Problem, Failure and Safety Analysis of Ammonia Plant: a Review

INVITED PAPER

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Abstract – *Ammonia is produced on large scale mainly for production of fertilizers using steam reforming of natural gas. Capacity of single stream has gone upto 4000 tpd. Large release of ammonia due to accident and vessel failure etc. has decreased considerably in the last three decades. But problems and failures do occurs frequently in the ammonia plant even after following the inherently safer design philosophy and risk assessment. Major areas of failures are reforming and synthesis loop. This paper presents the information on various kinds of failures taken place in ammonia plant in the last three decades. It also includes large releases of ammonia from the storage vessel as well as important information which are generally applicable to chemical industries with focus on ammonia plant. The collected information has been critically reviewed and conclusions drawn.* **Copyright © 2010 Praise Worthy Prize S.r.l. - All rights reserved.**

I. Introduction

Ammonia is one among the largest volume inorganic chemicals in the chemical process industries. About 80% or more of the ammonia produced is used for fertilizing agricultural crops [1]. From 1980 to 2000, the capacity of single stream ammonia plant was in the range of 1500-2000 tpd. But in the present decade, the capacity of new ammonia plant has increased drastically from approximately 2000 tpd to 3300 tpd. Presently, the largest ammonia plant has the capacity of 3300 tpd based on Uhde's proprietary Dual Pressure Process and utilizes Johnson Matthey's Katalcojm™ range of catalysts [2].

But very soon "Algeria Oman Fertiliser Company" will become as the largest single-stream ammonia plant in the world, which is due on-stream in the middle of 2012, has the capacity of 4000 tpd [3]. It is expected that capacity of single stream may even reach 5000 tpd, considering current pace of development. But these large plants also pose increased hazard and risk associated with it.

The main reason for increase in the capacity of a single stream to reduce specific production cost through so-called economy of scale, i.e., if design output is doubled, the capital cost increases by only 50- 60%. There are also some savings on operating costs, particularly in terms of the thermal economy and labour.

Though ammonia plant has well proven technology; however, there are problems and failures of process equipments, machineries, instruments and control systems etc., many of these are not reported in the literature. Safety of a plant can be improved, but cannot be guaranteed.

Generally plants are designed considering all the aspect of hazards and risks involved. If everything functions as per their intended purpose then plant will run smoothly. But this ideal situation is seldom achieved. There will be some deviations/ failures etc. which will result in economic, environmental and/or human loss. One factor which is out of control is human error but upto certain extent this is also taken care during plant design by following inherently safer design [4]. Still we are facing the problems and failure in our plants. The present review will focus and discuss all the problems and failure associated with ammonia plants which are available in open literature which appeared in the last 3-4 decades.

In the last 2-3 decades, the process almost standardized and being implemented on commercial scale in new plants as well as retrofit in existing plants where ever possible and economically beneficial. But retrofit and new developments may also create new kind of hazards which are to be anticipated, analyzed and suitable safeguards incorporated.

Let us take a quote from Livingstone [5] "How safe is your ammonia plant? Safety simply means freedom from danger and designers and operators should ask themselves, 'are the safety systems in operation on our ammonia plants to the best design and safety standards?'" The Safety record suggests the much room for improvement and definitely no room for complacency.

II. Process for Ammonia Production

The raw material source for ammonia plants is natural gas for producing hydrogen by steam reforming. An alternative raw material is naphtha, which requires partial oxidation. The synthesis of ammonia can be divided into following four stages which are discussed in detail.
1. Stage 1- Catalytic reforming of natural gas (Primary and Secondary Reformers);

2. Stage 2- Purification of Synthesis gas (Shift conversion, CO_2 removal and Methanation);
3. Stage 3- Compression of the Synthesis gas to the required pressure;
4. Stage 4- Ammonia synthesis loop (Conversion, Separation and Recycling).

In the subsequent parts of process description operating parameters are mentioned which are indicative only. Temperature and composition may not vary much but pressure of the system particularly in synthesis loop can have different value. The overall process for ammonia plant is shown in Fig. 1 [6].

II.1. Stage 1: Catalytic Reforming

II.1.1. Desulphurization and Primary Reforming

Synthesis gas is produced by steam reforming of natural gas. However, before this can be carried out, all sulfurous compounds must be removed from the natural gas because sulfur has an adverse effect on the catalysts used in the reforming and synthesis reactions. These are

removed by heating the gas to 400 ^{0}C and reacting it with zinc oxide as per following reaction:

$$ZnO + H_2S \longrightarrow ZnS + H_2O$$
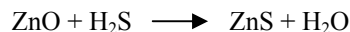
After desulfurization and scrubbing, the natural gas [NG] is sent to the primary reformer for steam reforming, where superheated steam is fed into the reformer with the methane. The gas mixture passed through reformer tubes which contains Nickel catalyst and externally heated by the combustion of fuel, normally natural gas and purge gas, to approximately 770 ^{0}C in the presence of a nickel catalyst where methane is converted into CO/CO_2 and H_2. At this temperature, the following endothermic reactions are driven to the right, converting the methane to hydrogen, carbon dioxide and small quantities of carbon monoxide.

$$CO + H_2O \longleftrightarrow CO_2 + H_2$$
$$CH_4 + 2H_2O \longleftrightarrow 4H_2 + CO_2$$
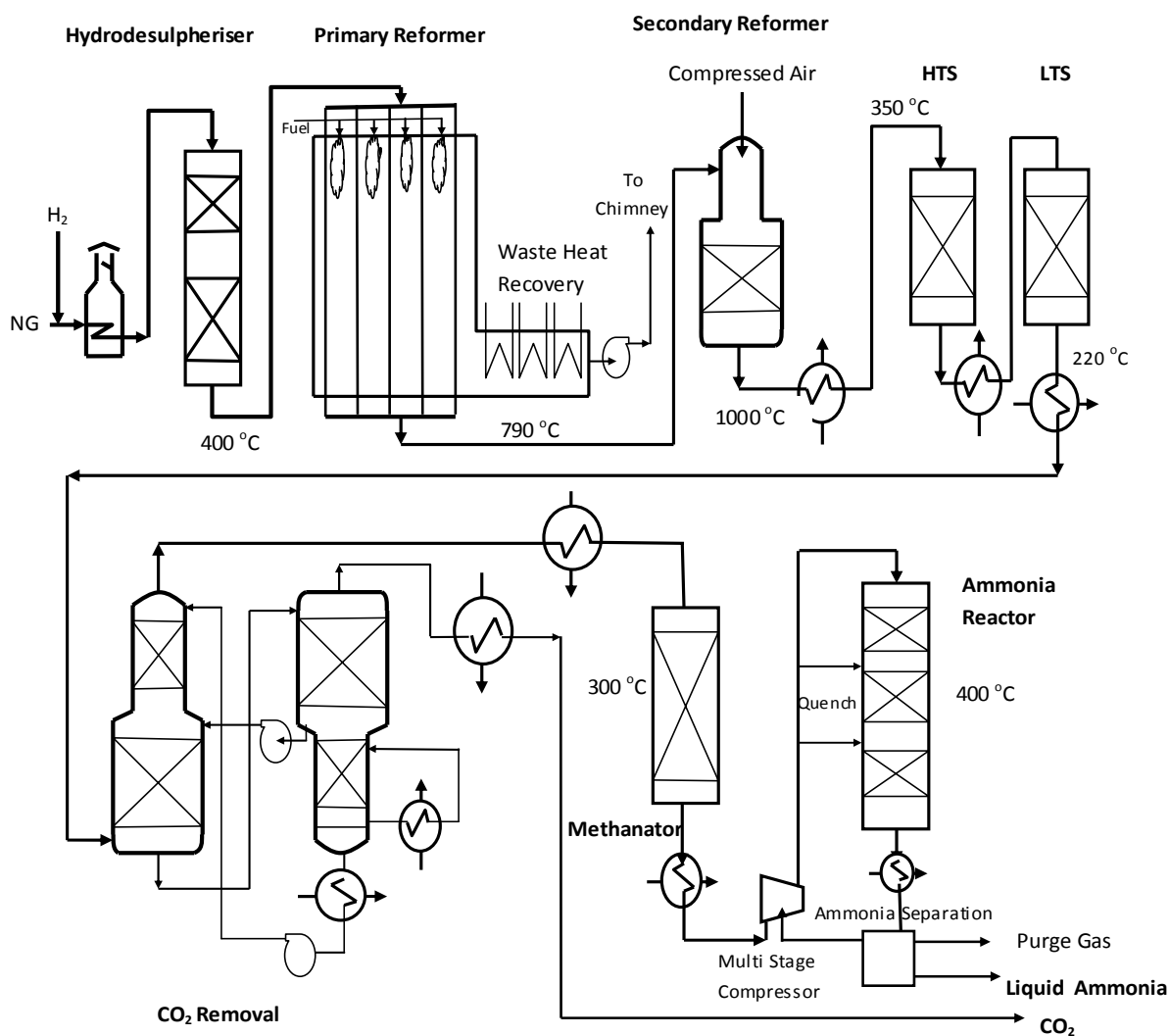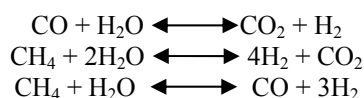$$CH_4 + H_2O \longleftrightarrow CO + 3H_2$$

