Ammonia Plant Converter Basket Failure

Investigation showed outside insulation had made possible a chloride concentration that, with existing temperature, results in stress corrosion conditions.

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The objective of this article is to report on the circumstances surrounding a catalyst basket failure at the Agrico Chemical Co.'s Faustina plant 1,000-ton/day Kellogg process ammonia unit.

Details of the failure, repair, and replacement are discussed with the hopes that the information will allow others to make better management decisions with regard to design of vessels, methods of hydrotesting, and operating environment of process equipment.

The discussion is limited to the facts surrounding the basket failure and the conclusions drawn by investigation at the time of the failure. Although there will be discussion of chloride stress corrosion in stainless steel, this is not intended to be the mechanism of stress corrosion cracking. Many of the conclusions reached depend on the generally accepted theory that austenitic stainless steel subjected to stress at temperatures above 120°F in the presence of high concentrations of chlorides are subject to stress corrosion cracking.

The greatest limitations posed for the presentation of information concerning the failure is the length of time since the failure occurred. Often records are found to be incomplete or contradictory.

The converter basket was fabricated from Type 304 stainless steel and insulated with a 1-in. layer of spun rock wool covered by a sheath of stainless steel. After fabrication, the basket was installed in the shell, and the shell was then hydrotested using city water with an analyzed chlorides content of 10 parts/million.

Following each hydrotest, the basket was dried using a burner to heat the inner shell of the basket ot 300°F for 14 hr. After completion of the second hydrotest, the assembly was shipped to the construction site.

In mid-September, 1968, the catalyst reduction began. After several startup problems were resolved, the plant was brought up to about 90% rates, 90% being the operating limit in the plant for several months due to problems in the front end.

During the months that followed startup, it was noted that the temperature of the gas leaving the annular space between the converter basket and the pressure shell was above design and gradually increasing with no corresponding increase on the inlet or on the interior of the basket. Figure 1 shows the trend. The conclusion was drawn that there was a leak between the hot gasses in the beds and the annular space.

By February, 1970, the temperature had stabilized but several hot spots had appeared with shell temperatures in excess of 420°F. These hot spots caused a great deal of concern because Kellogg design temperature for the pressure shell is 400°F. The fabricator of the shell was contacted and he assured plant personnel that the vessel was designed for 600°F for structural and mechanical properties. The prime concern then became whether the hot gasses impinging on the shell of the converter could cause hydrogen attack of the pressure shell.

The pressure shell was a laminated vessel with an inner liner of 0.18 vanadium steel to resist hydrogen attack.

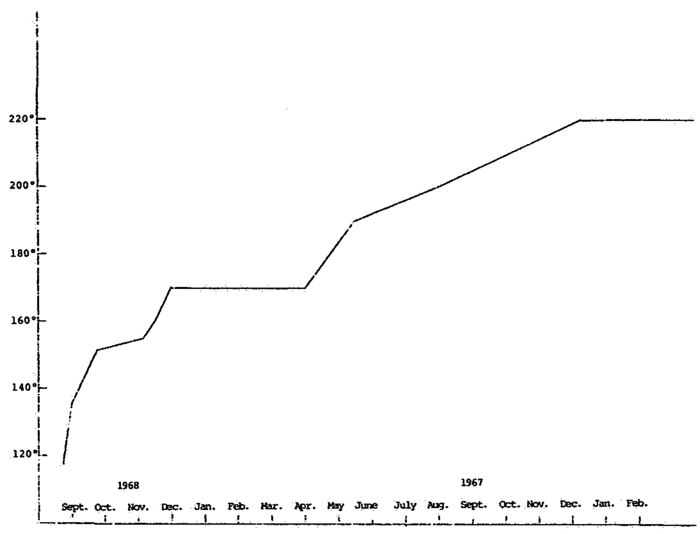


Figure 1. Temperature of recycle to syngas compressor vs. time.

In the center of the hottest point, a hole was drilled identical to the weep holes in the shell. A thermocouple was inserted in the hole, and the outside temperature of the inner shell was measured at 492°F. From the curves developed by G.A. Nelson, it was determined that the inner shell could withstand temperatures of 575°F without suffering hydrogen attack.[1]

The decision was then made to continue operations until a scheduled turnaround in June, 1970. The converter was shut down for repairs on June 14, 1970. Since the inspection and repair of the converter basket was new to everyone involved, the decision was made to make a video tape of the inspection and to have television monitors in the vessel during the repair. This TV capability would increase safety and allow observation of the job progress without vessel entry which would interrupt repairs.

