Failure Mechanisms and Material Degradations at High Temperatures in Ammonia Plants

The technical production of ammonia is nowadays nearly 100 years old. Nevertheless this process still represents a challenge for the materials and the material engineers, who conduct the materials selection. This is due to the temperatures and the media involved.

Failure mechanisms that are known in ammonia plants since the industrial ammonia production started are hydrogen attack, creep and embrittlement. The new generation of ammonia plants, having an optimized heat recovery widened the experience of failure mechanisms by bringing up metal dusting as a relatively common problem in the steam producing units downstream of the secondary reformer.

The wide range of failure mechanisms and material degradation experienced in BASF ammonia plants in the course of time will be illustrated by characteristic examples and remedies will be described.

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1. Introduction

The long history of ammonia production at BASF started with the development of the process in the early 20th century by Fritz Haber and Carl Bosch. In September 1913 in Ludwigshafen, Germany the first plant went into service with a capacity of 30 t (66 000 lbs) ammonia per day. Today BASF operates two plants in Ludwigshafen (Ammonia 3: conventional steam reforming plant, 1971, Ammonia 4: Braun purifier plant, 1982) and one plant in Antwerp/Belgium (Uhde design, 1990).

From the early days until now the limitation of material degradation processes at high temperatures to a tolerable scale was always a demanding task for the maintenance and operating staff in the plants to assure a safe and economic ammonia production.

2. History

During the development of the ammonia production process one of the main challenges for Carl Bosch and his team was the repeated failure of the pressure bearing shell of the pilot plant reactors after a short time of service (1). The reactors of the pilot plant were made of carbon-steel and were running at (400 to 500 °C (750 to 930 °F) at a pressure of about 200 bar (2900 psi) with a gas atmosphere consisting of H_2, N_2 and ammonia.

Fig. 1 shows an overview of a destroyed heat exchanger bundle and a catalyst-tube of a pilot plant.

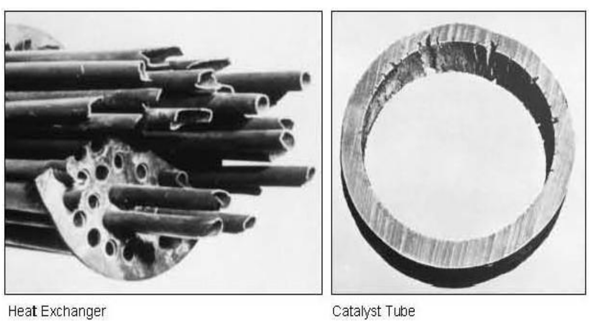

Fig. 1: Failure by Hydrogen Attack in an Ammonia Pilot Plant in 1910

Bosch, who not only studied chemistry but also had excellent knowledge of metallurgy, conducted metallographic failure analysis and found out that the carbon steel exhibited decarburization combined with internal fissuring. As we know today these are the typical features of hydrogen attack. Figure 2 demonstrates these metallographic findings.

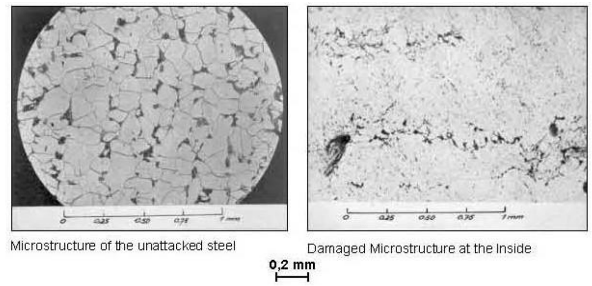

Fig. 2: Hydrogen Attack in a Catalyst Tube of an Ammonia Pilot Plant in 1910

It was the breakthrough with respect to this problem and probably also for the realization of the process in an industrial scale that Bosch discovered a solution. According to his observations he concluded that iron free of carbon would not show this kind of failure mechanism. The reactor construction was changed in a way that a liner out of soft iron was introduced in the pressure bearing shell. Hydrogen diffusing through the liner was led out through so called Bosch holes. These were holes in the pressure bearing shell, which do not degrade the pressure bearing capacity. By this way the carbon steel of the shell could be protected from hydrogen attack. Fig. 3 demonstrates this reactor design.

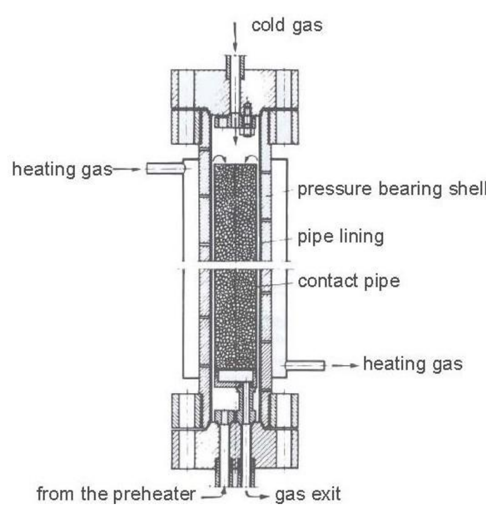

Fig. 3: Contact Furnace for the High Pressure-Ammonia-Synthesis with a Liner of Soft Iron and an External Heat Exchanger

In the following paragraphs some examples of typical failures and material degradations occurring at high temperatures in modern ammonia plants are presented which demonstrate that the ammonia process itself still represents a challenge for the materials involved and the materials engineers although the product is nowadays a commodity.

