracked studs although the strengths of all three studs were considerably above the minimum value of the material specification. The specific significance of the tensile strength levels in relation to the occurrance of cracking cannot be determined from the limited data available; however, studs with strength levels closer to the minimum value specified would be expected to exhibit improved stress corrosion resistance. A stress analysis of the studs indicates that the operating stress in the studs is within ASME code requirements. The critical defect size for stress corrosion crack growth is calculated to be 0.070 inches beyond the thread root and the critical crack size for final fracture is calculated to be 0.46 inches beyond the root, exclusive of any factors of safety. The material of the stud bolts examined was found not to be defective in either chemical composition or metallurgical structure.

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#### II Introduction

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This report presents the findings and conclusions of an investigation undertaken at the request of the Nuclear Regulatory Commission (NRC) to determine the cause of cracking in two steam generator manway cover stud bolts removed from Oconee Unit 3 of Duke Power Company. In addition, one uncracked manway stud bolt from the same unit was also examined.

Cracking in nine of the sixty-four stud bolts used to secure the upper and lower manway access covers to the steam generator was reportedly indicated by visual and ultrasonic examinations performed on June 25, 1980, during scheduled steam generator tubing maintenance. Three of the stud bolts, two cracked and one uncracked, were subsequently supplied to NRC for independent metallurgical analysis. The laboratory aspects of this analysis were conducted at Battelle Columbus Laboratories, Columbus, Ohio under the direction of carameter, Inc. Battelle's report of its findings is attached as Exhibit A. An evaluation of these findings is presented in Section IV of this report. In addition, stress and fracture mechanics analyses were performed by Parameter personnel and are presented as Attachments 1 and 2.

Oconee Unit 3 was manufactured by Babcock & Wilcox (B&W) and is located in Seneca, South Carolina. The stud bolts involved, which were reportedly supplied with the steam generator by B&W, are two inches in diameter with eight threads per inch. The material was specified as SA 320 grade L-43 low alloy steel which is equivalent in chemical composition to AISI 4340 steel. Sketches showing the general configuration of the manway access cover bolting are presented on Pages 5 and 6 of Attachment 1. The cracking experienced occurred in the non-engaged portions of the stud bolts which pass through the holes in the 5.5 inches thick manway covers.

Prior to being shipped to Battelle, and contrary to the request of NRC, the stud bolts were subjected to a number of solutions in an attempt at decontamination cleaning. Peportedly, the cleaning solutions included "M & S Germicidal Spray and Wipe Cleaner" (M&S Chemicals, Inc., Greenville, South Carolina), "Spotcheck Cleaner/Remover" (Magnaflux Corp., Chicago, Illinois) and a liquid soap solution

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of unknown manufacture. The application of these cleaners was particularly unfortunate in that the cracking was determined to have occurred by stress corrosion and thus the chemical composition of the deposits on the crack fracture surfaces was of utmost importance in detern ving the identity and source of the chemical agents which produced the cracking. Cleaning of the stud bolts not only removed a large portion of the surface deposits which were present, but may have introduced extraneous chemical species as well. In part, these problems were overcome by examining the deposits at the tips of small, corrosion-product-filled cracks which would have been less affected by the cleaning solutions, and by chemically analyzing the compositions of dried residues of the cleaning solutions themselves.

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Prior to installation, the stud bolts involved were reportedly sprayed with a commercial molybdenum disulfide ( $MoS_2$ ) lubricant in aerosol suspension, "Molykote G Rapid Spray" (Dow Corning Corp., Midland, Michigan). As a part of this investigation, special efforts were made to determine whether or not the cracks and corrosion pits contained sulfur which was not in the form of  $MoS_2$  as this compound has been reported (1) to react with water at elevated temperature to produce highly corrosive sulfur containing products such as  $H_2SO_4$ . In addition, the cracks and corrosion pits were also examined for other contaminants, and the stud bolts were fully characterized as to their metallurgical structure, chemical composition, and mechanical properties.