Fig. 1. Overall Process Diagram of Ammonia Plant

This gaseous mixture is known as synthesis gas. Conversion in primary steam reformer is about 70% of the hydrocarbon feed into synthesis gas. The reactions are endothermic, thus the supply of heat to the reformer is required to maintain the desired reaction temperature. The hot flue gases contain lot of energy and recovered upto maximum possible extent before releasing to atmosphere through chimney.

The process gas mixture further goes to secondary/ auto-thermal reformer. The approximate composition of gases leaving the reformer are CH_4- 6 mol%; CO- 8%; CO_2 -6%; H_2- 50%; and H_2O- 30%. The operating pressure is in the range of 25 to 40 bar.

Dai [7] has done studies on the prediction of remaining life and ageing of material for pressurized tubes of industrial furnace operated at elevated temperature. The results of mechanical properties tested at high temperature (800 and 850^0C) had shown that the aged metal's mechanical properties improved after the solution heat treatment. In other words, the outlet pigtail tubes after being employed to about 80000 h can be further used continuously, operated at high temperature, for another design life (100000 h) by using solution heat treatment processing based on their proposed methodology for predicting the remaining life and ageing of material of furnace tubes.

There are numerous incidents of reformer tube failure and fire in ammonia plant. The probable causes of fire are direct impingement of flame on the tube due to the partial blockage of burner tips is possible. This may cause overheating of the tubes, which ultimately led to one tube rupture. Flame impingement from a nearby leaky tube might result in overheating and the ultimate rupture of the other nearby tubes. Sometimes situation may lead to explosion in reformer furnace as well [8].

Singh et al. [9] discussed an incident of fire in an ammonia plant which began with leakage in tube of a natural gas preheater ignited a small fire. The small fire ultimately developed into tube to burst resulting in large fire and plant shutdown.

Reformer tube can also fail by stress corrosion cracking. During welding, the steel in the heat affected zone (HAZ) can get sensitized and this may subsequently lead to stress corrosion cracking. Bhaumik et al. [10] discussed the premature failure of a primary reformer tube in an ammonia plant in which number of catalyst tubes were found to have failed just below the inlet flange weld within about 2 years in service. Stress corrosion cracking (SCC) may get aggravated further due improper welding procedure. Preventing such failure by following the proper welding procedure is very important. Therefore it is recommended to cool the steel below 100^0C after every weld pass, use filler metal electrodes with low carbon content (such as the 321 grades) and stabilising the steel grades with addition of Nb (up to 1%) will help preventing sensitisation during welding [9].

Ray et al. [11] have presented their analysis of failed reformer tube. Reformer tubes from a fertilizer plant made of modified HK 40 steel which failed after 4 years service during startup of plant. At that time only 60 burners (out of 576 burners) were firing in the reformer. The gas in the catalyst tubes was mainly hydrogen and steam at low pressure of 3 kg/cm^2 only. Seven tubes had ruptured in the bottom portion in one corner of the radiant chamber. Their visual observation - the tubes had a rather black outer surface indicative of high temperature and the oxide scale was adherent. There was no indication of any localised damage in the form of pits.

Therefore they concluded that the overheating appears to be the dominant factor limiting the serviceability of the reformer tubes. Though the choking was observed only in one pigtail, the possibility of such choking in other failed tubes as well cannot be ruled out (choking may be partial and temporary). The main cause of failure, when only a few burners are on, appears to be damaged catalyst which choked the flow giving rise to an increase in temperature and pressure leading to creep rupture of tubes.

Subsequently, the hydrogen coming out of failed tubes might have increased the temperature excessively and caused some massive secondary damage. Since all the failure of reformer tubes had taken place at one location on the faces in the front of the burners where one could expect somewhat higher temperatures than at the rear surfaces of the tubes.

They concluded that the longitudinal cracks found in the tubes were caused by overheating because of inadequate feed flow caused by the choking of damaged catalyst. Therefore precautions should be taken while charging the catalyst that no broken piece of catalyst or any external material goes along. Overheating during service is primarily responsible for significant degradation in mechanical properties and microstructures in the bottom portion of the reformer tubes.

Swaminathan et al. [12] have done the comprehensive study on failed reformer tubes (failed after 8 years in service) and assessed the remaining life of other tubes. Catalyst filled tubes for reformer are made of cast HP-micro-alloyed grade (35Ni25Cr1NbTi alloy). High creep resistance of these alloys is the main criteria for their selection. Premature failure of process heater tubes are very often observed mainly because of service ageing leading to embrittlement, early onset of creep damage resulting from overheating. In addition, service related carburization/ oxidation also contribute to the premature failure.

The failure of primary reformer furnace tubes in service was due to localised overheating which led to poor creep strength. Microcracks have appeared only at the overheated portion of the tube. The tubes generally get overheated mainly due to flame irregularities. It was the extension of their earlier work by Ray et al. [11].

Primary reformer tubes are centrifugally cast alloy steel tubes which experience the extreme conditions. There are three factors in tube designs which are to be considered during design- need to balance heat transfer rates, optimize catalyst volumes and minimize thermal

stresses, while maintaining the tube life expectancy. It is also important to closely observe and inspect the reformer tube manufacturing process, including casting, machining and fabrication. Subsequent boring, machining, welding, and inspection operations are important to ensure the quality of the tube segments and the final reformer tube assembly.
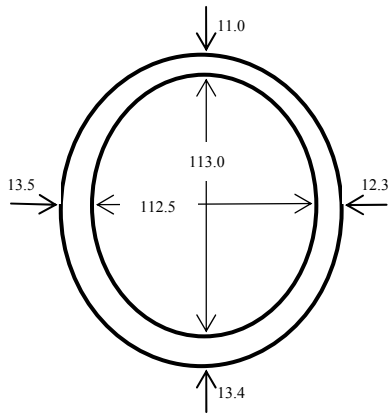

Fig. 2. Typical measured tube dimensions (mm) of a failed tube

In one of the case study, Webba and Taylor [13] found that the reformer tube failed after 7 years, which appear to be due an end of life creep failure, was actually caused by eccentricity of the tube, as shown in Fig. 2. Another case study revealed large number of tube having crack like defect (pin hole) at the weld joint (dissimilar metal).

