

# Causes of Stress-Corrosion Cracking of Steel in Ammonia

Recent studies show that small amount of water is effective as inhibitor in ammonia service, and provide useful new information on the impact of contaminants as well as oxygen.

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New research results in studies of stress-corrosion cracking of steel in ammonia service have brought out a number of useful observations. Among them are the effectiveness of 0.2% water in the ammonia as an inhibitor, some new light on the level of contamination capable of causing cracking in metallurgical-grade ammonia, and refutation of the contentions that 1) oxygen inhibits cracking, and 2) this type of failure is caused by hydrogen embrittlement.

This subject has been recognized as a phenomenon and has been under study for nearly 20 years. The first recognition of the fact that ammonia could cause stress-corrosion cracking of steel came as a result of the development of cracks and leaks in carbon-steel vessels in agricultural-ammonia service.

In 1954, a committee was formed under the auspices of the Agricultural Ammonia Institute (AAI) (presently part of the Fertilizer Institute) to determine the cause of and methods for preventing cracking of vessels in agricultural ammonia service. (1) Research conducted by the committee culminated in 1962 with the publication of a technical paper (2) showing that air contamination in ammonia was the cause of cracking and that it could be prevented by the addition of 0.2% water to the ammonia as an inhibitor.

The paper also contained recommendations by the committee for preventing stress-corrosion cracking of steel in ammonia service. These were: (1) that agricultural ammonia should contain 0.2% water as an inhibitor; (2) that extreme care should be exercised to avoid air contamination in ammonia systems; and (3) that vessels over 36 in. in diameter should be either fully stress-relieved or fabricated with heads that are hot-formed or stress-relieved.

In 1961, also, as a result of this research, the AAI issued a specification for "Inhibited Agricultural Ammonia" which required that 0.2% water be added to conform with the specification. These recommendations were very effective in practically eliminating stress-corrosion cracking in carbon-steel tanks used in the agricultural ammonia industry. Reports of cracking in this industry dropped off after the recommendations were issued and are now, insofar as this writer is aware, non-existent.

From the mid-1950's until about 1967, the service performance of transport tanks made from high-strength

quenched and tempered (QT) alloy steels (present specification, ASTM A517) had been excellent; there were no reports of failures or even indications of cracking in the several thousand vessels in service transporting all types of ammonia during this period.

In 1967, however, it became apparent that stress corrosion was being encountered in ammonia transports manufactured from this type of steel (even though the tanks had been post-weld heat-treated as is customary with tanks made of this type of steel). Although no specific information is available, a reasonable explanation of the unanticipated occurrences of stress-corrosion cracking in the A517 steel tanks after 1967 may lie in a gradual increase in the purity of ammonia being transported.

In January, 1968, the United States Department of Transportation (DOT) adopted an amendment to the Hazardous Materials Regulations requiring that MC-330 and MC-331 cargo tanks constructed of quenched-and-tempered steel be used only for anhydrous ammonia having a minimum water content of 0.2 wt.-%, or for ammonia of at least 99.995% purity. A time-table was also set for internal inspection of all MC-330 and MC-331 cargo tanks made of quenched-and-tempered steel which had been in anhydrous ammonia or liquefied petroleum gas service. The regulation also required that any tanks going to ammonia service be cleaned of any previous product and that tanks be purged of air before loading.

In 1971, inspections revealed that stress-corrosion cracking in ammonia service was continuing to be encountered in truck transports made from A517 steels. Moreover, a review of European experience indicated that stress corrosion was being encountered in storage tanks made from non-quenched-and-tempered steels; and it was indicated that several European firms were supporting a research program on this subject at the Fulmer Research Institute in England. (3)

In December, 1971, the Department of Transportation held a meeting in Washington, D.C., to review all aspects of stress-corrosion cracking of steel in ammonia. It was pointed out that the frequency of the occurrence of cracking was about the same as it had been in 1967 and 1968 (20 to 25% of the vessels inspected had shown indications of

cracking), and industry was asked to furnish any information it might have as to how the applicable regulations might be changed to effectively prevent the further occurrence of cracking.