After the catalyst was dumped, the converter basket was filled with demineralized water and each basekt was clenaed out. As soon as the vessel was safe for cutting, samples of the ¾-in. basket were removed and sent to Shilstone Testing Laboratory for examination. Visual inspection showed extensive cracking of the shell with the cracking more severe in the fourth bed. The cracking varied from none in the first bed to over 90 ft. in the third and fourth catalyst beds. The 90% of the cracking was on the south side of the basket.

Two plates, seen in Figures 2 and 3, were tested extensively in the Shilstone Lab with the following conclusions:

1. The plates showed nitirding to a depth of 0.001 to 0.0097 in., with the depth of the nitriding being the same inside the cracks as on the surface. This indicates the cracks were there before or during startup.
2. The primary mode of cracking was noted to have been by branching transgranular penetration from external surfaces.
3. The nitriding was not a factor in the failure.

Figure 2. Inside surface of the 7-in. sample, as received by the test laboratory.

Photomicrographs shown in Figures 4, 5, 6, 7, and 8 illustrate the results of testing.

After results of the inspection were evaluated, it was decided to repair the basket by grinding out all of the cracks, welding them up and then covering with a ¼-in. 321 stainless steel plate. This resulted in essentially all of the fourth bed being covered by ¼-in. plate. This repair was regarded as temporary, with hopes of it lasting until new basket could be fabricated.

After repairs had been made, it was later hypothesized that the studs which had been welded onto the shell might have been responsible. On investigation of the previous samples taken, this was shown not to be the case.

Since the cracking appeared to be a result of chloride stress

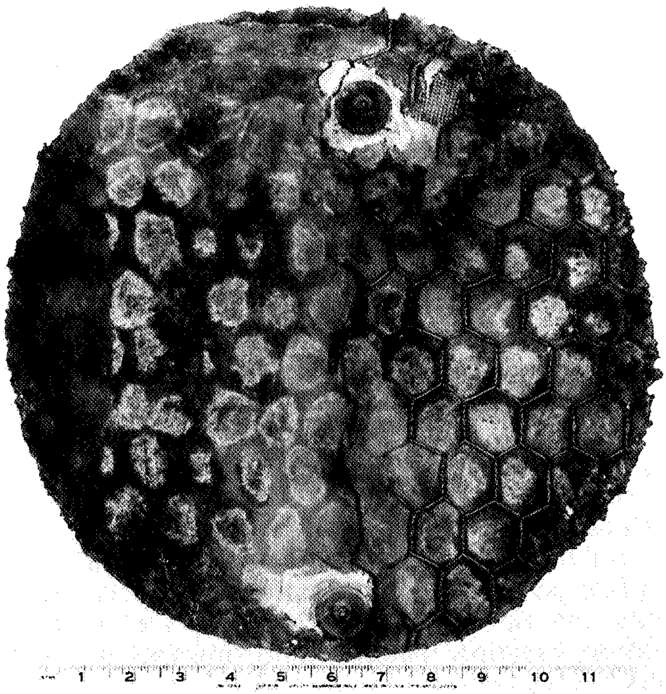


Figure 3. Outside surface of the 12-in. sample, as recieved by test laboratory.

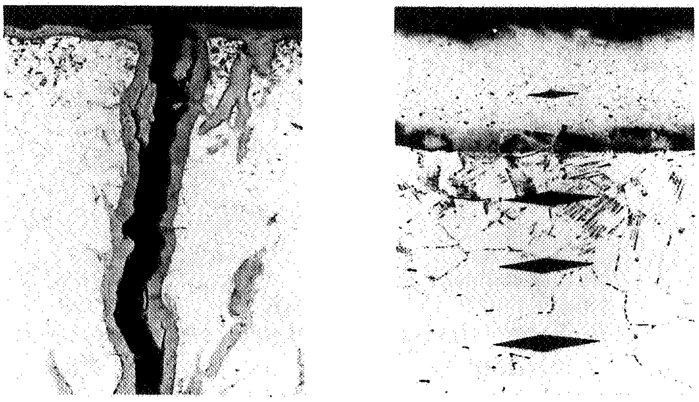


Figure 4. Photomicrographs illustrate nitriding at the outer surfaces and along the surfaces of cracks. Knoop microhardness impressions are visible in the phomicrograph at the right, a 12-in. sample at 200X. The one on the left is a 7-in. sample at 50X. (Oxalic acid, electrolytic etch.)

corrosion cracking, the source of chlorides became the subject of a detailed investigation.

