Investigation of Hydrogen Damage to an Ammonia Converter

In the very early stages of a hydrogen attack against the converter, actual damage was about 0.25 in. more than indicated by ultrasonic tests.

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Figure 1. Schematic of the original ammonia converter.

In January 1971, Valley Nitrogen Producers was informed by the original contractor of its El Centro, Calif. ammonia plant that a possible problem of over-heating of the 600 ton/day ammonia converter could exist. The areas of concern were the top and bottom forgings as shown in Figure 1.

An immediate check was made, and temperatures

ranging up to 600° F were found in the general vicinity of the inlet and exit gas nozzles. External steam cooling was immediately installed on these forgings to reduce the temperature to less than 450°F. The converter forgings were designed to operate in a process gas atmosphere of about 800- to 900°F, and a hydrogen partial pressure of about 1,300- to 1,600 lb./sq. in. absolute. The use of carbon steel for these forgings necessitated the design of internal insulation canisters to reduce the metal temperature to less than 450°F to protect against hydrogen attack. With actual temperatures of up to 600°F, it was assumed that hydrogen attack was occurring on these forgings in accordance with the Nelson chart of operating limits for steels in hydrogen service.

The next step was to determine the extent of hydrogen attack. Since the converter was in operation, ultrasonic testing was used. This initial check indicated hydrogen damage to a depth of about 1.5 in. It was decided that with steam cooling of the forging, enough of a safety factor remained to allow continued operation until a complete investigation could be made during the summer turnaround.

Investigation of the failure

In July 1971, the ammonia converter was taken out of service. At this point, the converter had been in operation a total of 9,600 hr. and had 68 startups. The enclosures of the top and bottom forgings were opened and the insulation canisters removed so that the inside surface of the forgings could be examined. After visual examination the forgings were checked with dye penetrants on the inside surface and examined from the outside surface by ultrasonic testing. A "pie-shaped" specimen was cut from the bottom forging for microstructure studies. The insulation canisters were found to be slightly deformed. However, the insulation within the canisters was completely deteriorated. This insulation was believed to be Kaowool, B. W. 8- to 10 lb./cu. ft.

Figure 2 shows the blistering on the inside surface of the bottom forging looking up from the bottom at the outlet gas nozzle. Note where the "pie-shaped" specimen was removed for microstructure analysis. At this location, a



Figure 2. Inside view of the bottom forging.



Figure 3. Inside view of the bottom forging.

longitudinal crack ran down the inside wall about 3 in., and back inside the gas outlet nozzle about 2 in.

Figure 3 also shows the blistering on the inside surface of the bottom forging. Blistering was noted in about a 1- to 2 ft. circumeference band running 360° around the inside wall. Within approximately 3 in. of the bottom edge and approximately 8 in. above the gas outlet nozzle, no blistering was apparent. The blistered areas corresponded with those areas of higher temperatures.

Figure 4 shows the condition of the inside wall of the top forging. The blisterings were fewer in number than those found in the bottom forging, but were larger. Around the inlet gas nozzle the blistering was more extensive, but the size of the blisters was smaller. No major cracks were found in the top forging.

When the blisters in the top and bottom forgings were surfaced ground and checked with dye penetrant, secondary cracks and pin hole defects were noted in the



Figure 4. Inside view of the top forging.

immediate metal under the blister.

The "pie-shaped" specimen removed from the wall of the bottom forging was found to be consistent with SA 266 Class 2, carbon steel. This specimen was sectioned and the microstructure of each was examined showing significant intergranular cracking and fissuring. Figure 5 shows the extent of this damage at 5 x magnification in the vicinity of the large crack. The nature and severity of fissuring shown here is typical of each section from this "pie-shaped" specimen.

Figure 6 shows the hydrogen damage at the grain boundaries and inclusions at 200 x magnification, and Figure 7 shows the hydrogen damage at the grain boundaries at 1,000 x magnification.

The ultrasonic readings taken of the top and bottom forgings indicated a maximum damaged depth of 1.4 in. in the bottom forging and 1.6 in. in the top forging.

Evaluation of ultrasonic testing

To determine the sensitivity of ultrasonic testing in detecting hydrogen damage, a 2 ft. longitudinal section from the bottom forging was removed for ultrasonic and microstructure inspection. The grid layout of this section



Figure 5. Hydrogen damage at 5 x magnification.



Figure 7. Hydrogen damage at 1,000 x magnification.



Figure 6. Hydrogen damage at 200 x magnification.

was prepared, and ultrasonics taken over the entire length covering both hydryogen damaged and undamaged areas. After ultrasonics, each area was sectioned for microstructure analysis. In the area of maximum hydrogen damage, ultrasonics indicated attack depth of 1.3 in. while microstructure analysis revealed an attack depth of 1.5 inches. The area in which ultrasonics showed no attack, microstructure analysis revealed attack depth of 0.25 in. Our conclusion to the ultrasonic sensitity to hydrogen damage is that in the very early stages of attack, actual damage was about 0.25 in. more than indicated by ultrasonics.

Two tensile specimens were prepared for testing, one from the area of maximum damage, and one from the undamaged area. Both specimens were from the longitudinal direction rather than transverse due to the length of specimen required. The results, shown in Table 1, indicate the reduction in yield and ultimate tensile strength and elongation as a result of the hydrogen damage. The reduction in mechanical tensile strength in the short transverse direction of the wall would be drastically less because the damaged area, being primarily fissures along the longitudinal direction, would be 90° to the applied stress.

Table 1 A comparison of specimens taken from the ammonia converter.

Hydro	gen Damaged Area	Undamaged Area
Dimension, in	0.506	0.508
Yield Strength, lb./sq. in	30,150	33,990
Ultimate Strength, lb./sq. in	57,460	72,410
% Elongation, 2 in	9.0	29.5
Reduced Area, %	13.9	51.5



Figure 8. Schematic of the revised ammonia converter.

Repairs

As a result of investigation of hydrogen attack on this ammonia converter, Valley Nitrogen Producers elected to remove both the top and bottom carbon steel forgings. Replacement forgings were of 2-1/4 Cr - 1 Mo, Type SA 182, F22 alloy steel. The ammonia synthesis catalyst was not removed from the converter during the repair. The internal insulation canisters were replaced by castable insulation, and the converter returned to successful operation. Figure 8 is a schematic drawing of the revised ammonia converter. #



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