It was apparent at the meeting that there were differences in opinion as to why the regulations were not preventing cracking. In particular, F.J. Radd disagreed with the use of water as an inhibitor and further proposed that any previous evidence that water was effective in preventing stress corrosion was a result of oxygen present in the added water. Radd based his conclusions on a series of hydrogen permeation studies he had conducted with D.H. Oertle. (4) In contrast to Radd's position, this writer presented the results of long-term specimen exposure tests in ammonia tanks in service in Georgia, Texas, and Idaho that demonstrated the effectiveness of water as an inhibitor. (5)

During the meeting, a representative of the National Association of Corrosion Engineers (NACE) indicated that the organization would be willing to organize a committee to develop possible solutions to the problem. This offer was subsequently accepted by DOT; and NACE Committee T-5E-10 was formed. This committee held four meetings during 1972 and has developed recommendations for certain interim rule changes which have been forwarded to DOT. DOT is expected to initiate rule-making action based on these proposals in the near future. The specific changes proposed by the NACE Committee will be reviewed later in this paper.

This paper is intended to be an up-to-date status report on stress-corrosion cracking in ammonia cargo tanks. Although the information presented to the NACE T-5E-10 Committee has been used where the author feels that the information is appropriate, this paper is not intended to be a committee report.

#### New research results

Additional research studies have been conducted at U.S. Steel Corp.'s research laboratory to develop new information on the effectiveness of water as an inhibitor and also to consider some of the questions raised by Radd and Oertle. The results obtained were made available to the NACE Committee and are also contained in a paper by Deegan and Wilde. (6) These authors used a modification of the slow-strain testing technique described by Parkins. (7)

In this test a tension specimen is slowly strained (strain rate  $2 \times 10^{-6}$  in./in./sec.) while exposed to the environment of interest. Stress-corrosion crack growth markedly decreases the ductility of the specimen and also results in the formation of a number of secondary cracks near the main fracture. A non-aggressive environment, on the other hand, results in a cup-cone fracture with normal ductility.

The advantage of the slow-strain test is that it produces a direct and unambiguous indication that a metal-environment combination can result in stress-corrosion cracking. The test requires less than two days, whereas much longer times (days, months, or even years) might be required for stress-corrosion cracks to develop in service.

Because of skepticism regarding the effectiveness of water inhibition of stress-corrosion cracking in ammonia, a series of tests was conducted by Deegan and Wilde in which

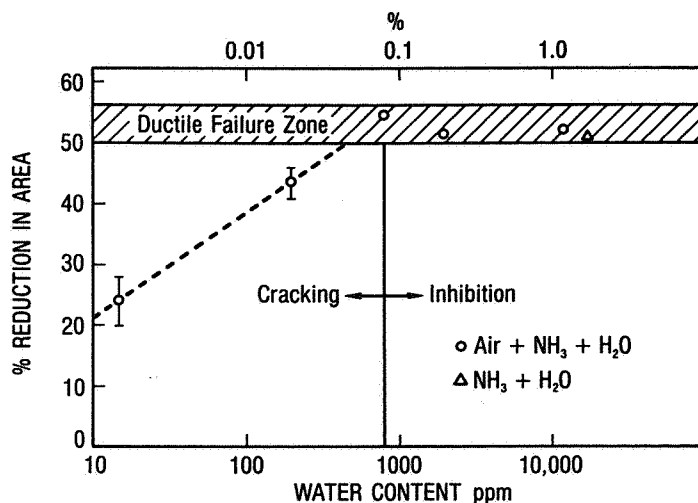


Figure 1. The influence of water content on the stress-corrosion cracking susceptibility of A517 Grade F steel in air-contaminated liquid ammonia.

water additions were made to air-contaminated ammonia (two atmospheres partial pressure of air). The results of the tests conducted with A517 Grade F steel are presented in Figure 1 for water additions over the range 0.02 to 1.2 wt.-%.

The data show that the previous findings (2) were correct: the addition of 0.2% water inhibits cracking of A517 steel in liquid ammonia. As the water level was decreased to 0.02%, a small but measurable decrease in ductility was noted, indicating the onset of stress-corrosion cracking. The results bracket the effective limits for water additions at 0.08% or greater and indicate that the previously recommended level of 0.2% is conservative.

As indicated, Radd and Oertle suggested that the effectiveness of water as an inhibitor is not due to the water *per se* but to dissolved oxygen in the water. To investigate this claim, Deegan and Wilde conducted tests in uncontaminated ammonia to which was added water containing only 20 to 30 ppb oxygen. No evidence of cracking was observed. The test was repeated but with air-contaminated ammonia to which the low-oxygen water was added. Once again no cracking was observed. Complete inhibition was also obtained by the addition of air-saturated water (approximately 8 ppm O<sub>2</sub> at 25°C). It is our opinion that these results refute the contention that it is the oxygen in the water that acts as an inhibitor.