A review of the hydrotest of the complete vessel showed that the complete converter had been hydrotested twice. During the first hydrotest, several superficial cracks were found on one of the nozzles. After these cracks were ground out, the vessel was hydrotested again. The water used for hydrotesting was city water with chloride content of 9 to 10 parts/million. After each hydrotest, the vessel was dried using a gas burner and heating to 300°F until the insulation was dry, which was about 14 hr. duration.

Construction and shipping photographs indicate that the side of the vessel which was predominately cracked was down during hydrotesting and shipping.

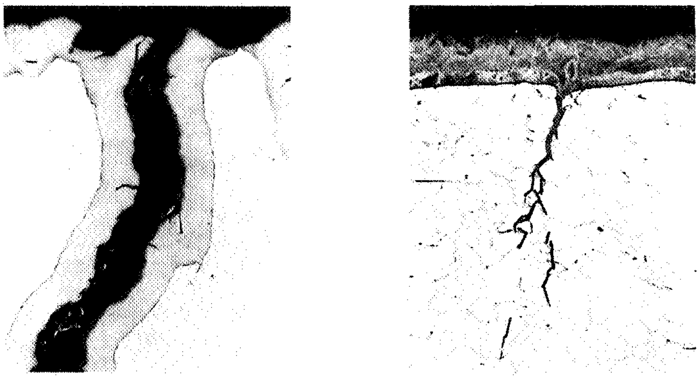


Figure 5. Photomicrographs of a section of the 12-in. sample, illustrating nitirding at the outer surface and along cracks. The one at the right shows nitriding along a transgranular crack extending inwardly from the outer surface. Note that the degree of nitriding along the crack is less than at the surface: this is the only instance in which nitriding was not of similar depth along crack surfaces to that at plate surfaces, and therefore probably represents the tip of a larger crack extending from another location. (Oxalic acid, electrolytic etch. 100X.)

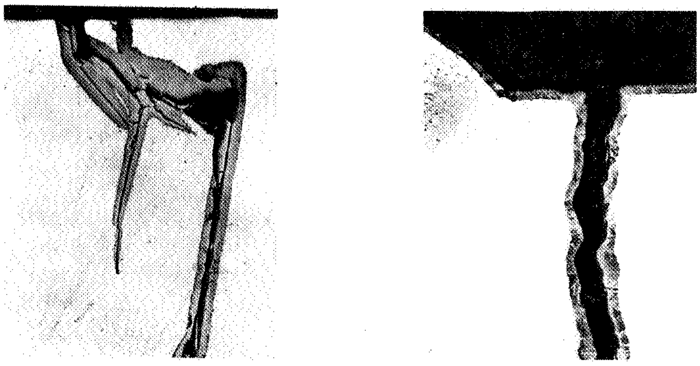


Figure 6. Photomicrographs of sections of the weld in the 7-in. sample, illustrating nitriding of the external surface and crack surfaces. The nitrided layer on the external surface in the view at the left (a longitudinal section of the weld) is absent, probably because of flaking or spalling during sectioning and preparation. The view at the right is a transverse section of weld and adjacent plates. (Oxalic acid, electrolytic etch. 50X)

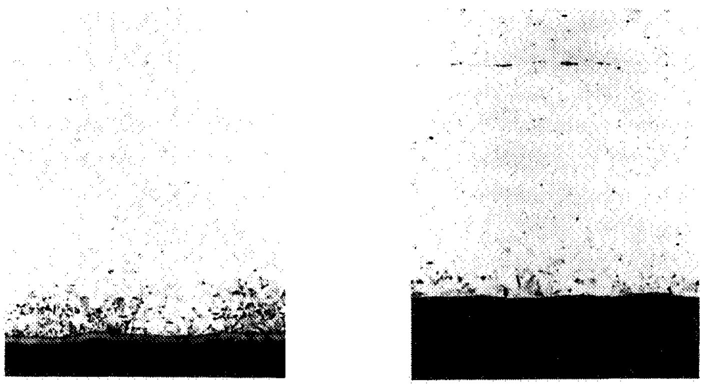


Figure 7. Photomicrographs illustrate nitriding of the inner surfaces of the two plates. Note that the degree of nitriding is less in the 7-in. plate (at left) than in the 12-in. plate (at right). (Oxalic acid, electrolytic etch. 50X.)