Pequeno [14] reported their experience gained during 7 years of operation of a topfired primary reformer using heavy fuel oil, difficulties faced, mainly corrosion and environmental problems.
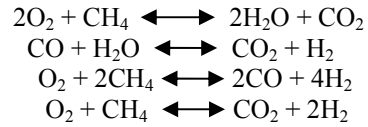
Shannon et al. [15] describes the recent developments in an inspection system, which uses multiple NDT techniques to provide the most comprehensive assessment of reformer tubes' current condition. This is coupled with a sophisticated remaining life assessment software model, which not only predicts the remaining life of each tube in a furnace but can also calculate the tube ageing.

It is also important to properly specify the requirements during purchasing of the tubes. In ammonia plant, the furnace tubes end in pigtails which are flexible pipes that allow expansion to take place. In a plant, over the years, many small changes were made to the pigtails' design. It had shortened the bending length and thus increased the stress in tube material at the bend. Ultimately, 54 tubes failed, producing a spectacular fire [16].

II.1.2. Secondary Reformer

The synthesis gas is cooled slightly to 735 ^{0}C and then flows to the secondary reformer where it is mixed with a required amount of air to supply the nitrogen required for 3:1 H_2 and N_2 synthesis gas. The highly exothermic

reaction between oxygen and methane produces more hydrogen. Important reactions are:

$$2O_2 + CH_4 \longleftrightarrow 2H_2O + CO_2$$
$$CO + H_2O \longleftrightarrow CO_2 + H_2$$
$$O_2 + 2CH_4 \longleftrightarrow 2CO + 4H_2$$
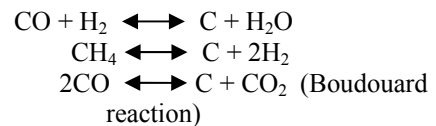$$O_2 + CH_4 \longleftrightarrow CO_2 + 2H_2$$

The heat required for reforming is generated by partial combustion of gases when air is mixed with process gas in secondary reformer. The reformed process gas is employed to generate steam and to preheat the natural gas feed. As the catalyst that is used to form the ammonia is pure iron, water, carbon dioxide and carbon monoxide must be removed from the gas stream to prevent oxidation of the iron which is carried out in the next three steps- shift conversion, CO_2 removal and methanation.

The gas composition and temperature during reforming are within the carburising range and metallic components in some parts of the reforming system are thus susceptible to degradation by a mechanism of accelerated carburisation known as "metal dusting" [17].

"Metal dusting" is the term used to describe the catastrophic degradation of metals in carbonaceous gases at elevated temperatures, usually in the range 450^{0}C to 900^{0}C, in which the metal surface rapidly becomes severely pitted. The term is generally used due to the appearance of the pits which often contain a dust of loose, powdery, magnetic corrosion product of graphite, metal carbides and oxides.

The burner liner is intended to protect the refractory lining from erosion by the incoming gas stream may be susceptible to metal dusting if improper material is used. Holland and Bruyn [17] described one such failure of Mossgas secondary reformer in which 200 mm hole was formed fortunately no much damage occurred. The failure was caused by localized overheating resulting in melting of refractory lining and shell.

Carburization and metal dusting can proceed by one of the following reactions when gas contains CH_4, CO, CO_2 and H_2:

$$CO + H_2 \longleftrightarrow C + H_2O$$
$$CH_4 \longleftrightarrow C + 2H_2$$
$$2CO \longleftrightarrow C + CO_2 \text{ (Boudouard reaction)}$$

They observed that the stable protective surface oxide film of Cr_2O_3 on Alloy 800 was able to resist metal dusting but only slight changes would be required to cross the boundary of Cr_3C_2, chrome carbide stability zone to initiate metal dusting [17].

In newer configurations, better energy integration allows heat evolved in the secondary reformer to be used for the primary reformer in which the fired tube furnace is replaced by a heat exchange reformer [18]. Over the years, the steam reforming process has been optimized with the design of better burners for the furnaces, highly creep resistant materials for the reformer tubes, and new

sulfur passivated catalysts which inhibit carbon formation.

In order to produce synthesis gas (syngas), four reforming processes including two stand-alone primary and secondary reformers and two combined configurations were investigated by Ebrahimi et al. [19]. With changing operating parameters and arrangement of the reformers (i.e. stand-alone, parallel, and series), the syngas may be obtained for different applications such as methanol, Fischer-Tropsch (FT), and ammonia synthesis. The Non-Sorting Genetic Algorithm II (NSGA-II) optimization method was applied by them for the problem based on the practical point of view. They found that the series configuration consumes lower fuel (361.1 kmolh^{-1}, in comparison to the parallel case with 437.2 kmolh^{-1}) and releases lower amount of CO_2 emission. It is reported that the series arrangement has an average of 13.2% of released CO_2 molar flow less than that of parallel arrangement [19].

Singh et al. [20] described the failure of the pressure shell of an interconnecting pipe between the secondary reformer and primary waste heat boiler that led to a fire in Ammonia plant.

The fire incident resulted the shutdown of the plant for 15 days. Details about the inspection and rehabilitation have been described with photograph of ruptured pipe.

They concluded that the failure of the pressure shell was a result of localized refractory damage and short-term high-temperature stress rupture.

Due to the rise in temperature and internal gas pressure, hot tensile rupture had occurred.

The failure of refractory might be due to repair work done during the turnaround at the boiler inlet nozzle area, or voids and cracks in the refractory in that region that might have gone unnoticed. Based on analysis of failure, they have taken necessary measures to prevent such occurrence.

Jahromi et al. [21] presented the case of failures of an old waste heat boiler tubes at the downstream of secondary reformer in their ammonia plant. On-site inspections revealed that the highest degree of burning, oxidation and damage had occurred in the lowest part of the tubes, where maximum heat flux is applied and the highest rate of evaporation exists.

Deformations and failures mostly occurred at the lower parts of the tubes, where the higher temperature of the flue gases results in maximum heat and evaporation.

They concluded that the overheating was the main reason for failure due to creep.

Overheating was mostly due to the formation of a thick deposit layer. Chemical analysis of deposits revealed 90% Fe_2O_3 which might have been due to corrosion by different mechanisms or remnants of chemical cleaning of the boiler.

With increasing metal temperature, creep becomes much faster leading to tube failure under internal pressure.

To prevent this type of failure they recommended the reconsideration of chemical cleaning, water treatment and corrosion protection programs as well putting a filter.

II.2. Stage 2 (Synthesis Gas Purification)
II.2.1. Shift Conversion

The secondary reformer is usually followed by two water gas shift (WGS) reactors to convert carbon monoxide into carbon dioxide (which is used later in the synthesis of urea).

$$CO + H_2O \longleftrightarrow CO_2 + H_2, \quad \Delta H = -9.8 \text{ kcal/mol}.$$
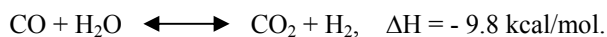
A high temperature shift over iron oxide/chromium catalysts at 400^0C is followed by a low temperature shift at 200^0C on a copper-based catalyst to attain favorable equilibrium in WGS. The same reaction occurs in both steps, but using the two steps maximizes conversion.

The shift conversion involves two stages. The first stage employs a high-temperature catalyst, and the second uses a low-temperature catalyst. The shift converters remove the carbon monoxide produced in the reforming stage by converting it to carbon dioxide by the reaction as given above.

The reaction produces additional hydrogen for ammonia synthesis. The shift reactor effluent is further cooled to 40^0C at which temperature the water condenses out and is removed. Both WGS stages have superficial contact times greater than 1 s [18]. The syngas next goes to absorber for removal of CO_2.

II.2.2. CO₂ Removal

The synthesis gas is purified by removing carbon dioxide by absorption with hot potassium carbonate, Selexol, or methyl ethyl amine (MEA) which is regenerated and circulated back to absorber. CO_2 content in the synthesis gas must be reduced to 5 to 10 PPM by volume.