Radd and Oertle also suggested that the phenomenon of stress-corrosion cracking of high-strength steels in liquid ammonia is due to hydrogen embrittlement caused by absorption of hydrogen into the steel as a result of corrosion. To investigate this possibility, Deegan and Wilde conducted a number of hydrogen permeation tests to clearly establish the conditions under which hydrogen absorption did indeed take place in air-contaminated ammonia. At the corrosion potential (zero applied current), no evidence of hydrogen absorption was observed. However, at 40  $\mu\text{A}/\text{cm}^2$  a readily detectable flux was observed with a classic hydrogen-permeation transient profile.

If hydrogen absorption were responsible for cracking under the freely corroding condition, then testing under conditions of known hydrogen permeation should lead to

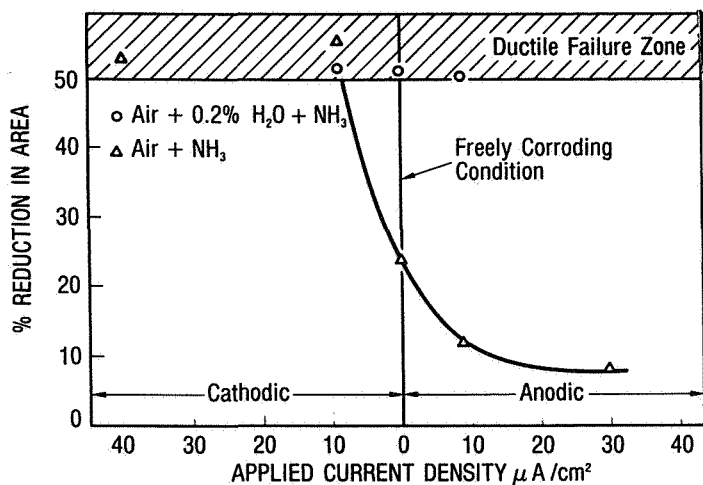


Figure 2. The influence of applied current on the stress-corrosion cracking susceptibility of A517 Grade F steel in air-contaminated liquid ammonia.

premature cracking. Stress-corrosion tests conducted in air-contaminated ammonia at a cathodic current density of  $40 \mu\text{A}/\text{cm}^2$  resulted in no stress-corrosion cracking and complete ductility, as shown in Figure 2. The overall influence of applied current density on the susceptibility of A517 Grade F steel to stress-corrosion cracking in liquid ammonia is shown also in Figure 2. At all cathodic currents, cracking was inhibited. At all anodic currents, cracking was stimulated, leading to the conclusion that the crack-propagation mechanism involves an anodic-dissolution step to the complete exclusion of a hydrogen-embrittlement contribution. It is of interest to note in Figure 2 that the addition of 0.2% water inhibited the stress-corrosion cracking in the anodic-current region as well as without applied anodic current.

In addition to the above studies, an investigation was conducted to determine the tendency to cause cracking of a number of samples of metallurgical grade ammonia from different sources. All these samples met the 99.995% purity definition in terms of water and oil content. The previously mentioned slow strain-rate stress-corrosion tests were also conducted with these samples and specimens of A517 Grade F steel. Also, a chromatographic analysis of the vapor phase above each sample was made to see whether there was any correlation between noncondensable gas content and cracking tendency.

The ammonia samples were taken from storage tanks by fitting a 1.5-liter, Type 304 stainless steel bottle, which had been purged with helium (until free of air contamination by chromatographic analysis) to the sample point; and evacuating all connecting lines and the bottle with a vacuum pump. Liquid ammonia was then admitted to the bottle after isolation from the vacuum pump. This resulted in filling the sample bottle approximately half full with liquid ammonia. Noncondensable gas analyses were conducted using a gas chromatographic procedure which could detect less than 4 ppm by weight of  $\text{H}_2$ ,  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{CH}_4$ , and  $\text{CO}$  in a gas sample.

The results of the slow strain-rate-stress-corrosion cracking tests are summarized in Table 1. Noncondensable gas analyses conducted on the vapor phase above the various

Table 1. Stress-corrosion cracking behavior of A517 Grade F steel in metallurgical-grade ammonia from different sources

Source Code	Result*
1	Severe SCC
2	Severe SCC
3	NC
4	SCC
5	SCC
6	NC
Control**	NC

NOTE: All samples were of 99.995% purity, based on water and oil content.