Figure 8. A photomacrograph of the inner portion of a longitudinal section of the weld in the 7-in. sample, illustrating the branching crack patterns. (Oxalic acid, electrolytic etch. 5X.)

The spun rock wool insulation was tested and found to have a total chloride content of about 80 to 100 parts/million. New insulation obtained for the repair was found to have chloride contents of 136 parts/million.

In addition to the insulation, samples of the old catalyst—both used and unused—were analyzed. The result showed the catalyst to have a chloride content of less than 0.2 parts/million. The water soluble chloride content in the catalyst was all that was measured.

After these data were analyzed, the conclusion was that the stress corrosion cracking was caused by chlorides in the insulation which could have been concentrated during the drying procedure. The chlorides could have been in the insulation or could have come from the test water. Several instances of stress corrosion cracking caused by insulation or coating agents have been reported. *(2) (3)*

The converter was returned to operation with no apparent leaks while plans were made to repair the basket permanently. Careful analysis was made to insure that the replacement basket would not crack also. The two alternatives arrived upon were: 1) fabricate the basket of material with at least 40% nickel which would not be susceptible to stress corrosion cracking; or 2) prevent chloride from coming in contact with the basket. Due to the high cost of materials with more than 40% nickel, such as alloy 600, the second alternative was selected.

The key to that alternative was to do away with the insulation, allowing any contact water to drain. Since there were several converters operating with only 20-gauge stainless steel heat barriers, it was determined that the insulation could be eliminated. Although some investigators have found that 321 stainless steel is no more resistant to stress corrosion than 304, the 321 was selected as the material of construction. It was felt that the titanium would reduce the sensitization of the surface and reduce the probability of surface cracking.

At that time, an economic evaluation was made to determine the best way to replace the basket. Three alternatives were studied: 1) shutting down when the repair failed and taking 60 to 70 days to replace the basket in the field; 2) repair the basket with a three-week outage, and then replace the basket in two more years; and 3) repair the basket, and at the end of two years replace the converter. The first was found to be the most economical and the most logical, and it was the path followed.

Within a few months, the temperature had started to climb. The increases were generally associated with upsets in plant operation. By February, 1971, there were hot spots in the vicinity of the fourth bed again. The hot spots grew larger, and Arkansas coolers were installed to cool them.

In June, 1972, after extensive planning, the plant was shut down to replace the basket. Prior to turnaround, a 200-ton truck crane was moved into the area to lift the basket. The replacement of the basket took 43 days from the time the converter was cleared of catalyst until the basket was turned back over to Operations for catalyst loading.

The basket was replaced using the following general procedure:

1. All external lines connecting to the basket through the shell or connecting to the heat exchanger were removed.
2. The pressure shell around the exchanger was unbolted and removed.
3. The weld between the exchanger and the basket was ground out.
4. The gas return line to the exchanger was cut.
5. The exchanger was removed and stored in a special frame constructed for that purpose.
6. The bottom head cover was removed and the catalyst drop-out chute was cut. The bottom head of the basket was then supported.
7. The pipes connecting the basket to the shell were then cut.
8. The neck of the basket was cut, allowing the basket to rest on bottom supports.
9. The head of the shell was then cut off, using a burning track, and removed.
10. The head was sent to a shop to prepare the weld surface, and the shell was ground down in preparation for rewelding the top head.
11. The old basket was removed.
12. The shell was inspected to determine if there had been any hydrogen attack. Hardness results along with X-ray and ultrasonics showed no damage to the shell.
13. Guides for centering the new basket were installed on the basket and the shell. A cone guide was installed in the bottom nozzle to aid centering the basket.
14. The bottom head was then replaced to allow filling with demineralized water to float the basket.
15. The basket was then floated into the shell slowly, allowing pressures to equalize, using the guides to center the shell.
16. The head was installed and the dismantling procedure was reversed.
17. The vessel was pressure-tested without the exchanger in place.
18. The exchanger was welded to the basket, and then all piping replaced.
19. The catalyst was loaded and the unit started up.