3. Primary and Secondary Reformer

Temperatures of 700 – 1000 °C (1290 – 1830 °F) and pressure of 30 – 40 bars (435 – 580 psi) in the reforming part of an ammonia plant render this area vulnerable for creep damage and other types of high temperature material degradation. Especially with the reformer tubes these severe conditions are likely to cause changes and damage in the material's microstructure with increasing time of operation sometimes leading to unscheduled expensive shutdowns of the plants.

3.1 Reformer Tube Failures

Due to the coarse microstructure of the cast tubes their creep resistance is higher compared to similar wrought alloys. The failure mode of the tubes is complicated as the creep damage starts at about 30 % of the wall thickness away from the inner surface. This is because of the superposition of the membrane stresses due to the internal pressure and the high heat flow leading to the maximum strains at this location. Classical methods to judge creep damage and remnant life like the field metallography or NDT-methods aiming to the surface are not applicable here. Fig. 4 demonstrates a cross section of a reformer tube, which has failed also showing some creep damage near the inner surface. Good experiences have been made in the BASF plants with the combination of eddy current and dimension measurements from the inside in estimating the actual damage state and the remnant life of reformer tubes.

In spite of periodic inspection during scheduled turnarounds ammonia plant No 3 experienced 4 tube failures after 11 years in service, which forced shut downs. All failed tubes were installed in the same area near the furnace wall and showed typical indications of creep damage.

Fig. 4: Creep Damage in a Reformer Tube

Based on the analysis of infrared pictures of the furnace the premature failure of the tubes could be correlated with a local higher temperature of 30 – 40 °C (50 – 70 °F) in combination with a higher average furnace temperature over the last three years.

One of the consequences was to replace 50 tubes, which were estimated to be creep damaged, another was to adjust the roof burners to get a more uniform temperature distribution. Additionally, the furnace temperature and the temperature distribution were extensively analysed by temperature measurements with an infrared camera every two months.

5 years later another 5 tubes failed within 1 year. This time the tubes failed in different sections of the furnace. It was concluded that all tubes had nearly reached the end of lifetime. So the plant management decided to replace all tubes during the next turnaround.

3.2 Secondary Reformer and Transfer Lines

Inside the secondary reformer the highest temperatures of the process occur (2100 °C (3800 °F) in the flame, 1200 °C (2190 °F) in the catalyst bed). To protect the wall of the pressure vessel from these high temperatures a multi layer refractory is installed on the inner side (castable and/or bricked material). The outer side is cooled by the atmosphere or by a water jacket. Problems for the integrity of the vessel can arise, if the refractory material fails. The BASF Antwerp plant several times suffered loss of refractory material close to the process air line, supplying the burner. The resulting hot spots on the vessel shell forced the shutdown of the plant. The root cause for the incidents were fatigue cracks in the process air line near the inner refractory layer, induced by an unfavourable design of the air burner. The remedy was to optimise the burner design and to replace the Silica containing refractory by a non-reducible high Alumina type material.

The adjacent transfer lines to and from the secondary reformer are also at risk when the refractory fails because the pressure bearing external shells are made of un- or low alloyed steel and cannot resist the combination of the high temperature and the pressure of about 30 bars (435 psi).

Figs. 5 to 8 show a failure of the transfer line from the secondary reformer to the boiler. In the course of the regular inspection with an infrared camera a hot spot was observed in the upper part of the line. Although the plant was directly shut down a bulge and a leak developed as demonstrated in fig. 5. After cutting away the steel shell the refractory seemed to be intact (fig. 6).

Fig. 5: Leakage and Bulging of the Transfer Line after a Short Term Overheating

Fig. 6: Refractory after cutting away the Steel Shell

In the course of an internal visual inspection with an endoscope which was led through a hole drilled in the refractory it became very evident that the bricks were gone in the area where the hot spot occurred (fig. 7).

Fig. 7: Bricks Broken off the Refractory Lining in the Over Head Position of the Transfer Line

As indicated in fig. 8 showing a cross section through the crack in the metallic shell of the transfer-line the failure has developed comparable to a high temperature tensile test with a considerable necking of the material at the crack.