\*Severe SCC—Significant reduction in ductility and many secondary cracks.

SCC—Moderate reduction in ductility with some secondary cracks. NC—No reduction in ductility over that obtained in air (cup-cone failure).

\*\*Matheson Electronic Grade.

Table 2. Noncondensable gas analysis of vapor phase above metallurgical grade ammonia samples from various sources

Source Code	Vapor Composition (ppm-wt.)				CO
	$\text{H}_2$	$\text{O}_2$	$\text{N}_2$	$\text{CH}_4$	
1*	ND	ND	ND	ND	ND
2*	ND	14,200	15,350	37,200	ND
3	D	ND	162	ND	ND
4*	D	7.5	148	ND	8.3
5*	ND	190	495	ND	ND
6	D	ND	82.4	ND	ND
Control	ND	ND	ND	ND	ND

ND—Not detected.

D—Detected qualitatively.

\*—Indicates samples causing SCC (see Table 1).

\*\*—Matheson Electronic Grade.

ammonia samples are summarized in Table 2.

Stress-corrosion cracking was observed in four out of the six samples of metallurgical-grade ammonia investigated. With the exception of the ammonia sample designated Source Code 1, the incidence of cracking appeared to parallel the detection of oxygen in the vapor phase. It should be noted, however, that in addition to oxygen contamination, nitrogen was always present.

The fact that oxygen and nitrogen contamination can induce cracking was demonstrated by intentional contamination of the control sample (Matheson ammonia) with pure oxygen and pure nitrogen. In the uncontaminated condition, no cracking was observed whereas, after contamination severe cracking was noted. Addition of only oxygen or only nitrogen failed to produce cracking; both appear to be necessary to cause cracking. Moreover, the present results with oxygen and nitrogen contamination indicate that carbon dioxide contamination is not essential for cracking, as was previously considered. (2) With respect to the Source Code 1 sample, it is believed that quantities of oxygen and nitrogen below the limits of detection

(<4 ppm) were present and were sufficient to cause stress-corrosion cracking.

In considering the analyses in Table 2, it is noteworthy that the results shown are vapor-phase results. The partition coefficients for the noncondensable gases have been found by analysis to be on the order of several hundred to one; hence, the concentrations of the gases in the liquid phase are much lower than the levels shown in the table.

#### **Service experiences reported to the NACE Committee**

During the four NACE Committee meetings, a number of reports were made concerning service experience. A concerted effort had been made by the committee to obtain examples of cracking in tanks where it had been known that water was actually present in the ammonia. No such cases were reported. On the other hand, several reports were made to the committee which supported the effectiveness of water as an inhibitor. Specific instances of the occurrence of stress-corrosion cracking in cargo tanks that had been in dedicated metallurgical-grade ammonia service were also reported.

With respect to the performance of ammonia cargo tanks made from steels other than A517 steel, it was reported to the committee that stress-corrosion cracking has not been observed in tanks made of A202 Grade B steel (Cr-Mn-Si alloy steel customarily supplied in the as-rolled condition). This steel had commonly been used for ammonia and liquefied petroleum cargo tanks until the advent of the A517 higher strength steels, and many of the A202 tanks made in the 1950's are still in service.

Tanks made of this steel have apparently all been post-weld heat-treated either to utilize the maximum allowable joint efficiency factor (E) in design or in recognition of the ammonia stress-corrosion phenomenon. Although these tanks have not been subject to inspection by the magnetic particle method to anywhere near the extent that the A517 tanks have, nevertheless some inspections have been made in conjunction with repairs necessitated by accidents.

Inspection results have been negative for stress-corrosion cracking. Moreover, there are no reports of the development of slow leaks with vessels made of this type of steel; and it is known that some of these vessels have been used in dedicated metallurgical-grade ammonia service. Thus, there is at present no available service information indicating that this steel is unsuitable for transporting any type of ammonia.

It is of interest to note that unpublished results of tests conducted at U.S. Steel's research laboratory have shown that this steel, and other carbon and alloy steels as well, are susceptible to stress-corrosion cracking in the slow-strain test with contaminated ammonia. For these steels it appears that the test is more severe than actual service conditions and hence does not correlate with service performance. The reason for this anomaly is believed to be that the slow-strain test imposes a much higher stress on these steels than is actually present in the steel in a post-weld heat-treated vessel under load conditions.