Conclusions

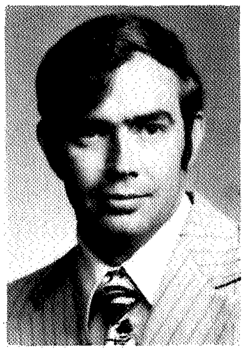
Good design for operating equipment materials of construction under process conditions is not enough to prevent stress-corrosion cracking. The environment of the piece of equipment from the time fabrications begins until the unit is operating at normal operating conditions must be considered.

Particular attention must be given to all water which comes in contact with the material of construction. Analysis of insulating materials which do not include tests for particular contaminants such as chloride in the 0 to 500-parts/million range should not be accepted. Even rain water with atmospheric contamination has caused failure of high alloy reformer tubes.

The failure of a piece of equipment as vital and as large as a converter basket requires careful planning and evaluation to effect the best possible solution. The hours spent planning the replacement of the converter basket were vital to the exceptionally smooth operation that followed. #

Literature cited

1. Nelson, G.A., *WRC Bulletin*, No. 145, p. 34 (1969).
2. Ashbaugh, W.G., *Materials Protection*, May, 1965.
3. Alessandria, A.V., and N. Juggard, "Stainless Steel in Petroleum Refining and Petrochemical Processes." API Division of Refining, *Proceedings*, **40** (III), 1960.



THOMPSON, R.L.

BROOKS, J.B.

DISCUSSION

BILL SALOT, Allied Chemical, Hopewell: I don't have a question. A comment or two. Three papers have been delivered on failures of Slim Jim converters. The first one was in Boston last year, and covered the Shahpur converter, and the two that we just heard today makes three. It's been mentioned that there were four, but I don't know where the 4th one was. I think maybe the #4 should apply to the number of Slim Jim converter baskets that have been inspected. There is a fourth one that has been inspected, and that is Allied Chemical's at Hopewell, Va., last November.

We went into it fearing the worst, rather than looking for the best, and I felt we were very well prepared for any eventuality. We spent two days round the clock with teams of inspectors going through it with ultrasonic equipement, trying to find cracks, and they never could find a single one. So we gave the basket a clean bill of health. We attribute the absence of cracks to two factors. One was that the chloride content of the Hopewell hydro test water was reportedly only 0.06 parts per million. The other was that we had pre-reduced catalyst in our converter from the beginning.

Some of the data that goes along with this might help Mr. Blanken. The temperature rise in the annular space in the Hopewell converter was only 17 degrees F. If his rise is less than that, I would expect his basket is better than ours.

It has been mentioned that there were six Slim Jim converters, but Mr. Blanken's was not included in that six. I think the six referred to those that had a low gas temperature in the annulus, whereas there are others that were designed for a higher gas temperature in the annulus. The latter may be susceptible to the same problem, but they weren't included in the group of six.

Allied has another Slim Jim converter at Geismar, La., which has not been inspected, but the temperature rise in its annular space is less than at Hopewell.

Q: Will you elaborate a bit on the detection of hydrogen intact using radiography, ultrasonic testing, and hardness testing. I wasn't aware that this is possible.

THOMPSON: Well, I'm not a metallurgist, but I can give a good try that might give you some help. Hardness testing, which compares the hardness in the area of concern with the hardness of other areas and the original specifications can give indications of attack.

If I'm mistaken, I'd certainly appreciate you correcting me on that. The radiography and ultrasonic testing effectiveness was primarily due to the nature of the vessel itself, it being a laminated, multilayer vessel, and what they are looking for here is a separation of blister that could have taken place between two layers, none were found. I did not mention that when the old basket was removed the entire converter being empty, it was once again inspected, and the same conclusions were reached.

As I say - I'm not too well versed on that. Possibly an over-simplification.

GARY L. PIGG, Agrico Chemical Co.: I'd like to ask Bill one question. It is my understanding that the two Allied converters do not have insulation on the baskets; they just have the radiation shield. Is that correct?

SALOT: No. Last year at Boston I made the comment that they didn't have insulation, and I take this opportunity to say I was wrong. It was explained to me in detail by several people that I was mistaken. If you'll get the publication of last year's symposium, you will find the correction is made there in print.

PIGG: I'd like to add one thing. The fact that we found our basket crack by virtue of a hot spot on the shell, doesn't mean that that's the only way you find cracks. Our crack was such that it occurred near the junction of the insulation support shield on the outside of the basket. This shield joint had separated, and all the gas from the cracks was channeling down through this shield, and was actually impinging on the pressure shell.

So the fact that you don't have a hot spot, as evidenced by these others, doesn't mean that you don't have possible cracks.