The most commonly used physical absorption process is Selexol. The chemical absorption process can be classified in three main categories; the hot potassium carbonate process, the alkanolamines process and other chemical absorption process. Commercially available Hot potassium carbonate processes are Benfield process, Glycine Vetrocoke process and Catacarb process. In Alkanolamines processes, commonly used solutions were monoethanolamine (MEA), diethanolamine (DEA) etc. In recent years most preferred solution is activated Methyl Diethanol Amine (a-MDEA) [22].

The most part of the solution can be regenerated by simple flashing, leading to very low energy consumption. After water removal, pure CO_2 is sent directly to the urea plant for compression and use in urea synthesis.

The Benfield process uses a chemical absorbing solvent based on 30% potassium carbonate (K_2CO_3) in water plus an activator and corrosion inhibitor. For many years, DEA (diethanolamine) has been the standard

activator and is still used today in many operating plants, but now it is being replaced by ACT-1.

There are corrosion problems in CO_2 removal system. Where sour gas is removed by adsorption in media like aqueous amine (DEA/MEA), sulphinol or potassium carbonate solutions (Benfield and Catacarb), problems can appear in coolers, condensors and reboilers. It involves rapid localised corrosion of carbon steel vessel walls by CO_2 containing process solution. The possible root causes are (1) inadequate concentration of anodic inhibitor, (2) the presence of hydrocarbons, (3) process conditions of high gas loading and/or frothing/ foaming, (4) formation of a sulfide layer and (5) galvanic interaction. Harjac et al. [23] presented a summary of a desktop review, using the Six Sigma methodology, of root causes for corrosion incidents in Hot Potassium Carbonate acid gas removal plant.

Today, 304L and 316L are used, as carbon steel has unsatisfactory service life. Even 304L and 316L may fail. It is then important to establish the type of failure to identify the root cause of failure [24].

Pande and Tonheim [25] analysed the explosion in the transfer pipeline for CO_2 gas from Ammonia Plant to Urea plant which reulted no injuries, but 850 meters of the line was totally destroyed. This is somewhat a unique combination of so many parameters/ conditions together which, generally, has rare chance of occurrence. The real cause was the ignorance of the presence of hydrogen in the line. The line was temporarily out of service, and the investigation team concluded that:

1. Trip system had been disabled prior to the explosion,
2. Hydrogen enriched gas had entered the pipe line,
3. Nitrogen purge had not been effective,
4. Air had leaked into the line and formed an explosive mixture and
5. Mixture had ignited.

They insist that the process knowledge must not be limited to "normal operation" or "design condition," but include understanding of possible deviations, especially during start-up and shutdown. It seems unbelievable but a painful surprise/ reality. Independently from each other, each group in operation, maintenance, and process should have asked questions and taken action. Interdisciplinary communication and close cooperation is vital. It has highlighted a unique problem which will help others to avoid similar pitfalls.

II.2.3. Methanation

Methanator is an adiabatic fixed reactor (purifying unit), preceding the ammonia converter and is necessary for the removal of traces of CO and CO_2 in the syngas before they enter the ammonia converter. Since syngas contains small amount of CO and CO_2 which are poisonous to the ammonia synthesis catalysts and removed in methanator. It acts as a guard for the ammonia synthesis catalysts against CO and CO_2 poisoning.

The remaining CO_2 as well as CO are converted to methane (methanation) using a Ni/Al_2O_3 catalyst at approximately 325^0C (reverse of primary reformer reaction, hence same catalyst for forward and backward reactions) as per the following reactions:

$$CO + 3H_2 \longleftrightarrow CH_4 + H_2O$$
$$CO_2 + 4H_2 \longleftrightarrow CH_4 + 2H_2O$$
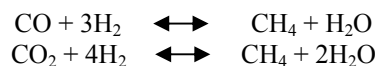
The water which is produced in these reactions is removed by condensation at 40^0C. The resulting methane is inert. The purified hydrogen-nitrogen mixture with the ratio of $3H_2$: $1N_2$ is compressed to the pressure selected for ammonia synthesis.

Because of the highly exothermic nature of the methanation reactions and the potentially large quantities of CO and CO_2 that can be supplied to the reactor, methanation reactor is potentially one of the more significant hazards in a conventional ammonia plant.

Many accidents in the petrochemical industries are due to temperature runaways. The most difficult and dangerous of these runaway problems are the ones which are not caused by disturbances local to troubled units, but are due to problems associated with upstream units (or downstream units when there is a recycle).

The situation becomes even more complicated when the proper correction needed is not a local parameter to the troubled unit. In such situations, certain thoughts of corrective action may have an opposite effect, complicate the problem further and lead to an accident, or lead to an emergency shut-down which is usually quite expensive [26].

When the catalytic reaction is highly exothermic, the reactor becomes liable to temperature runaway. In recent years, an actual accident in an ammonia production line happened [26]. Disturbance introduced into the shift converters section of the ammonia production line may lead to problems in the ammonia production line which manifest themselves in other units of the production line.

The drop in activity of the catalyst due ageing in high temperature shift converter (HTS) and low temperature shift converter (LTS) causes an increase in the amount of CO leaving the shift converter section. CO is not absorbed in the CO_2 absorption unit and, hence, the CO entering the methanator increases, causing more CO methanation reaction, which is a highly exothermic reaction and, therefore, the temperature of the methanator (which is an adiabatic reactor) rises. Alhabdan and Elnashaie [26] developed a model based on a real incident and simulated for effect of various types of disturbances introduced to the shift converter's section. The effects of these disturbances on the methanator have been reported. They demonstrated, in a realistic manner, the sequences of events leading to temperature runaway in the methanator due to these disturbances in the HTS and LTS. The results are very valuable for understanding this complex interaction between the units and finding

out the best operator response to avoid a shutdown or an accident.

Normally, HTS operates very close to its optimum feed temperature therefore increase and decrease in feed temperatures will cause the conversion to decrease resulting in higher CO leakage from the shift converter passing unaffected through the CO_2 absorption unit, thus causing higher temperatures in the methanator. It is important to notice that the moderate temperature increase in the methanator as a result of this disturbance is due to the fact that the LTS still operate as a guard for the extra CO leakage from the HTS.

The protective system that was generally installed in old methanators did not reflect the relatively high risk of overheating this vessel with potentially catastrophic consequences [27]. In addition many older plants had Methanator vessels that were constructed of a material that has proven unsuitable for long term operation in high temperature Hydrogen service and as a result are suffering high temperature hydrogen attack (HTHA) and consequent cracking. Using better material of construction (MOC) and providing additional layers of protection in both the initiating and final elements of the instrumented protection system can greatly reduce the risk of a serious failure of this vessel and contribute to a safer Ammonia plant.

II.3. Stage 3: Compression Process

The purified synthesis gas is cooled and the condensed water is removed. The syngas is then compressed in centrifugal compressors with interstage cooling to a pressure of approximately 150-300 bar. The centrifugal compressors are driven by steam turbines using steam generated in the plant itself. This reduces the overall power consumption.

Krivshich et al. [28] discussed the application of dry gas seal (DGS) systems which makes it possible to maximize the reliability of seal systems of compressors. Absence of systems for lubrication of oil seals significantly increases operational fire safety. Instead of laborious and time-consuming servicing of oiling systems of seals (oil change, maintenance of pumps and accessories, etc.) and additional costs arising from unscheduled shutdowns, dry gas seal systems need simple scheduled work every 3–5 years during uninterrupted operation.

The compression of syngas is done in a multistage centrifugal compressor. Interstage cooling is done to reduce gas temperature between stages. In ammonia plants, duplex stainless steels (refers to the two-phase microstructure containing roughly equal amounts of austenite and ferrite) have been successfully used in many heat exchangers operating up to 600^0C in cooling water polluted by chlorides. The duplex stainless steels offer a safe and cost-efficient alternative to carbon steel and stainless steels in the 300 series. The performance of older plants gives indications of where the problems usually turn up. The initial cost for better material might be higher, but it is always more costly to replace it later with a more corrosion resistant material. In ammonia plants the duplex grades have proven to be a good solution for waterside corrosion at a low cost [24].