The test does appear to correlate with the service performance of A517 steels, however. These steels, with

their higher content of alloy additions which impart elevated temperature strength, are known to have a higher level of residual stress after post-weld heat treating than is present in less highly alloyed steels, such as A202 Grade B. The A202 Grade B steel is also used at a lower design stress level than the A517 steel.

In view of the difference between the laboratory test results and service experience with the A202 steel, it is considered desirable that periodic inspections be made of cargo tanks made of this steel (and tanks made of other non-quenched-and-tempered steels as well) to confirm the service data presently available.

#### **Summary of NACE recommendations**

The substance of the NACE recommendation to DOT can best be summarized with the words of the Chairman, R.M. Hurd, in his covering letter.

"It is the unanimous conclusion of the committee that there is considerably less likelihood of obtaining cracks in the quenched and tempered tanks normally used in this service if 0.2% water is present as an inhibitor in the ammonia, than there is in using these tanks in 99.995% pure ammonia without the inhibitor. The committee cannot conclude that carrying "pure" ammonia will necessarily lead to cracking, rather that it is not possible at present to define "pure" ammonia and to insure that this required purity of ammonia is actually maintained in field service."

The changes proposed include increased testing and inspection requirements for cargo tanks in ammonia service, the requirement that all cargo tanks for ammonia service be post-weld heat-treated, the restriction of the use of A517 steels to water inhibited ammonia, and requirements for periodic analyses of inhibited ammonia for water content.

The specific changes are paraphrased as follows:

1. That a wet fluorescent magnetic particle inspection be conducted in conjunction with and just prior to the hydrostatic retest required every five years. This inspection procedure is required for tanks made of any type of steel.

2. MC-330 and MC-331 cargo tanks constructed of other than quenched-and-tempered steel are authorized for all grades of ammonia. MC-330 and MC-331 cargo tanks constructed of quenched-and-tempered (A517) steels are authorized for ammonia having a minimum water content of 0.2 wt.-%.

3. Any water additions must be made by using de-ionized or distilled water or steam condensate.

4. For inhibited ammonia, a periodic analysis must be performed for prescribed water content. Such analyses must be performed at least weekly in cases where the ammonia in a storage tank contains 0.2% water. In those cases where the water is added at the time of loading, provisions must be made to assure that the water injection equipment is operating and at least one load in each 10 loads or one load in every 24-hr. period, whichever is less frequent, must be analyzed for prescribed water content.

5. All cargo tanks for ammonia service must be post-weld heat-treated.

#### **Other factors**

It is essential to recognize that mechanisms other than

stress-corrosion cracking caused by ammonia can lead to service-induced cracking of MC-330 and MC-331 cargo tanks made of any type of steel. For example, any cargo contaminated with hydrogen sulfide (or hydrogen sulfide itself as a cargo) can cause sulfide-induced stress-corrosion cracking of A517 steels. For this reason, Note 15 of paragraph 173.315 of the applicable DOT regulations requires that:

“Only grades of liquefied petroleum gases determined to be ‘non-corrosive’ are authorized in specs MC-330 and MC-331 cargo tanks constructed of quenched and tempered steel (QT). ‘Non-corrosive means the corrosiveness of the gas does not exceed the limitation of classification 1 of the ASTM copper strip test classification when tested in accordance with ASTM D1838-64 Copper Strip Corrosion by Liquefied Petroleum (LP) Gases.’ ”

Liquefied petroleum gases containing any significant amount of hydrogen sulfide will not pass this test. Hence, if this regulation is followed, sulfide corrosion cracking in LP gas service can be avoided.

Fatigue is another significant factor that can cause cracking of MC-330 and MC-331 cargo tanks, especially those in which the cargo tanks constitute in whole or in part the stress member used in lieu of a frame. For this type construction the applicable DOT regulation (paragraph 178.337-13) requires that:

“The design calculations shall include beam stress, shear stress, torsion stress, bending moment, and acceleration stress for the cargo tank as a whole, using a factor of safety of four, based on the ultimate tensile strength of the material. Maximum concentrated stresses which might be created at pads and cradles due to shear, bending, and torsion shall also be calculated in accordance with Appendix G of the ASME Code 1962 edition. Fully loaded vehicles shall be assumed to be operating under highway conditions equal to 2 “g” loading. The effects of fatigue shall be taken into consideration.”