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#### III Conclusions and recommendations

### Conclusions

- Cracking occurred by an intergranular stress corrosion mechanism.
- Sulfur and chlorine, both of which promote stress corrosion cracking in the stud bolt alloy, were detected in crack cross-sections in sufficient quantities to have caused the cracking experienced.
- 3. The source of sulfur not present in the form of MoS<sub>2</sub> is likely the partial decomposition of the MoS<sub>2</sub> lubricant which was applied to the stud bolts at the time of installation.
- 4. The source of the chlorine found in the cracks could not be associated with any of the various substances known to have been applied to the stud bolts either before or after cracking. Therefore, it is concluded that chlorine contamination resulted from contact with an unknown chloride-containing material.
- 5. Moisture present at the time of the manway cover sealing would become entrapped in the region of non-engaged stud bolt threads where cracking was experienced and would contribute to the stress corrosion cracking process.
- 6. The tensile strengths of the stud bolts, particularly those of the cracked studs, were considerably higher than the minimum required by the SA320, grade L-43 specification to which the stud bolts were reportedly manufactured. However, although lowering the tensile strengths of stud bolts to values closer to the minimum specified would improve stress corrosion resistance, immunity should not be expected in view of the contaminants detected.
- The material of the stud bolts was not defective in either chemical composition or metallurgical microstructure.
- 8. The critical flaw size for stress corrosion crack growth was calculated to be 0.070 inches beyond the thread root. However, the application of a factor of safety to the stress intensity factor results in calculated crack dimensions which are substantially reduced from this value.

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9. The critical crack size for final fracture of the stud bolts is calculated to be 0.46 inches beyond the thread root, or greater, depending on the degree of load relaxation from prior crack growth and exclusive of a factor of safety.

#### Recommendations

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- Alternate lubricants should be investigated for this application.
- Procedures should be established and enforced to insure that stud bolts are not contaminated by unauthorized substances. In particular, these studs should not be exposed to chloride-containing materials.
- Care should be taken to insure that the studs and manway cover holes are dry and free of moisture when the cover is sealed.
- The remaining cracked and uncracked studs should be surveyed as to hardness level in order to determine if a correlation exists between hardness and incidence of cracking.
- 5. Excessive tensile strength levels in these stud bolts should be avoided.

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## IV Evaluation and Discussion of Results

The relevance and significance of the results of the various metallurgical tests conducted at Battelle Columbus Laboratories are discussed below. The details of the testing are presented in Battelle Report BCL-585-20 which is appended as Exhibit A. Also discussed below are the results of the stress and fracture mechanics analyses which were performed by Parameter, Inc. personnel.

#### Metallurgical Tests

The optical and scanning electron microscopy which was performed on opened crack fracture surfaces and on metallographically prepared longitudinal stud sections show that the stud cracking is a result of intergranular stress corrosion. Furthermore, electron microprobe and energy dispersive x-ray analyses of crack fracture surfaces and crack and corrosion pit crosssections revealed the presence of the elements sulfur and chlorine, both of which promote stress corrosion cracking in alloy steel such as that of the stud bolts (2).

The localized presence of sulfur and chlorine in surface corrosion pits and in surface-connected cracks is clearly shown by the x-ray image micrographs of Figures 18 through 21 of Exhibit A. It is significant that the detected molybdenum concentrations in the regions shown were much lower, in relation to the concentrations of sulfur, than would be expected if the sulfur were present in the form of MoS<sub>2</sub>. In this regard, it should be noted that the x-ray energies of molybdenum and sulfur are too close together to be resolved reliably by energy dispersive x-ray analysis in the scanning electron microscope. Thus, the element mappings shown in Figures 18 through 21 of Exhibit A were produced in an electron microprobe, which can unambiguously isolate each element of interest.

The excess sulfur detected in the cracks is present in quanities far greater than can be attributed to the concentrating of matrix sulfides by iron dissolution. Rather, the source of the excess sulfur is likely the result of a partial decomposition of the MoS<sub>2</sub> lubricant at elevated temperature in the presence of moisture as has been previously associated with the corrosive attack of various alloy steels (1).