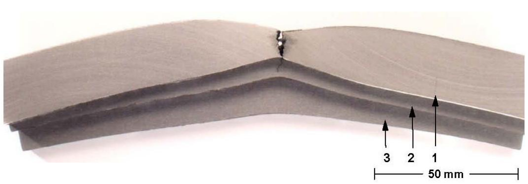

Fig. 8: Cross Sections Through the Crack in the Metallic Shell of the Transfer Line

The remedy in this case comprised several actions. One was to build up the refractory lining in a different and more stable way than it had been. Secondly the steel shell was changed from an unalloyed steel to a 0,5 Mo-steel with a higher high temperature strength which is believed to be beneficial as it will withstand the hot gas and the high pressure for a longer time if a similar incident occurs. Finally the inspection periods of the transfer lines and the secondary reformer with an infrared camera were shortened so that it will be possible to react earlier when the first indications of a failure of the refractory lining are observed.

4. Process Air Line and Air Preheater

In the modern steam reforming ammonia process the preheating of compressed air to high temperatures makes the selection of austenitic materials necessary. Design temperatures of more than 600 °C (1110 °F) are common. This was the case in BASF's Antwerp ammonia plant [2]. The air preheater coil and the headers are therefore made out of alloy 800 H. Fig. 9 shows the air preheater coil and the headers in a sketch.

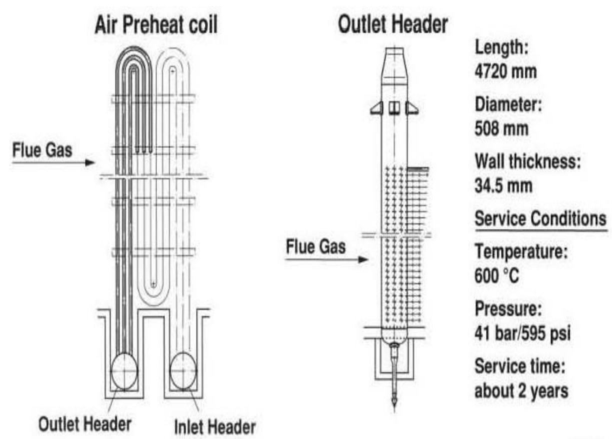

Fig. 9: Air Preheat Coil and Outlet Header

In the outlet header cracking was experienced after 2 years of service. The outlet header (see fig.9 on the right side) has a diameter of 508 mm (20 in), a length of 4720 mm (186 in) and a wall thickness of 34,5 mm (1,36 in). The operating temperature is about 600 °C (1110 °F) (design temperature 630 °C (1165 °F). The header was manufactured out of solution annealed alloy 800 H plate material, which was sequentially cold bent into a cylindrical shape and then longitudinally welded with an alloy 600 Ni-base filler.

After manufacturing no further heat treatment was performed. When the leakage was detected in the outlet header, extensive dye-penetrant testing was conducted which revealed cracking close to the pipe-to-tube connection weld in the pipe material and in the vicinity of the longitudinal weld. The crack in the pipe material close to the pipe-to-tube connection welds followed part of the circumference of the connection weld, then crossed over in the longitudinal direction to the next pipe-to-tube connection weld, where it again followed part of the connection weld etc. In the vicinity of the longitudinal weld, cracking was found in the longitudinal direction in the heat-affected zone and in the transverse direction crossing the weld. Figures 10 and 11 show the cracking in a segment, which was cut out of the header.

Fig. 10: Relaxation Cracking in an Alloy 800 H Outlet Header

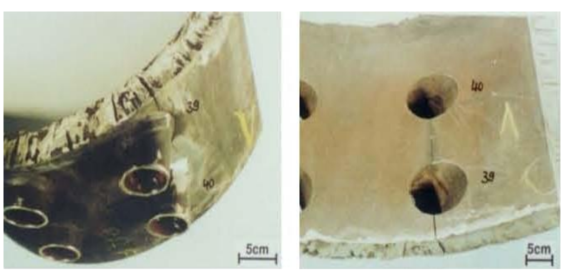

Fig. 11: Relaxation Cracking in an Alloy 800 H Outlet Header

Metallographic examinations of cross-sections revealed intergranular cracking starting from the outside in all cases. At the borders of the cracks, oxide layers are present with a metallic filament rich in Nickel in the middle (see fig. 12).

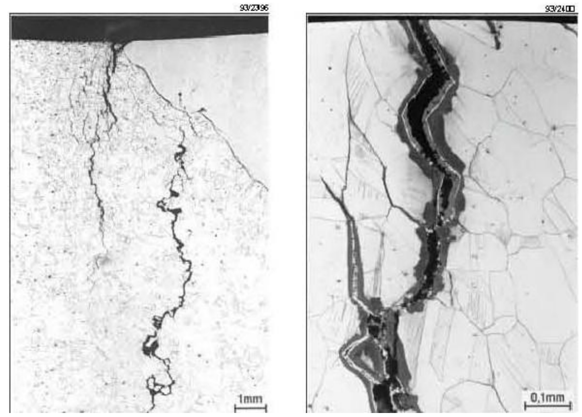

Fig. 12: Relaxation Cracking in an Alloy 800 H Outlet Header

These are indications of relaxation cracking. Relaxation cracking in materials like alloy 800 H is caused by high residual stresses from fabrication (cold deformation, welding) or by cyclic deformation during service at temperatures in the range of 550 to 760 °C (1020 to 1400 °F) [3]. In this temperature range, the critical nucleation radius for carbide precipitation is very small. The fine carbides (mainly chromium carbides) are precipitated within the grains at dislocations and slip bands after short periods of time leading to a strengthening of the grains (see fig. 13).