Inasmuch as different mechanisms may cause service cracking in cargo tanks, it is important to make every effort to establish the cause of the cracking when cracks are found during inspection of a vessel. Establishment of the cause can be very difficult if only a visual examination of the surface can be made. Whenever possible, a section of the affected area should be removed for metallographic examination.

The following guidelines are suggested for distinguishing between different types of cracks. (8)

#### 1. Visual Surface Appearance

a) Stress-corrosion cracking usually occurs in multiples or “families”. Fatigue (or corrosion fatigue) may cause some multiple cracking, but in general it will not be anywhere near as extensive as in stress-corrosion cracking.

b) Stress-corrosion cracking occurs predominantly as a result of residual stress. Fatigue cracks, on the other hand, are typically a result of “live” loads. Either or both types of cracking may be apparent at attachments or fittings or in any welded area and will be most likely to develop around support pads and stiffeners.

c) Fatigue cracks are transgranular and usually exhibit very little, if any, branching.

#### 2. Metallographic Examination

a) Stress-corrosion cracking caused either by contaminated ammonia or hydrogen sulfide may progress along either transgranular or intergranular paths through the metal. A transgranular mode of propagation is more common in base metal, however, for cracking from either cause.

b) Some degree of branching is usually, but not always, observed in stress-corrosion cracking.

c) To distinguish between sulfide corrosion cracking and ammonia cracking, an attempt should be made to determine whether a substantial amount of sulfur is present in the corrosion product inside the cracks. This can be done with an electron-probe micro-analyzer. A metallographic technique known as sulfur printing is also available.

#### Summary

1. The presence of 0.2% water in ammonia prevents stress-corrosion cracking of steel in ammonia service. Cargo tanks made of A517 steel should only be used to transport ammonia with 0.2% water added as an inhibitor.

2. It is not possible at present to define a “pure” ammonia that will not cause cracking of A517 cargo tanks and it is furthermore not possible at present to specify methods to maintain purity in field service. The present interpretation of 99.995% purity in terms of water and oil content is inadequate.

3. All vessels made of carbon or alloy steels for ammonia service should be post-weld heat-treated. This includes stationary vessels of any type as well as over-the-road tankers.

4. Stress-corrosion cracking in ammonia is caused by the presence of contaminants in the ammonia. Oxygen and nitrogen in combination have been identified as agents that can cause cracking. The presence of carbon dioxide is not necessary but may aggravate cracking. With the very low water content routinely present in modern-day ammonia, extremely small concentrations of contaminants can cause cracking.

5. Vessels made of any type of steel in ammonia service should be periodically inspected using the wet-fluorescent magnetic particle method.

6. The studies discussed herein refute the contentions 1) that oxygen inhibits cracking, and 2) that this type of failure is caused by hydrogen embrittlement.

#### Acknowledgment

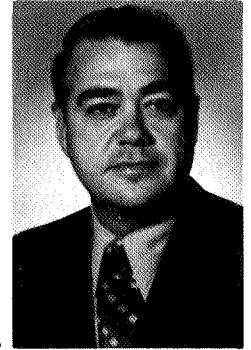
The U.S. Steel research studies reported in this paper were conducted by B.E. Wilde and D.C. Deegan. Their contribution to this work is acknowledged along with that of A.W. Loginow, with whom the author has worked on this problem since the original AAI research studies. #

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PHELPS, E. H.

## DISCUSSION

**DAVID FYFE**, ICI Billingham, England: I've been associated with the Fulmer Research Project for ICI. I'd first like to make a point, then to ask you a question. We would agree that there is absolutely no evidence of cracking at minus 33 degrees Centigrade, but there is one European company who inspected a large 10,000 tons minus 33° atmosphere pressure storage tank and found absolutely no cracking in that tank, but in European service we tend to have a recondensation of the boiled off vapor, and there was a small tank on top of the main tank which collected the recondensed vapor. When this was inspected it was found to be riddled with stress corrosion cracks.

There was a crack roughly every centimeter along most of the welds, so bad that the vessel had to be scrapped. So one should not assume that the whole system is immune, even though the main storage tank may be.

**PHELPS**: Yes, that's a good point. We have also seen stress-corrosion cracking in steel piping transmitting boiled-off vapor from the top of refrigerated storage tanks.