Several attempts were made to identify the source of the chlorine detected in the stress corrosion cracks. For example, "Molykote" solid lubricant spray was chemically analyzed by energy dispersive

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x-ray analysis after being applied to a carbon substrate and allowed to dry. However, although the label of the product lists "chlorinated solvent" as a constituent, significant chlorine concentrations were not detected in the dried residue. The possibility of volatile chlorine-containing compounds in the "Molykote" being trapped and concentrated in tight cracks or crevices was also experimentally investigated and was found not to occur in laboratory tests. It was also suspected that the chlorine might have been introduced subsequent to cracking. However, testing of the reported decontamination solutions in a manner similar to that of the lubricant spray did not reveal significant chlorine concentrations. Furthermore, it should be noted that chlorine was detected at the very tips of tight cracks which were filled with corrosion product whereas it was not detected on external stud surfaces or on the surfaces of large, open cracks. This suggests that the chlorine actively participated in the stress corrosion cracking but was washed out of the larger cracks during decontamination. Thus, on the basis of the testing described above, it must be concluded that the stud bolts were contaminated by an unknown chlorine-containing substance prior to or during installation or that, possibly, such a substance was utilized after stud removal in addition to those reported. It should be emphasized that the stud bolt alloy is quite sensitive to chloride-induced stress corrosion cracking (2). Furthermore, prior or simultaneous exposure to sulfur and sulfide environments, as was the case for the stud bolts, is reported to further reduce resistance to chloride cracking (3) in addition to promoting stress corrosion cracking in and of itself.

The tensile tests which were performed on samples from each stud resulted in consistent values being measured for the two specimens from each stud, but with a relatively wide range of yield and tensile strengths occurring between stud bolts. Stud bolt Number 2 (uncracked) showed the minimum average yield streng a, 145 ksi, while the two cracked studs, Numbers 1 and 3, had average yield strengths of 164 ksi and 152 ksi, respectively. Similarly the tensile strength of the uncracked stud, Number 2, averaged 158 ksi, while those of the clacked studs, Numbers 1 and 3, averaged 177 ksi and 167 ksi, respectively. These tensile values were consistent with the as-received hardness measurements of 35.5 Rc for the uncracked stud, and hardness levels of 39.9 and 36.4 Rc for cracked stud Numbers 1 and 3, respectively. Furthermore, of the two cracked studs, Number 1, the stud with the higher tensile strength and hardness value, showed significantly more pronounced cracking. Thus, for the three stud bolts examined, the incidence of cracking followed the generally observed pattern of the harder, higher strength condition being more susceptible to stress corrosion cracking. However, because of the

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extremely limited number of samples which were available in this study, no general conclusions should be drawn as to hardness values or tensile strengths below a particular value being immune from stress corrosion cracking in this application. Nevertheless, it should be pointed out that the material specification to which the stud bolts were reportedly manufactured (SA320, grade L-43) requires a tensile strength of only 125 ksi (4), which is substantially lower, and thus less susceptible to stress corrosion cracking, than the strength levels measured. Thus, some benefit should be realized by reducing the tensile strengths of the studs to values closer to the minimum specified level, provided that the specification is appropriate in relation to other strength requirements. It is emphasized, however, that, even with reduced strength levels, stress corrosion cracking would still be quite possible with the levels of sulfur and chlorine contamination detected in the stud cracks examined.

Impact toughness values measured at 40°F reflect the tensile strengths and hardness levels discussed above with the uncracked stud, Number 2, showing the maximum toughness (44 ft. 1bs.), and with the cracked studs, Numbers 1 and 3, having impact toughness values of 21 ft. lbs. and 40 ft lbs., respectively. Thus, as would be expected, the measured toughness values displayed an inverse relationship to the tensile strengths and hardness levels. The toughness values measured are somewhat lower than literature values for AISI 4340 steel samples of the respective yield strengths (5). Examination of the fracture surfaces of the Charpy impact samples by scanning electron microscopy showed that fracture was by a mixed mode consisting primarily of void coalescence and quasi-cleavage with some intergranular fracture. The void coalescence dominated near the notches with the brittle modes being more prominent in the center regions of the fracture surfaces. No embrittlement was apparent from the tensile testing with reduction in area values for each stud surpassing the specification minimum of 50% (4).

