

Hydrodesulfurization Incidents Teach Lessons

Two types of failures in ammonia plant hydrodesulfurization vessels provide guidance to help preventing recurrence

W. D. Clark,
Imperial Chemical Industries, Ltd.,
Billingham, England

Lessons learned from two incidents with hydrodesulfurization vessels in an ammonia plant in Billingham have led to the adoption of preventive measures that are expected to protect against the possibility of a recurrence. One of the problems involved failure in a hydrostatic test and the other stress corrosion cracking.

The vessels were identical, built about 1961; 54 ft. in height; 7 ft. 3 in. inside diameter; 1-7/32-in. 1% Cr-Mo plate; with ultimate tensile strength of 65,000 lb./sq. in. Design code was similar to ASME VIII Division I for 300 lb./sq. in. gauge at 450°C. The design stress was 13,500 lb./sq. in. The vessels were fully radiographed and post-weld heat treated. Test pressure was 600 lb./sq. in.

Operating conditions were about 200 lb./sq. in. gauge at 400°C, with a hydrogen partial pressure of 80 lb./sq. in. There had been about 50 pressure cycles. The vessels' function was to remove sulfur from naphtha vapor. Sulfide scaling was expected, and a corrosion allowance of 0.25 in. had been made.

Failure on hydrostatic test after modification. In 1970, carbon deposits had choked the top of the top catalyst bed and it was decided to burn these off. Unfortunately control was inadequate and part of the top strake was grossly overheated. As a smaller catalyst volume was acceptable, the vessel was modified on site by cutting out a 5 ft. band and rewelding and applying local stress relief. The vessel was then pressure tested with water at 13°C, and split at 540 lb./sq. in. The crack was about 10 ft. long,

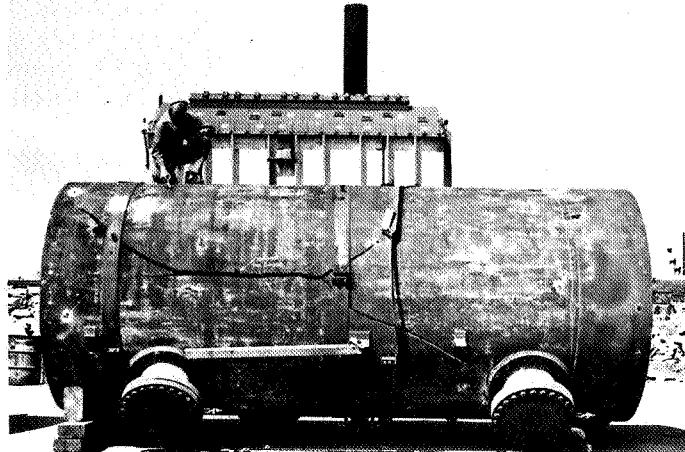


Figure 1. The result from the first incident (part of the vessel only), showing failure on a hydrostatic test.

basically vertical and had been initiated about 14 ft. below the repair weld. It is shown in Figure 1.

It had been initiated at an internal 1½-in. square section catalyst grid support ring. This had been installed in two pieces with two butt welds, and fillet welds above and below it. The butt welds were of the lowest quality and had cracked and caused a crack to grow through the fillet welds into the shell. Part of the crack was "old." This defect had initiated the failure. Similar defects were found at each of the two butt welds in each of two support rings in each vessel, as seen in Figure 2. The defects in the other vessel were locally repaired.

A variety of tests including some fracture mechanics work was done on the steel. The fractured plate had about 30 ft. lb. Charpy V at 13°C, other plates in the vessel were tougher—70 ft. lb. There was no clear evidence that ageing in service had reduced the toughness of the material: re-heat treatment gave very variable results.

We now insist that any butt welds in support rings have butt welds made to a high standard.

Stress corrosion. In 1974, the other vessel of the pair was observed to be leaking through its shell. A crack 28-in. long, seen in Figure 3, was found on the inside, which penetrated the wall for 5 in. It was in one of the double-V main seam welds made by the submerged arc process. No other crack was found except at a small fillet weld to a hand grip. A patch 36 x 12 in. was cut out and replaced *in situ*, stress-relieved, hydrotested, and the vessel is back in service.