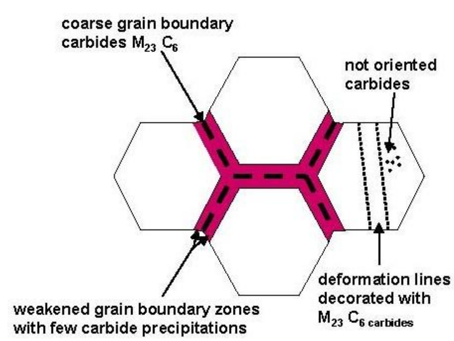

Fig. 13: Microstructural Constitution of Alloy 800 H after Service at about 550 – 760 °C

At the grain boundaries, coarse chromium carbides are present, leading to chromium depletion in the vicinity. Consequently precipitation free zones having low strength occur beside the grain boundaries. Because of the relative difference in strength between the grains and the grain boundaries, deformation under relaxation conditions will take place at the soft grain boundaries leading to crack formation at relatively low overall deformations. A similar mechanism was proposed to explain the stress relief cracking in microalloyed steels.

The crack formation starts stepwise on both sides of the grain boundaries in the weakened zones, producing a metal filament in the middle, which by oxidation processes becomes rich in Ni. Because of the comparatively higher dislocation density and the existence of high residual stresses, the heat-affected zones of weldments are the most critical areas for crack formation.

Practically all austenitic and Ni-base materials usually applied at elevated temperatures are more or less susceptible to relaxation cracking, if they contain carbon in an amount considerably above the solubility. A remedy with respect to this kind of cracking is a final heat treatment, which in case of alloy 800 H should be in the range of 950 °C (1740 °F). At this temperature the carbide precipitations are coarser and therefore the difference in strength between the grain boundaries and the grain interior is reduced. In the course of the fabrication of a new header, a two stage heat treatment was performed. After cold bending a heat treatment at 950 °C (1740 °F) was performed followed by a heat treatment at 850 °C (1560 °F) after welding.

Another way to handle relaxation cracking problems is to select the austenitic steel 1.4910, X 3 CrNiMoN 17-13 which is low in carbon content and nitrogen alloyed. This has proven to be a good remedy as well in other cases where relaxation cracking took place.

One candidate of austenitic material with a high creep strength is the grade 347. Therefore it is often selected in high temperature units. Because of the metallurgical condition with a Nb-stabilization this steel has been proven to be very sensitive to relaxation cracking as well. Also with this steel a stabilising heat treatment is advisable when handled at 600 to 650 °C (1100 – 1200 °F) in service.

5. Waste Heat Recovery Units downstream of the Secondary Reformer

Fig. 14 gives a schematic drawing of the waste heat recovery units downstream of the secondary reformer. At first there is a boiler, which in some modern plants is followed by one or two superheaters. In the boiler the hot gas from the secondary reformer is led through the tubes, the boiler feed water is on the outside. The superheaters in many cases consist of U-tube bundles.

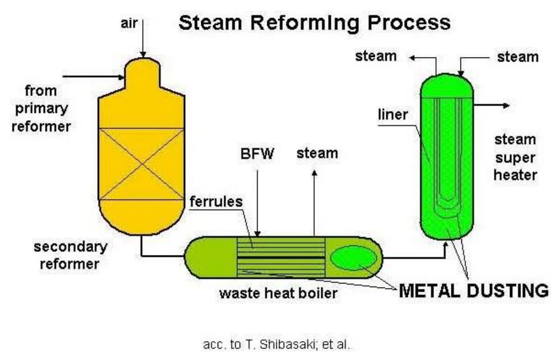

Fig. 14: Location of Metal Dusting Found in the Outlet Portion of Waste Heat Boilers

In both apparatus the gas consisting of CO, CO_2, H_2 and N is cooled down under non-equilibrium conditions, where the carbon activity considerably exceeds the value of 1. The carbon activity is determined by the partial pressure of carbon in the gas phase. The vapour pressure of carbon above a solid carbon body represents per definition a carbon activity of 1. Carbon activities in excess of 1 are a prerequisite for metal dusting. In the areas indicated, metal dusting has been experienced in boilers and superheaters [4]. In the ammonia plant of BASF in Antwerp, a serious case of metal dusting occurred in the first superheater after the waste heat boiler, which also was affected by metal dusting in the outlet area. [2]

Fig. 15 shows the schematic drawing of the superheater with a U-tube bundle and the pulled U-tube bundle together with the service conditions. Due to a design temperature of 650 °C (1200 °F), the U-tube bundle and also the liner were initially made of alloy 800 H.