II.4. Stage 4: Synthesis Loop

The compressed synthesis gas is dried, mixed with a recycle stream, and introduced into the synthesis reactor after the recycle compressor. The gas mixture is chilled and liquid ammonia is removed from the secondary separator. The feed mixture is preheated inside the converter prior to entering the catalyst bed. The reaction occurs at $450–600^0C$ over reduced iron oxide catalyst. The ammonia synthesis reaction between nitrogen and hydrogen is given below.

$$N_2 + 3H_2 \longleftrightarrow 2NH_3 + Heat$$
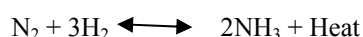
The above reaction is an equilibrium reaction that is exothermic. Lower temperatures favor the production of ammonia. High pressures in excess of 21 bar are required to achieve sufficient conversion. Conversions of 20%–26% ammonia per pass are achieved. However, the conversion of hydrogen per pass is still less than 30%, therefore, the process requires a large recycle of unreacted gases. The converted vapor product is cooled by ammonia refrigeration in the primary separator to condense the ammonia product. A purge is removed from the remaining gases to prevent the buildup up of inerts (in particular, CH_4 and Argon) in the synthesis reactor.

The ammonia is rapidly decompressed to 24 bar. At this pressure, impurities such as methane and hydrogen become gases. The gas mixture above the liquid ammonia (which also contains significant levels of ammonia) is removed and sent to the ammonia recovery unit. This is an absorber-stripper system using water as solvent. The remaining gas (purge gas) is used as fuel for the heating of the primary reformer. The pure ammonia remaining is mixed with the pure ammonia from the initial condensation above and is ready for use in urea production, for storage or for direct sale.

Ammonia process licensors are employing new technologies that can be retrofitted to existing plants to increase the capacity by 20%–40%. A wide range of newer more reactive catalysts are now replacing the iron-based catalysts. These catalysts are found to be advantageous in operating at lower synthesis pressures. Iron-titanium metals, ruthenium alkali metals, or ruthenium promoted by potassium and barium on activated carbon have exhibited high efficiency. Additionally, phosphorus, sulfur, and arsenic compounds tend to poison the catalyst in the subsequent reaction. M. W. Kellogg employs a ruthenium on graphite as the catalyst on Kellogg Advanced Ammonia Process (KAAP) [6]. Process technology licensors have developed alternative techniques to the primary and secondary reformer processes. These technologies integrate process units with steam and power systems,

thereby using heat exchange networks to capture waste heat. Additionally, they provide the energy required for reforming methane.

Hayes [29] reviewed the failures of six pressure vessels- Sizewell boiler (1963), John Thompson ammonia converter for ICI Immingham (1965), Cockenzie power station boiler drum (1966), Typpi Oy heat exchangers (1970), Robert Jenkins pressure vessel (1970) and the Union Oil Co. amine absorber tower (1984).

Anwar et al. [30] discussed the failure of expansion bellows of a synthesis loop heat exchanger, which could be due to hydrogen attack combined with fatigue loading. Shah and Zhu [31] provide the design of ribbon wound pressure vessels useful for Ammonia, Urea and Methanol plants. Of particular interest has been the application of this technology in the Ammonia and Urea plants, where the design allows fabrication of these vessels at substantial reduction in cost, and early delivery, when compared to the mono wall technology.

Sutherland et al. [32] described a piping failure and fire resulting from inadequate support of an ammonia synthesis loop pressure relief valve and the pipe failure mode. They also addressed support criteria for relief valves and other piping systems illustrating the importance of evaluating both static and dynamic loads.

With regard to recovery of the hydrogen in the purge gas from the ammonia synthesis loop, two methods are used now days in ammonia plants [33].

In first case hydrogen is recovered in a membrane unit. The driving force used for the hydrogen to permeate the membrane is pressure difference. The hydrogen-rich stream is recovered at a lower pressure. The recovered hydrogen is added to the make-up gas upstream the synthesis gas compressor. In second case, cryogenic separation is used for recovery of hydrogen from the purge gas. The operating principle is to liquify and separate the inerts (nitrogen, methane and argon) from the hydrogen. As the hydrogen-rich stream is recovered at high pressure it can be recycled to the ammonia synthesis loop between the two casings of the synthesis gas compressor. The recovery based on cryogenic technology is more energy efficient, whereas the recovery based on membrane technology requires a lower investment. Which technology to choose therefore has to be decided on economic evaluation of the separation for a given case.

Gases purged from the ammonia synthesis loop and gases collected during ammonia decompression are mixed and sent to the ammonia recovery system. Ammonia is recovered by absorption in cold water in the counter-current column where gas mixture is introduced at the bottom of absorption tower.

Approximately 96% of the ammonia in the gas is recovered, leaving a gas mixture that is used as a fuel gas to heat the primary reformer.

The ammonia is distilled out of the ammonia water mixture, condensed and pumped to join the rest of the ammonia from the ammonia synthesizer.

II.5. Ammonia Storage

Anhydrous ammonia is usually stored as a liquid in refrigerated tanks at -33.3^0C and atmospheric pressure, often in doubled-walled tanks with the capacity for hundreds or thousands of tonnes [34]. Normally low temperature is maintained by the venting of ammonia gas. The vented gas is reliquefied for recycling, or absorbed in water to make aqueous ammonia. Sometimes small quantities of anhydrous ammonia are stored under pressure in spherical vessel at ambient temperature [35], [36].

Liquefied ammonia has a high level of dispersive energy. This is partially due to its relatively high vapour pressure [35].

In case of large amount of liquid ammonia have been released, temperature will decrease due to evaporation of the liquid. However, the rate of conduction of heat from the surroundings generally maintains the temperature of the ammonia pool sufficiently close to its atmospheric pressure boiling point, to enable continual evaporation [37], [38].

The rate of dispersal of ammonia clouds is highly dependent of environmental conditions including wind speed and humidity. Even after large releases of ammonia, it is possible for levels of ammonia vapour in air to return to background levels within a matter of hours. Therefore, concentration of ammonia in the air measured just hours after a release do not give the correct measure of the concentration present in the air at the time of the release [39], [40].

Al-Abdullally et al. [41] questioned the construction of a concrete bund wall around the ammonia storage tanks, which is not justified. Instead, it would be adequate to upgrade the safety in the common dyke around the tanks by adopting additional conventional methods.

The discipline of putting a minor modification through a formal design verification system particularly for aged vessel and equipment is very important. Such an instance was discussed by Shipman and Davies [42] which had helped them to identify that an existing ammonia storage vessel (20 years in service) was unfit for further service and decided to replace it with new tank with improved design. Similarly, Paltabathuli et al. [43] had done in-depth hazards review and subsequent installation of overpressure protection system on a 30-year-old ammonia storage tank and other hardware and software modifications which have improved the reliability of pressure systems in the ammonia storage area.

Periodic inspection of storage tank is very important and inspection frequency to be increased in case of older vessel. Sometimes uneven settlement of the tank foundation after long period increases the stress and results in stress corrosion cracking (SCC) [44].

Christou et al. [45] discussed the 'Seveso II Directive' requirements for Land-Use Planning with respect to major accident hazards. They have taken Ammonia storage facility as reference for understanding and comparison of the analysed approaches. The approaches analysed were grouped into three broad categories,

namely, establishing 'generic distances', 'consequence based', and 'risk based'.

McGowan [46] justified indefinite operations of an ammonia tank without the need to inspect internally for tanks having advanced safety feature. A risk-based inspection can be performed which gives the probability of failure and confidence level for ammonia storage tank can be used further for period without internal inspection. The future integrity of the tank can be evaluated by a properly conducted acoustic emission test instead of an internal inspection [47].