**FYFE**: The question I'd like to ask you is, you imply but don't quite say that nitrogen is as important or is also important vis-a-vis oxygen?

**PHELPS**: Our results indicate that the presence of nitrogen is as important as oxygen in causing stress-corrosion cracking. Nitrogen alone does not cause cracking. We don't understand this result and have not as yet established how nitrogen participates in the stress-corrosion mechanism.

**FYFE**: So that means that the amount of free nitrogen which is in solution from the ammonia equilibrium is not enough by itself. You say there must be nitrogen there from air.

**PHELPS**: I don't know how much free nitrogen is present as a result of the ammonia equilibrium reaction.

**FYFE**: It would be very easy to prove.

**PHELPS**: In our controlled experiments with Matheson ammonia, we had to add both oxygen and nitrogen to cause stress-corrosion cracking. Introduction of oxygen alone did not cause cracking. This indicates that the amount of nitrogen originally present is insufficient and that some has to be added.

**Q**. Can you explain to me? I don't quite understand this, how 0.2 percent of water is effective in the vapor phase? This appears to be the case, I take it, but does this mean that in the vapor phase one has a sufficient presence of water vapor as a consequence?

**PHELPS**: Our view is that there is a partition of water between the liquid and vapor phase and the amount in the vapor is sufficient to inhibit cracking.

**Q**. Did you try oiling any of your samples, because the old-fashioned way of making ammonia involved recipro-

cators and things, and usually there was a lot more oil in the ammonia, and an oil coating would be present on storage vessels and the like?

**PHELPS**: We did not oil any of our samples. From the standpoint of service experience, stress-corrosion cracking has been encountered in vessels heavily contaminated with oil and also in vessels that showed no residual oil film. On balance it appears that the presence of oil is not a factor in stress-corrosion cracking.

**W.D. CLARK**, ICI, England: I would like to make some reference to the Fulmer Institute research programme on this subject which was described to this conference two years ago with an invitation to American companies to join the European companies sponsoring it. Unfortunately, no American company or organisation has joined in, and as a result I cannot give any details of the results obtained at Fulmer as the sponsors have not all agreed to this.

The work is however progressing well, and is concerned with ordinary constructional steels rather than high strength steels such as T1. It was reported that there has been some trouble in Europe with storage tanks, etc. Fulmer are now in a position to draw lines on graphs of H<sub>2</sub>O-O<sub>2</sub>-CO<sub>2</sub> content separating safe from dangerous areas. They have not considered nitrogen, which is rather an unwelcome shock to them, as it is to me and to Dr. Phelps.

One of the questions which is now arising from the results of the work is that unsafe conditions seem so significantly remote from the analyses of the ammonia that we think we have in our tanks, that it is surprising that there has been cracking in service. We have been analysing samples of ammonia from various sources to see whether oxygen contents can in fact run higher, water contents lower, etc. than we would expect, because of e.g., inefficient purging. Can Dr. Phelps tell us if similar analytical checks are being done in the U.S.A. to find whether the ammonia in your tanks always has the intended composition?

**PHELPS**: Yes, as part of this work we have analyzed a number of different ladings and find that air contamination is quite often present. This occurs both in metallurgical-grade ammonia and in water-inhibited ammonia. The contamination appears to be transmitted by the return lines that are used in transferring ammonia. For example, if a transport tank containing uncontaminated ammonia is used to fill a tank the contamination can enter the transport tank through the return line. This contamination in turn is transmitted to the shippers tank by the return line when the transport tank is refilled. In this way air contamination can be spread throughout a system.

With respect to ordinary strength steels, the service

experience in this country has been that these steels are only subject to stress-corrosion cracking in ammonia service when they are used in the non-post-weld heat-treated condition. To the best of our knowledge, there have not been any reports of stress-corrosion cracking of post-weld heat-treated tanks or vessels made of these steels. As I mentioned in the paper, these steels can be subject to stress-corrosion cracking in the slow-strain test probably as a result of the fact that in the test the steel is subjected to stresses that are much higher than those encountered under

load constructions.

**BARNEY O. STROM, CF Industries:** We are building two Horton spheres in India, of all places, and we are using A516 grade 60 material, impact specification for  $-33^{\circ}\text{C}$ . We are not stress relieved. Is there any danger in storing ammonia in it at something like 50 pounds? This is a fine grain structure.

**PHELPS:** Service experience in the United States has been that stress-corrosion cracking is not a problem in this type of low-temperature storage.