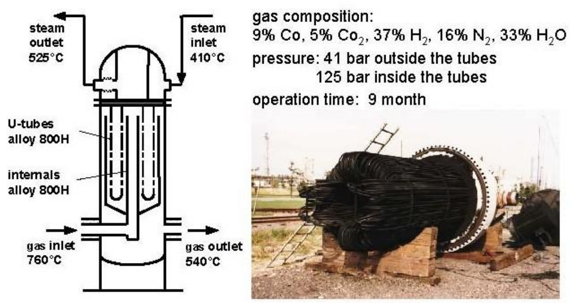

Fig. 15: Metal Dusting at a Superheater of the NH_3-Plant at BASF Antwerp

After about 9 month of service, heavy coking was observed on the U-tube bundle and liner. After removal of the coke, heavy pitting shaped metal dusting attack was observed. Fig. 16 shows an attacked part of the bundle in an overview, and metallographic sections of pits indicating the heavy carburization in the pitted area. In the unaffected area no carburization is observed. The 800 H liner was also attacked by metal dusting, while the alloy 600 weld of the liner was undamaged.

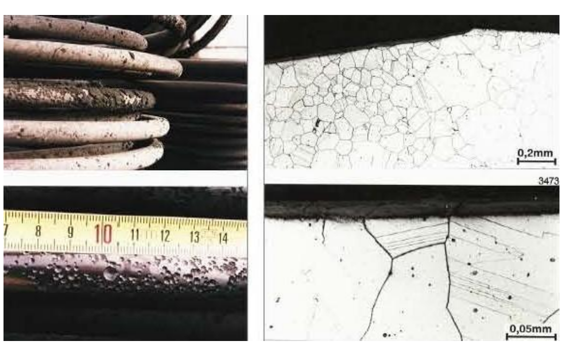

Fig. 16: Metal Dusting at a Superheater of the NH_3-Plant at BASF Antwerp

In Fig. 17 the failure mechanism by metal dusting is shown in principle and a simplified quantification to estimate the carbon activity is demonstrated on the right side. Metal dusting is an attack, which requires a carbon activity in excess of 1. For austenitic steels and Ni-base alloys, it has been experienced that temperatures of about 600 to 650 °C (1110 to 1200 °F) are the most critical ones in syngas atmospheres.

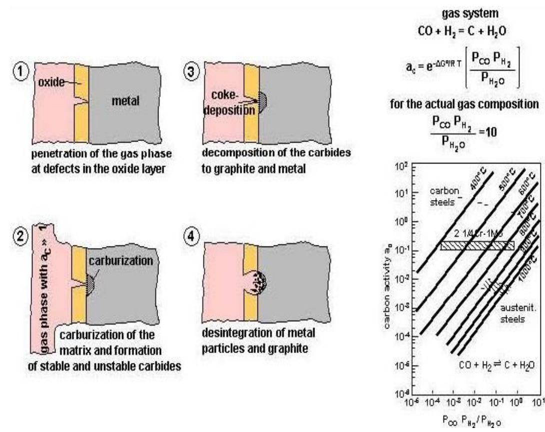

Fig. 17: Failure Mechanism by Metal Dusting

The surface of the material is, for the most part, protected from gas phase carburization by an oxide film. At defects like cracks etc., which may arise in the oxide layer, the gas phase having a carbon activity above 1 comes into contact with the bare metal surface directly leading to carburization and the formation of stable and unstable carbides. Since the bare metal surface also acts as an active site for coke deposition from the gas phase, coke depositions will form which lead to a dramatic change of the thermodynamic reference system. By this, carbides that are only stable in equilibrium with the gas phase will decay into graphite and metal. Due to the higher specific volume of the graphite, disintegration of the material into metal particles and graphite takes place. In the specific case of the superheater bundle, the tubes made of alloy 800 H lost about 0,9 mm (35,4 mils) within 9 month, which is about 1/3 of the total wall thickness. This indicates the high rate of the attack. The observation that the weld metal made of an alloy

600-type was not attacked already gave an idea of the remedy. However since the first bundle several others using alloy 600, alloy 601 finally demonstrated that alloy 600 is obviously an appropriate material selection for this case. Alloy 601 was considerably worse and in another alloy 600 replacement bundle other failures occurred. One of the main problems when looking at many processes in the petrochemical industry is, that all of them are running under conditions where the gas phase has carbon activities much higher than 1. Although several research programs on metal dusting have been run, there is still a problem predicting the criticality of the gases conducting an economically and technically appropriate material selection. What has been elaborated in a research program at TNO [7] is a ranking of materials as shown in fig. 18, which is in a certain way reproducible. However the surface treatment is also important. Ground and sandblasted surfaces seem to be superior compared with other surface treatments.