Abdulla and Narasimha [48] have described the successful inspection of two large ammonia storage tanks which were in operation for more than fifteen years.

The discovery of SCC in a refrigerated ammonia storage tank has been described by Byrne et al. [49]. The acoustic emission testing had detected no SCC. Inspection performed using magnetic particle inspection discovered SCC. The SCC was found to occur mainly at the site of internal cleats and was attributed to defective welding procedures, the effect of which was aggravated by the use of high strength steel. A detailed account of the treatment of defects in this tank has been given by Selva and Heuser [50].

II.6. Accidental Releases of Ammonia

II.6.1. Toxicity of Ammonia

Ammonia is a material of special importance in hazard control, because of its toxicity and its very widespread use. An additional factor is its ability to form denser-than-air mixtures on release to the atmosphere, in spite of its low molecular weight relative to that of air.

The threshold limit value (TLV) for ammonia is 25 PPM (TWA), short term exposure limit (STEL) 35 PPM and IDLH 300 PPM [51]. Ammonia gas is extremely corrosive and irritating to the skin, eyes, nose, and respiratory tract. Exposure by inhalation causes irritation of the nose, throat, and mucous membranes. In case of low concentration (<TLV) no relationships between level or length of ammonia exposure and lung function results were demonstrated [52]. At 400-700 PPM severe eye and respiratory irritation can occur, with the potential for permanent damage.

At 1700 PPM convulsive coughing and bronchial spasms occur, and half hour exposure to this concentration is potentially fatal. Exposure to high concentrations (above approximately 2500 PPM) can be life threatening. At 5000-10000 PPM, death can occur from suffocation [36], [52].

Eyes should be protected from contact with ammonia vapour which is severely irritating as well as liquid ammonia or mists which can result in serious damage or permanent eye injury and blindness. Contact with the liquid results in cryogenic burns.

Ammonia is not a carcinogenic substance or to show reproductive or developmental toxicity in humans [35], [53].

Michaels [55] has done the critical evaluation of proposed AEGIS for ammonia. Acute Exposure Guideline Levels (AEGLs) for ammonia are critically evaluated. The technical bases for concern about AEGL-2 and AEGL-3 values derived by the committee are summarized and recommendations made by them.

II.6.2. Dispersion in Atmosphere and Large Industrial Releases

Ammonia is corrosive to many metals and alloys particularly those of copper and zinc. Iron and steel are generally used in ammonia storage tanks, piping and fittings [36]. But under certain conditions and with few specific steels, anhydrous ammonia is liable to produce embrittlement [35]. It is important and necessary to follow strict guidelines otherwise equipment failure may lead to large release of ammonia liquid or vapour. Ammonia vapours can disperse extremely quickly when released. These vapours can be highly toxic by inhalation at high concentration as already mentioned. Flashing of ammonia takes place in case of loss of containment particularly where ammonia is stored under refrigerated conditions or as a liquefied gas under pressure. The proportion flashing depends on temperature conditions [35]. Though ammonia is less dense than air, there is evidence that some ammonia/air clouds formed by flashing have been denser than the surrounding air, resulting in ammonia clouds at ground level. Observational and theoretical evidence is reviewed by Griffiths [54] that demonstrates the formation of such mixtures. It is shown that the combination of factors determining the density of the mixture is highly dependent on the mode of containment and the mode of containment-failure; the physical properties of ammonia and the meteorological variables also have an important influence. A compilation of accidental releases of ammonia is included by them.

Ammonia gas can be safely vented to atmosphere during emergency situations and plant upsets as suggested by McConnel and McGrath [56], provided it is in gaseous form, and that the discharge point is sufficiently high to prevent the downwind plume hitting the ground before the ammonia concentration has diluted to insignificant levels. Suitable measures, including adequately sized liquid knock-out pots, are required to prevent liquid or liquid droplets being discharged to atmosphere because the plume is then no longer buoyant, and the ammonia plume will hit the ground much earlier with a much higher concentration [56].

Thyer [57] has summarized the availability and utility of experimental data on the rates of spread and vaporisation of spillages of pressure liquefied and cryogenic liquids on a variety of surfaces including: soil, sand, concrete, and water.

There are number of large scale spillage/ releases of ammonia which had occurred from various industrial activities. It had varying impacts on humans and the

environment, including aquatic systems which may be sensitive to ammonia at high concentration.

A release of 19 tonnes of ammonia from a road tanker in Houston, Texas in 1976 resulted in 6 fatalities and more than 100 injuries. Just after 2.5 hours of the incident, air levels of ammonia had returned to background levels. It had amply demonstrated the inability of air measurements taken only hours after a release to give an accurate measure of the levels present in the air at the time of the release [39], [40].

There have also been a number of non-fatal releases of ammonia. A large spill of 600 tonnes of ammonia from a burst pipe in Floral, Arkansas, USA in 1971 did not result in any human fatalities. However, the released ammonia reached a watercourse and killed thousands of fish [59]. Due to an error warm ammonia transferred to the tank from bottom where it formed a layer on the bottom. In due course this layer rose suddenly to the surface like 'rollover'. The higher vapour pressure of the warmer liquid caused a sudden rise in pressure in the tank, which the pressure relief valves were unable to handle, and the tank burst.

A list of ammonia accidents has been given by Marshall [60] which may be referred for old data. There was a release of 160 ton of ammonia from a storage tank at Blair, Nebraska, in 1970, but there were no casualties [61]. The overfilling was the result of an operator error due to improper indication of level. In addition, the high-level alarm and shut-down system failed to operate and apparently the overflow discharge valve also failed to operate at the set pressure, so that the liquid level in the tank rose until it reached the roof, at which point the overflow valve did open. There was an isolation valve on the overflow line just below the relief valve. But it was not possible to reach and close the isolation valve because of ammonia cloud. If isolation of the overflow line had been possible, much of the overflow could have been prevented. The discharge continued for 2.5 h. A low visible cloud had formed and later moved by a light breeze and helped to keep the cloud away from populated areas [61].

Brittle fracture is catastrophic, since the fracture can propagate at a velocity close to that of sound. In 1973 at Potchefstroom, South Africa, an ammonia tank suffered brittle fracture [39], [58] which resulted in the total release of some 38 ton of ammonia which resulted 18 deaths. It appears to be the worst accident involving ammonia. There were no overpressure or over temperature of the tank contents and no other triggering event was determined. The fracture occurred in a dished end which was fabricated in carbon steel and which had not been stress relieved after manufacture. Subsequent tests were conducted and found the minimum transition temperatures 20^0C for the fragment and 115^0C for the remaining part of the dished end. Thus, the metal was below its transition temperature under normal operating conditions.

The failure resulted in rise of a gas cloud of approximately 150 m diameter and 20 m deep. At the time of the accident the air was apparently still, but within a few minutes a slight breeze arose which caused the cloud to move towards a township.

It had prompted the South African authorities to lay down guidelines that all vessels containing dangerous substances shall be given appropriate heat treatment irrespective of the (construction) code requirements.

Commenting on this Lonsdale (1975) [58] states "Stress-relieving does not overcome fully the damage done by progressive cold-forming of a dished end. This is particularly so where seam welds has had to be made in the dished end." [62], [58].

There are large numbers of recorded ammonia releases in excess of 100 tonne but much lower casualties. In 1989 a refrigerated atmospheric ammonia storage tank containing some 7,000 te of liquid ammonia failed at Jonova, Lithuania [63]. The ammonia ignited and due to domino effect ammonium nitrate storage caught fire. It was estimated that some 1400 te of ammonia evaporated and further 700 te of ammonia and nitrous fumes participated in the toxic fire plume. The plume had affected an area of some 400 km^2. Seven people were killed 57 injured. The tank with 29 m dia and 20 m high with single wall construction and perlite insulation held in place by an outer steel shell and with a surrounding 14 m high reinforced concrete wall. The pool of ammonia had taken 12 h to evaporate completely whereas fertilizer continued to burn for 3 days.