Microexamination of the crack showed it to have a transgranular branching nature, and it is ascribed to stress corrosion by hydrogen sulfide, etc., derived from the

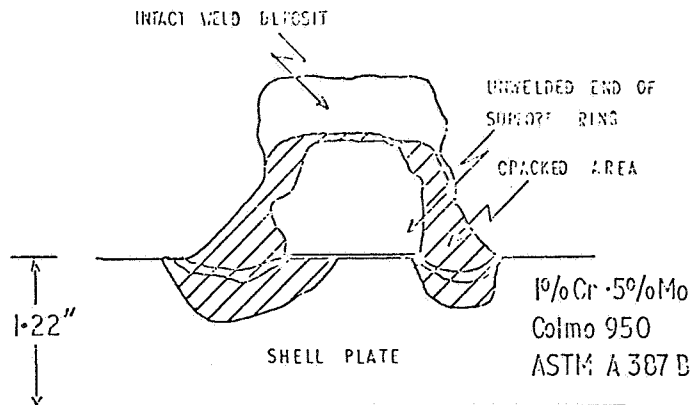


Figure 2. Profile of crack at support ring joint, 180° to failure.

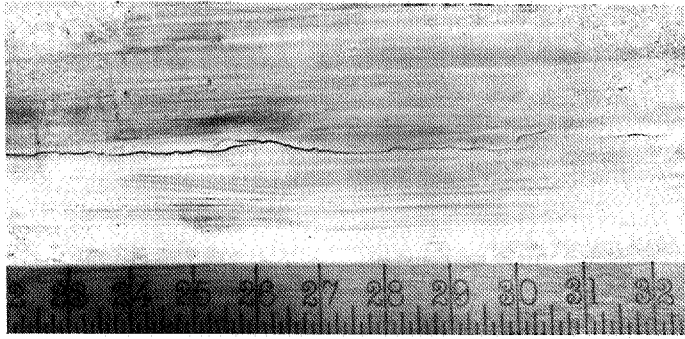


Figure 3. The second incident; a vertical crack inside the vessel.

scale when the vessel was wet. The hardness of weld and the heat-affected zone (HAZ) was in places up to 260 Hv, which implies susceptibility of H₂S cracking. The crack showed obvious tidemarks and had propagated in three stages, and the initial part was sulfided. It seems improb-

able from the nature of the crack that any part of it was caused during fabrication.

We now treat all vessels which have a sulfide scale and are of alloy steel as under suspicion, especially if the weld hardness may be above 240 Hv. Crack detection of all welds will be done as soon as possible, and start-up procedures avoid the pressure reaching more than 30% of the design pressure before the vessel is warm. #



W.D. Clark

DISCUSSION

R.W. PARRISH, Benfield Corp.: Your early discussion about the first failure in which you commented about the dismay of the man on top of the vessel during the hydrotesting, reminded me of a widely told story of 25 years ago when I first got out of school. The story probably bears repeating since the topic here is safety.

The commonly told story was about a man similarly placed on top of the tower during a hydro test. Just at the time of achieving the proper test pressure, the hose that was feeding the water at the bottom of the tower broke; the water proceeded to run out of the tower, and he failed to open the vent valve at the top and thus created a vacuum, collapsed the tower, and the man ended up being a fatality.

It's very common, it seems, for hydrotests to be run with a man sitting on top of the tower. This is probably the worst place he should be. Go ahead and make the hydrostatic correction, to obtain the proper gauge reading mount the gauge on the bottom of the vessel, use a man to vent the air, but then get him off the tower before any pressure is applied.

CLARK: That sounds good sense to me.

DON BAGNOLI, Exxon Chemical: When you mentioned that you did some experimental aging treatment, were you considering the possibility of temper embrittlement at all?

CLARK: Yes.

BAGNOLI: And if so, were you using a step cooling treatment?

CLARK: Using a what?

BAGNOLI: You mentioned that you did some laboratory

heat treatment Step cooling, an artificial agent used to simulate long-term conditions that would produce temper embrittlement.

CLARK: Yes

BAGNOLI: Was this a step cooling?

CLARK: No, what we were really doing was more taking the material in its somewhat brittle condition and seeing whether retempering it at increasing temperatures would increase its toughness, because by and large, if you've got strain aging or temper embrittlement, if you take it back up to the original tempering temperature, you expect to get rid of the effect of aging, and we got no significant improvement. It certainly didn't seem that heating up to 700°C, was improving the toughness of this particular plate, so we decided it was not obviously due to strain aging or the long time at 400°C and of course, as I say the other plates in the vessel—they were tough as cut from the failed vessel. If it was a phenomenon common to this kind of steel, then you'd expect that all the plates would have shown some fall-off in toughness.

BAGNOLI: Some work the API has been doing suggests that even the chrome material you're referencing is subject to some degree to embrittlement.

CLARK: Well that's what we were suspicious of ourselves but as I say, we didn't find evidence that this had been temper embrittled. I think that with 2¼ chrome if you take it up to about 700°C, the temper embrittlement is removed; you get back to the original toughness. Well that's what we expected in this case and it didn't happen. So we think it's not temper embrittlement.