Diffusion layers		Ranking
Alcroplex™ Chromeplex™ Alonized ™		++
Material		
671	45 Ni / 55 Cr.	
Cast 35.35Nb-pipe	45 Ni / 35 Cr.	
Cast 25.35-pipe	35 Ni / 35 Cr.	
Kanthal APM	22 Cr. /5,8 Al	
MA 956	/ 20 Cr./ 4,5 Al / 0,5 Y_2O_3	+
602 CA	62,5 Ni / 25 Cr./ 2,3 Al / Y + Zr.	
693	62 Ni / 29 Cr. / 3 Al	
690	61 Ni / 29 Cr.	
353MA	35 Ni / 25 Cr.	
446	26,6 Cr.	—
601	62 Ni / 22 Cr./ 1,35 Al.	
304	8 Ni / 20 Cr.	
800H	30,5 Ni / 20,5 Cr.	

Fig. 18: Metal Dusting Resistance of Materials (acc. to TNO)

6. Ammonia Reactors

In the ammonia reactors there is potential for nitriding of materials in long-term service. Temperatures in the reaction zone are normally about 520 °C (970 °F). Fig. 19 shows a thermowell tube made out of the austenitic steel grade 321 (X 6 CrNiTi 18-10), which failed due to nitriding. Normally these tubes, which are pushed through the different catalyst layers, are changed on a regular time basis. This tube was missed to replace, so that the nitriding reached a critical amount. In the external dark area the nitriding is so heavy that practically all of the chromium contained in the material is consumed. Because of the residual stresses combined with the formation of such a zone, which is brittle in nature, an equidistant cracking takes place, which leads to locally deeper nitriding. Finally the remaining cross section was no longer sufficient and rupture occurred.

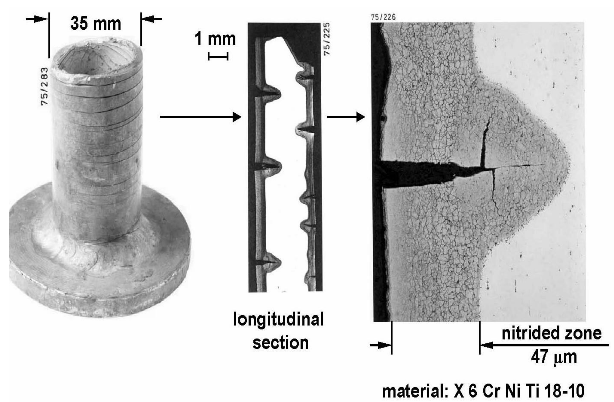

Fig. 19: Fracture of a Thermowell Tube due to Nitriding

At BASF in Ludwigshafen one of the ammonia plants is running with hot walled reactors. These are reactors where the pressure bearing wall is heated by the hot inlet gas to a temperature of about 400 °C (750 °F). Fig. 20 shows the process scheme of the conversion consisting of two reactors in series where the ammonia content is increased just up to 16 % of the stream at the outlet of the second reactor. The material of construction was a 2 ¼ Cr-1 Mo-steel.

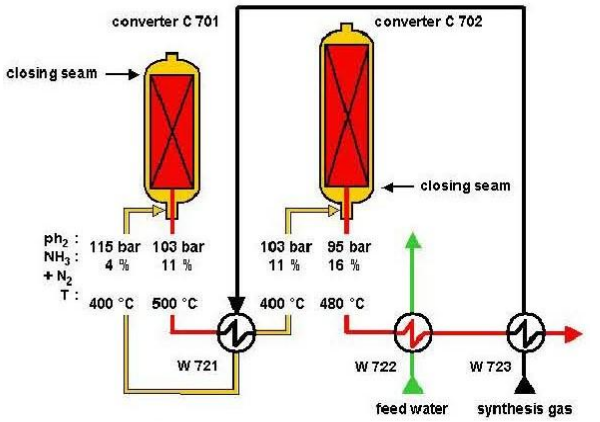

Fig. 20: Process Scheme, NH_3-IV-Plant, BASF Ludwigshafen, Synthesis-Loop

Fig. 21 demonstrates where the operating conditions of the two reactors are located with respect to the temperature and the hydrogen partial pressure in the Nelson diagram. After eight years of operation a leak was observed within a 175 mm (6,9 inch) thick girth weld – the closing seam - of the second reactor, the reactor with the higher ammonia concentration. Extensive ultrasonic testing and metallographic examinations revealed a considerable amount of cracking in the whole circumference of the weld [5].