The occurrence of stress corrosion cracking in ammonia storage tanks/spheres has created a considerable problem in the maintenance of such storages. Accounts are given by Brown (1982) and Guth and Clark (1985) [64], [65]. They illustrate both the procedures for isolation, emptying, purging and entry, and for bringing back on stream for such storages and the procedures for inspection and repair.

III. Safety/Risk Analysis of Ammonia Plant

Probabilistic safety assessment (PSA) in chemical installations is generally performed using state-of-the-art methodology. Papazoglou et al. [66] have described the procedures and corresponding methodologies for PSA and demonstrated on a refrigerated ammonia storage facility. The procedural steps comprise hazard identification, accident sequence modelling, data acquisition and parameter estimation, accident sequence quantification, hazardous substance release categories assessment, consequence assessment and integration of results.

In another article Papazoglou et al. [67] presented a computer package DECARA for the integrated risk assessment of accidental releases of hazardous substances. It provides an integrated risk analysis including source-term strength evaluation, estimation of the hazardous cloud dispersion and quantification of health impacts. In case of multiple accidents, each with a certain probability of occurrence can also be handled. DECARA can be used for simulation of dispersion of

heavier as well as lighter-than-air gases released instantaneously or continuously.

McConnell [68] has felt the need to re-examine the vulnerability of control rooms and other occupied buildings of the existing plants. They described "Risk Management Plans for Existing Control Rooms" with specific application to ammonia and similar plants.

Vapor cloud explosions have occurred on ammonia plants due to sudden release of hydrogen-rich synthesis gas followed by a delay in igniting the released jet of gas. However, an analysis of incidents by Hawksley [69] indicates that unconfined hydrogen vapor clouds have unusual characteristics. Since the flammable region is high 4 to 74% v/v and large clouds of gas are unlikely to form due to the rapid diffusivity in the atmosphere therefore quantity of gas in the cloud is relatively small. There appear to be no recorded major damage explosions resulting from unconfined or semi-confined releases of hydrogen-rich gas on ammonia plants. However, confined explosions such as in enclosed compressor houses have caused very serious damage. High energy releases are unlikely to fill large structures in an ammonia plant before ignition occurs.

Williams et al. [70] have analyzed the causes for ammonia plant shutdowns in their survey (V). They have given data on major equipment failures causing shutdowns and down-time.

These data are tabulated based on area-wise and equipment–wise failures, avoidable and unavoidable shutdowns etc. The on-stream factor for large tonnage ammonia plants has decreased to 80.8 percent and almost fifty percent of total downtime was mainly due to inventory control and feedstock curtailment. Majority of failure results in fire which have been given in the Table I and classified based on source of failure resulting in fire. We can take the inference from these data that the weakest point related to failure resulting in accident is mainly associated with the combined failure of flanges and valve packings ($\approx 40\%$).

TABLE I
CLASSIFICATION OF FIRES (%) EXTRACTED FROM [70]

Location	Survey Period		
	1973-1976	1977-1981	1982-1985
Flange	36	32	31
Valve Packing	8	4	8
Flange and Valve Packing combined	44	36	39
Oil Leak	20	29	19
Transfer Header	7	-	1
Piping	10	9	11
Electrical	2	3	3
Miscellaneous	17	23	27

Williams [71] has done the survey on safety performance in ammonia plants. Safety incidences

including fires and explosions were reviewed by him. This recent safety performance is compared with the performance history from past surveys as given in Table II. It is evident that there is room for improvement in all aspects of safety. It is encouraging to note that more plants are operating without any fire incidents. It is recommended that operators review the explosion/rupture incidents and assess the exposure in their own plants.

TABLE II
HISTORY OF AMMONIA PLANT FIRES EXTRACTED FROM [71]

Survey Period	1973-1976	1977-1981	1982-1985	1994-1996
Total no. of Fires	125	257	520	215
Plants having no fires	2	22	41	35
Plants having fires	27	74	95	50
Total Plants Reporting	29	96	136	85
Frequency, in months	11.1	14.6	12.2	14.2

Remark: The number of plants reported in this survey is much lower compared to previous one. The above data are mainly from plants located in Europe and American continent.

The Dow Fire and Explosion Index (F&EI) is universally used in evaluating the hazard category of a process plant, area of exposure, expected losses in case of fire and explosion, etc. The effects of the loss control measures (LCMs) on the F&EI value are not taken into account which makes the plant look more hazardous, makes it more spread out, requires more elaborate emergency measures and alarms the public and the civil administration more than is necessary. Gupta et al. [72] suggested taking the effects of the LCMs into account in the F&EI value which they termed as 'Offset F&EI' value.

Papazoglou et al. [73] have developed an integrated quantitative risk assessment method for hazardous installations, taking into account management as well as technical design and producing risk level measures, called I-Risk. The key components of the I-Risk methodology are the technical model, the management model and their interface.

Shiryaeva et al. [74] have indentified two groups of maximum undesirable events as potential hazards for an ammonia plant - unplanned shutdown in the ammonia synthesis and possible accidents (explosion, fire, and staff poisoning).

In chemical industries accidents involving heat/ mass/ momentum transfer may result in explosions, fires, and toxic releases. Risk assessment tool and techniques are often employed in order to identify hazards and to forecast potential disasters. These efforts are generally focused on single events or 'stand alone'' accidents.

Khan and Abbasi [75] have studied the domino effect i.e. interaction of an accident with other hazardous units and developed models and computer-automated tools to study chain of accidents or "domino effects" and

presented an illustrative case study of a major fertilizer industry. A detailed consequence analysis, by them, covering all the hazardous units reveals that, as far as the primary event is concerned, ammonia converter has the maximum damage potential as it causes lethal overpressure and toxic load over the largest area as well as due to the cumulative effect of the primary and secondary accidents or domino effect.

Mason et al. [76] discussed the methodology used for modernization of an ammonia plant safety shutdown system.

Idrees and Aslam [77] discussed the most important aspect of prevention of major accidents and failures with well-trained and empowered operators based on their experience at ammonia plant involving the prevention of major failures of the synthesis gas compressor turbine by taking prompt action to shutdown the compressor. As per their assessment continuing to run the turbine may have resulted in a safety incident or an even longer plant shutdown for significant repairs. They also described the techniques used by the operation team to train and empower their people. A well-trained and empowered operator is always helpful by making prompt decisions to shut down equipment, rather than relying solely on instrumentation to protect equipment.

Philosophy of inherently safer design (ISD) can be applied not only at the early stages of process development and design, but there are opportunities throughout the life cycle particularly where modifications or other changes takes place in the process [78]. Many plants significantly reduced the quantity of toxic intermediates stored in existing plants in the 1980s (prompted by the Bhopal tragedy) by focusing on the reliable operation of the plants, so that it was not necessary to maintain a large inventory to keep other parts of a plant running when an unreliable plant unit shut down. For ISD it requires a thorough understanding of the process and creative engineering to address the problem of how to eliminate or significantly reduce hazards. The literature on inherently safer design can help in identifying these opportunities.

IV. Other Important Studies

Shah and Weisenfelder [79] have discussed the control and optimization of a large ammonia plant with a digital computer. Digital computer control of a single train, large capacity ammonia plant with an IBM 1800 was described by them which were way back in 1969.

Khan and Kabir [80] describes a simulation modeling technique for assessing the availability of an ammonia plant using field data on failure and down times which can be collected and analyzed by fitting Weibull distributions. They estimated the plant availability using the existing plant configuration and actual failure and repair time distributions. The possibility of improving plant performance by modification of plant configuration and change of overhaul strategy was also been studied by them.