Samples from each stud bolt were retempered at successively increasing temperatures in order to determine the initial effective tempering temperature and to detect any abnormalities in the material's temper response. The basis of this type of a test is that the tempered hardness of alloy steels is much more sensitive to the maximum temperature experienced than to the time at temperature. Thus, holding for one hour at a temperature less than that to which the steel had previously been subjected has little effect on the hardness while holding at a temperature greater than that of the initial tempering heat treatment will cause a reduction in hardness in accordance with the tempering behavior of the particular steel, data for which is available in the

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literature. In the particular case of the three stud bolts examined, the test results (Table 2 and Figure 23 of Exhibit A) indicate that the difference in initial hardness between stud Numbers 1 and 3 is due to stud Number 1 being tempered at a lower temperature then was Number 3. This conclusion is based on the virtually identical tempering response of the two samples at the higher tempering temperatures but the divergence in hardness for the as-received condition and after tempering at 950°F. The hardness levels of the sample from stud Number 2 were consistently lower than for stud Number 3 at all tempering temperatures. This hardness difference is attributed to minor variations in composition, microstructure, or quenching conditions. On the basis of comparisons of the as-received and re-tempered hardness values with literature data for the tempering of AISI 4340 steel (6), it is concluded that stud Numbers 2 and 3 were tempered at a temperature between 1000°F and 1100°F. Stud Number 1 appears to have been tempered at a somewhat lower temperature between 900°F and 1000°F.

Bulk chemical analyses of samples from each stud revealed chemical compositions which were within specification for the alloy (4). Furthermore, very little variation was observed between the compositions of the three stud bolts with the exception of the copper content which was substantially greater in stud Number 2, which was uncracked, than in the two cracked studs. However, this difference is not believed to be significant in relation to the cracking observed.

Optical metallography of polished and etched cross-sections from each stud bolt confirmed that the microstructures of the studs were tempered martensite, as would be expected. No significant differences in microstructure between the stud bolts were noted and no abnormalities were detected which would affect the materials resistance to stress corrosion cracking.

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### Stress and Fracture Mechanics Analyses

A stress analysis of the stud bolts (Attachment 1) indicates a total nominal operating stress of 33,700 psi, based on the root area. This stress, which includes the effects of both the initial pre-load and the operating pressure, is intensified by approximately a factor of three at the roots of the nonengaged threads, where the stress corrosion cracks initiated. The analysis also indicates that the operating stresses are within the ASME code-allowable values for the material utilized.

A fracture mechanics analysis (Attachment 2) indicates that stress corrosion cracks would grow from sharp flaws 0.070 inches deep in the thread roots. Such flaws might represent corrosion pits or intergranular attack on the thread surface and, in fact, stress corrosion cracks were observed to have initiated from such surface features. However, the application of a factor of safety to the stress intensity factor markedly reduces the calculated critical defect size for stress corrosion because of the highly non-linear relationship between stress intensity factor and defect depth. The critical cruck depth for final fracture is estimated as 0.46 inches beyond the thread roots, neglecting the effects of stud bolt load relaxation effects, and exclusive of a factor of safety. Load relaxation from prior stress corrosion cracking would increase the critical crack size for a particular stud, but could also accelerate stress corrosion cracking in adjacent studs because of load transfer.

It is significant that the cracking initiated in the non-engaged threads of the stud bolts even though the stresses would be higher at the first engaged thread at the nuts and at the threaded holes. This behavior is a reflection of the importance of the environment in producing stress corrosion cracking. In this regard, it should be noted that the configuration of the stud bolting, as shown by the sketches in Attachment 1, is such that any moisture which is present on the stud bolts or in the manway cover holes at the time of sealing the cover is effectively trapped. Such a situation would provide an environment conducive to stress corrosion cracking, particularly if the decomposition of MoS<sub>2</sub> were thus promoted.

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