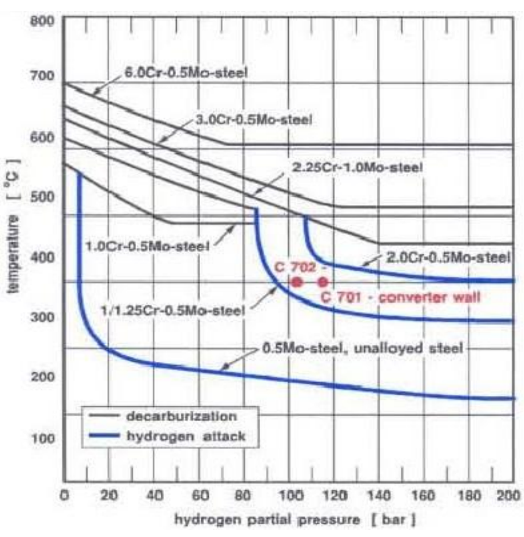

Fig. 21: Operating Limits of Steels in Hydrogen Service to Avoid Decarburization and Fissuring

Fig. 22 shows a sketch of converter C 702 and the position of the closing seam, which was cut out in the course of the repair work.

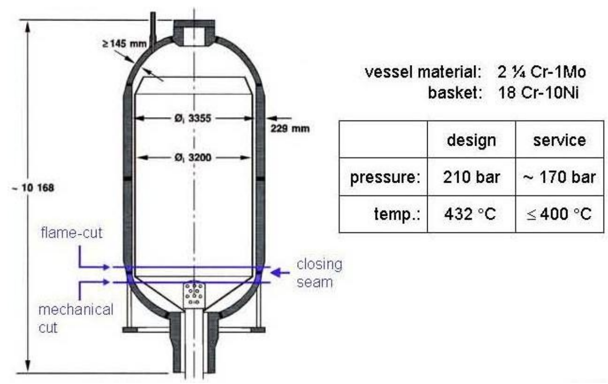

Fig. 22: Converter C 702

Fig. 23 indicates the depth of cracking attained over the circumference of the weld.

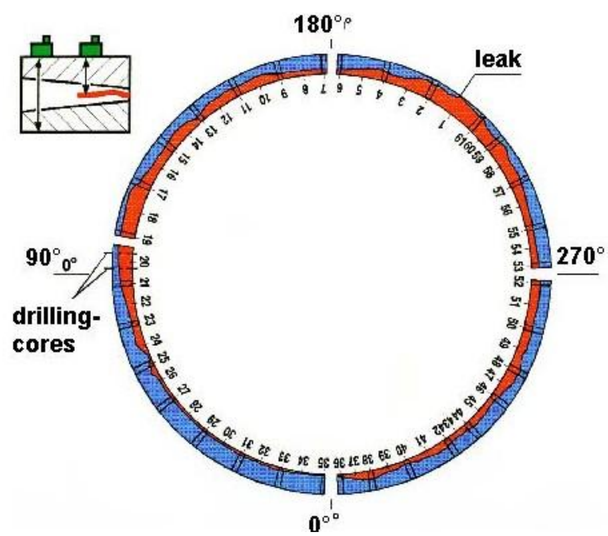

Fig. 23: Converter C 702 – Crack Path and Partition of Cracked Welded Seam

Metallographic cross sections of the weld revealed a very distinct appearance of the microstructure along the cracks (Fig. 24).

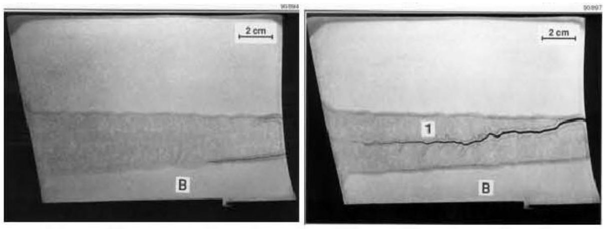

Fig. 24: Converter C 702 – Failed Closing Seam

Cracking was intergranular and the cracks were accompanied by a band indicative of decarburization [6]. Obviously cracking was assisted by hydrogen attack. The mechanism responsible for the damage is demonstrated in fig. 25.

The initial step of cracking is nitriding by ammonia. Since the chromium carbides in the steel, which protect it from hydrogen attack, are less stable than chromium nitrides, chromium carbonitrides are formed and a certain amount of carbon is released. This carbon methanates in combination with the hydrogen dissolved in the material. In case of low mechanical stresses, a nitrided zone is formed in which cracking by hydrogen attack occurs parallel to the surface as shown in the upper part of fig. 25. The decisive point for the cracking perpendicular to the surface was that, in the closing seam, high residual stresses were present after fabrication, because this seam was locally heat treated. Under the action of these high stresses, cracking by hydrogen attack no longer happens parallel to the surface but perpendicular to it, as indicated in the lower section in fig. 25.