McConnell [81] has stressed the need for use of Slam Shut Valves instead of safety relief valves for certain important relief duties. Their 15 years of operating and maintenance experience on the 2 plants at ICI's Severnside factory have justified the decision to use Slam Shut Values. They use an alternative approach of pressure containment rather than pressure relief as applied to ICI's Leading Concept Ammonia plants at Severnside Works in England. The valves have proved reliable in operation. Nevertheless the original concepts of less emission to atmosphere following plant trips, and more rapid plant start-ups were achieved, without compromising the safety integrity of the pressure relief systems.

Scheerder [82] has shared his experiences with non-destructive testing of static equipment in ammonia plant. Ultrasonic inspection method is presented with respect to specific failure modes in ammonia plants. Creep damage in reformer tubes is detected by ultrasonic method of NDT.

Lababidi et al. [83] has presented a case study in which energy saving can be achieved by retrofit in an ammonia plant when energy integration study using Pinch technology does not provide any possible benefit. Alternatively, the retrofit study concentrated on better placement of available utilities. However, they recommend that the economical evaluation of the proposed modifications must be considered before implementation.

Morrison et al. [84] has discussed common cause of the fire and explosion in an improperly inerted vessel. Fires and explosions can and do occur in inerted vessels. Vessels that contain or have contained flammable vapors are generally inerted for many reasons, but one of the most common is explosion prevention. Main inerting gases are carbon dioxide, nitrogen, steam, and air; depending upon the requirement & application. Causes for such incidents ranged from procedures to design issues. However, rigid adherence to safety standards may not be sufficient to prevent an accident. Some specific recommendations for preventing explosions include methods for improved mixing of the inert gas, the use of blinds, filling the vessel with water, improved work procedures and improved monitoring procedures [84]. For example- supply flow rate of inert gas must meet or exceed the design objective, stagnant zones must be minimized and degree of mixing in the vessel should be determined through atmospheric monitoring.

Babic and Losso [85] studied the reasons for the explosion of an auxiliary boiler occurred in the process of lighting, during plant start-up. Explosion happened because the manual valves for burners were not sufficiently closed and that they allowed larger quantities of gas into the boiler; moreover, the dampers were not sufficiently open for air and the flue gas.

There are large numbers of ammonia plants which are quite old and needs retrofit to improve the energy efficiency. Panjeshahi et al. [86] conducted one such study by dividing whole plant into two parts: the hot-end

and the cold-end, applying pinch and exergy analysis. It can result in a considerable reduction in cooling water, HP steam, fuel gas consumption, reducing the amount of shaft work or power consumption in the refrigeration system is achieved.

There are many hazardous wastes generated in chemical plants. In ammonia plant, apart from other hazardous waste, disposal of spent catalyst is a problem as it falls under the hazardous industrial waste. The recovery of metals from these catalysts is an important economic aspect as most of these catalysts are supported, usually on alumina/silica with varying percent of metal; metal concentration could vary from 2.5 to 20%. Many workers have adapted pyrometallurgy and hydrometallurgy process for recovery of precious metals. Many workers have studied the recovery of nickel from a spent catalyst in an ammonia plant by leaching it in sulphuric acid solution (Hydrometallurgy). The various aspects of treatment of spent catalyst were reviewed by Singh [87].

There are instances when new and special type of problems / hazards appears which looks impossible to materialize in reality. Such events are eye opener for everyone concerned to remind that never be complacent with safety and follow all procedures to prevent accidents. An excellent account of observation, action taken and preventive measures for vibration of self-supported flare-stack in ammonia plant is given by Stevens and Desai [88].

Excessive vibrations/shaking of the flare and flare structure due to mixing of NH_3 and CO_2 rich gases in the presence of water were observed by plant personnel. The partial blockage increased the pressure at the flare-stack bottom and the sudden release of the gases gave vibrations (surging effects) to the flare stack. It took three days to clear the blockage without interrupting the process and vibrations completely stopped. No vibrations were observed during all normal/emergency plant shutdowns faced after the incident. They described the root cause of unique experience of flare stack vibrations, and the way the problem was solved online.

Kletz [16] had described few reasons for failures in his classic book "What Went Wrong". Many failures have occurred because the wrong grade of steel was used for a pipeline. Normally correct grade is specified, but because of some reasons the wrong grade is delivered to the site or selected from store. The most spectacular failure of this sort occurred when the exit pipe from a ammonia converter was constructed from carbon steel instead of 1.25% Cr, 0.5% Mo. Hydrogen attack occurred creating a hole at a bend. The hydrogen leaked out, and the reaction forces pushed the converter over. Such incidents have alerted many companies insisted for checking all the materials for composition before use. This applied to flanges, bolts, welding rods, and the like, as well as the raw pipe.

In one of the cases, a gas leak occurred through the weep hole in a multiwall vessel in an ammonia plant. The plant stayed on line, but the leak was watched to see that it did not worsen but unfortunately vessel disintegrated after ten days, causing extensive damage. The multiwall vessel was made from an inner shell and multi layers of wrapping, each drilled with a weep hole. The disintegration was attributed to excessive stresses near a nozzle but it was not recognized when the vessel was designed [16].

A return bend on a furnace failed after 20 years of service. It was then found that it had been made from carbon steel instead of the alloy specified [16].

Checks carried out on the materials delivered for a new ammonia plant showed that 5,480 items (1.8% of the total) were delivered in the wrong material. These included 2,750 furnace roof hangers; if the errors had not been spotted, the roof would probably have failed in service [16].

Ammonia explosions are not common, as the lower explosive limit (LEL) of ammonia is unusually high (LEL-16%, HEL-25%). In addition, the auto-ignition temperature of ammonia is high, about 650^0C; compared with hydrocarbons (Propane- 480^0C, Cyclohexane- 270^0C) therefore ammonia is difficult to ignite. Ammonia has never exploded in the open air, and it is doubtful if a concentration as high as 16% could be attained out of doors [16].

In Oklahoma, in 1978, the refrigeration system on an ammonia storage vessel failed which caused the warm up of ammonia resulted in pressure rise subsequently opening of relief valve. The discharged ammonia was ignited by a nearby flare. In another incident a welder was killed by an explosion in New Zealand in 1991 while working on an empty 28-m^3 tank, which contained a flammable mixture of ammonia vapor and air. In 1989, at Jovona, Lithuania, a storage tank split from top to bottom, and 7,000 tons of liquid ammonia were spilled. The pool caught fire, but according to later reports, the fire was due to rupture of a natural gas line that passed through the area [16].

Attenuation is an important method of making plants inherently safer. Attenuation is achieved by using hazardous materials in the least hazardous form. For example, while small quantities of liquefied toxic or flammable gases such as chlorine, ammonia, and propane are generally stored under pressure at atmospheric temperature while large quantities are stored at low temperature at or near atmospheric pressure. Because of low pressure, the leak rate through a hole of a given size is smaller, and because the temperature is low, evaporation will also be much less. The possibility of a leak from the refrigeration equipment has to be considered as well as the possibility of a leak from the storage vessel, and for this reason only large quantities are refrigerated [4].

V.   Conclusion

Ammonia is one among the largest volume inorganic chemicals in the chemical process industries and used mainly for production of fertilizers using steam

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reforming of natural gas. Large release of ammonia due to accidents and vessel failures etc. has decreased considerably in the last three decades. But problems and failures do occurs frequently in the ammonia plant even after following the inherently safer design philosophy and risk assessment. Major areas of concerns/ failures are reforming and synthesis loop causing fires and shutdowns. Above study reveals that all the stages/aspects of a plant like design, risk assessment, material of construction, fabrication process, inspection (including material supplied by vendors), maintaining operating parameters during normal operation, adherence to the guidelines during operation as well as maintenance, periodic inspection of equipments and machineries can prevent failures and shutdowns in the plant resulting in improved performance. Further, periodic training, motivation and feedback from operating personnel are most important and should be followed in right spirit to improve the performance of plant.

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