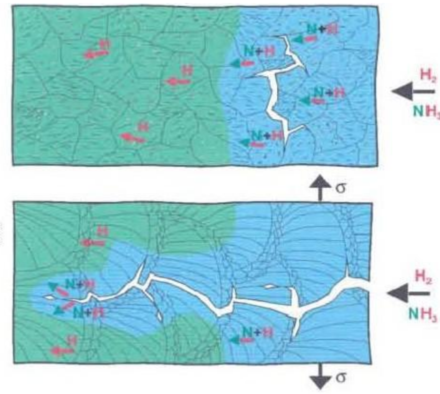

Fig. 25: Damage by Synthesis Gas

The plant management later on decided to replace the reactor. Again a 2 ¼ Cr-1 Mo was selected. The wall thickness, however, was adjusted to comply with an ASME-design. Furthermore it was decided that a final heat treatment had to be conducted in a furnace. Still during the regular shut downs, the girth welds of the reactors are inspected with a highly sophisticated ultrasonic testing program. Up to now, no cracks have been detected in the new reactor.

As indicated in the process scheme in fig. 20, heat recovery is conducted in the conversion by preheating the synthesis gas (W 721, W 723) and by steam production (W 722). As the vessels are charged with ammonia and hydrogen containing gas at high temperatures, there is always the potential of nitriding, hydrogen attack and embrittlement. Due to the high pressures involved, conventional low alloyed Cr-Mo-steels are applied, having excellent strength at the service temperatures. In order to handle the problem of nitriding and hydrogen attack and embrittlement, intelligent designs have been developed which make it possible to run heat exchangers fabricated from these materials under such critical conditions in long term service. Fig. 26 demonstrates an example of such a design for a boiler directly heated with hot, high ammonia concentration gas.

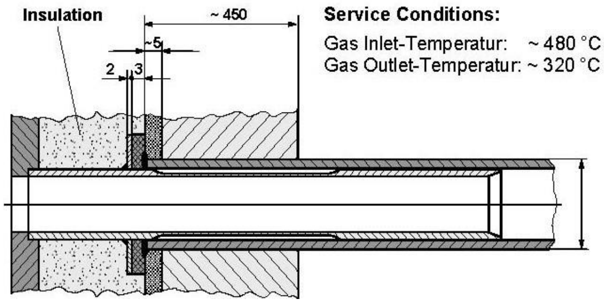

Fig. 26: Inlet-Part of a Process Cooler of an Ammonia Plant

For pressure and temperature reasons, the tube sheet of the gas cooler has a thickness of more than 400 mm (15,75 inch). The hot gas is led through the tubes. To protect the tube sheet from direct contact with the hot gas having a temperature of more than 400 °C (770 °F) there is high temperature insulation on the tube sheet. The hot gas is led through alloy 600 H type ferrules in this area.

The shape and length of the ferrules is chosen in a way that they act as insulation against the hot gas in the region of the tube sheet, where no cooling due to heat exchange occurs. The tube sheet itself is weld overlayed with alloy 600-material. Here the tubes made of the Cr-Mo-steel are welded in. The design makes it possible to perform repairs because the Ni-base material is resistant to nitriding and hydrogen attack under service conditions. As can be recognized, this kind of intelligent design takes the possible material degradations in the hot product gas stream into consideration and makes it possible to use conventional Cr-Mo- steels under such critical conditions.

7. Summary

Although ammonia itself is a commodity nowadays, the production of ammonia still remains a challenge for the materials involved. As demonstrated by several examples following the process route, a wide variety of high temperature corrosion processes and mechanisms of material degradation may act in an ammonia plant in service. The material concept for conventional older plants is based mainly on the use of plain carbon or low-alloyed steels. This is not only due to economic considerations but also because of technical aspect.

Un- and low-alloyed steels have a higher heat and temperature conductivity, a higher strength and lower thermal expansion rate compared with austenitic steels. In order to make them applicable regarding especially the temperature as critical parameter intelligent designs were developed. In the new plant concepts where a higher energy efficiency was realized border line temperatures for the conventional un- and low alloyed steels were exceeded. This made it necessary to introduce austenitic steels mainly the readily available alloy 800H in some vessels of the process. That this step was not without risk is a lesson learned in many of the modern ammonia plants, as some unexpected failure cases occurred due to mechanisms which were unknown or forgotten to the community of process designers. Relaxation cracking is one of these mechanisms which to our mind can be handled by a stabilising heat treatment or the selection of specific material grades insensitive to this failure mechanism.

The other failure mechanism is metal dusting, a multistage process, which is quite well understood from its basic steps. However regarding the prediction it is up to now still difficult to specify a material that will not metal dust. Much research efforts have been performed in programs in the US and in Europe, which more and more bring the light of a quantitative understanding in this darkness.

For the materials engineer the ammonia process is a challenge because most of the important vessels in this process run under conditions where time dependent degradation mechanisms act. These are creep, nitriding and hydrogen attack. The challenge is to judge on the damage state and to estimate on remnant life of the components and to develop repair procedures in case that failure occurs.

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