

Classic paper presentation



Nobel lecture

The development of chemical high pressure method during the establishment of the new ammonia industry

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by

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The eminent distinction of the Nobel Prize award which imposes on me the pleasant duty of delivering a lecture to you to-day is in recognition of the development of the high pressure techniques, i.e. the methods used to perform chemical reactions at elevated temperature under pressures of 100 atmospheres and higher.

Partly as a result of the Lecture given by Haber here in Stockholm in 1920 it has been learned that in 1908 he approached the Badische Anilin- und Soda-fabrik with the suggestion to attempt the technical synthesis of ammonia from hydrogen and nitrogen under high pressure. In the course of his studies on the position of the equilibrium in the system ammonia \rightleftharpoons hydrogen + nitrogen he ascertained that the foreseeable shifts in equilibrium for high pressures actually occur in favour of the ammonia, but - and this was a particularly encouraging and for us decisive fact - he also found that osmium and uranium markedly accelerated the attainment of the equilibrium and brought it about at a far lower temperature than the catalysts exclusively used hitherto, i.e. manganese, iron, platinum, etc.

When the management at the time entrusted me with the task of expanding the conversion of this high pressure synthesis to an industrial scale with a view to its economic application, it was obvious that there were three main problems which had necessarily to be settled before the construction of a plant could be undertaken. These were: supply of raw materials, i.e. of the gases hydrogen and nitrogen, at a lower price than hitherto possible, the manufacture of effective and stable catalysts, and lastly the construction of the apparatus. These problems were dealt with simultaneously.

To begin with, sufficient pure hydrogen for the tests was available from chlorine electrolysis and yielded the required nitrogen-hydrogen mixture by combustion with air. None of the then known hydrogen processes was suitable for large-scale manufacture. We examined all of them but without exception they were too expensive or yielded insufficiently pure gases. It must not be forgotten that the procurement of hydrogen is the largest item in the prime cost, at least nowadays since because of the efficiency of the high pressure synthesis the conversion of the ready-made gas mixture into ammonia is only a minor cost factor. Since we were dependent on coal as a starting material we at once decided on the sole eligible source in our situation, water gas from which nowadays, after a short transition period - separation of the hydrogen by Linde's low temperature liquefaction method - hydrogen is manufactured in enormous quantities by a catalytic process developed by us.¹

The accomplishment of the second task was no less significant. Osmium, a very good catalyst, was difficult to maintain since when in its active, i.e. finely disperse, form it comes into contact with air (never wholly avoidable under industrial conditions) it readily volatilizes as osmium tetroxide, but in particular because the whole world's supply amounted to only a few kilograms, hence at best we could have based only a very moderate manufacture on osmium. Uranium was expensive, of course, yet nevertheless obtainable in some quantity but proved extremely sensitive to oxygen and water and it could not, and still cannot, be converted by any means into a form that can be used for a mass-production process. However, as a result of very broadly

nique, we succeeded in preparing relatively fast-acting, technically perfect, easily manipulable, stable and inexpensive catalysts, chiefly those with iron as the active substance* which are currently in universal use presumably exclusively for the synthesis of ammonia. They are a completely new type of

The successful conclusion of the third task, which is the only one I intend to discuss to-day in more detail, related to the apparatus. There were no examples in industry. The only technique, and that of a physical nature, involving high pressures, was Linde's liquefaction of air. The conventional apparatusments of long duration. We took as our starting point the apparatus used by Haber for his tests and of which I append an illustration (Fig.1). The contact mass, surrounded by tightly packed insulation, is in the centre of the steel vessel which is cooled with ice water. The gas lead, heating arrangement and temperature measuring device are plainly shown in the figure. As this apparatus suffered from many drawbacks for prolonged experiments, it was completely redesigned and finally in this form (Fig. 2) 24 of them were in continuous day and night service for years.

The state of affairs was soon to change when we started to build a smaller convertor as a production apparatus. Haber had already attempted something along those lines on a very small scale but had only run it for one or two hours when it failed for design reasons.

We therefore then built an apparatus of which the chief, and for us the most important and interesting part, except for a circulation pump and the ammonia separator, was a contact tube of the shape and size illustrated in Fig. 3 (wall thickness about 30 mm). This tube was heated on the outside.

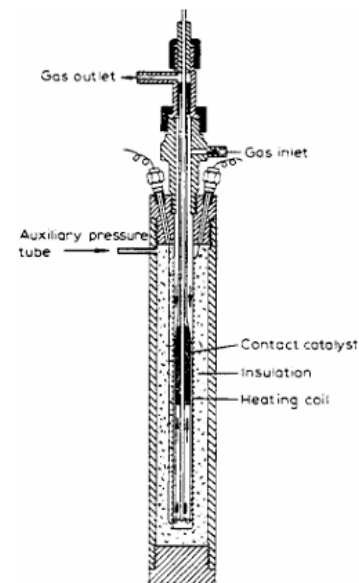


Fig. 1. Haber's experimental convertor, 1909.

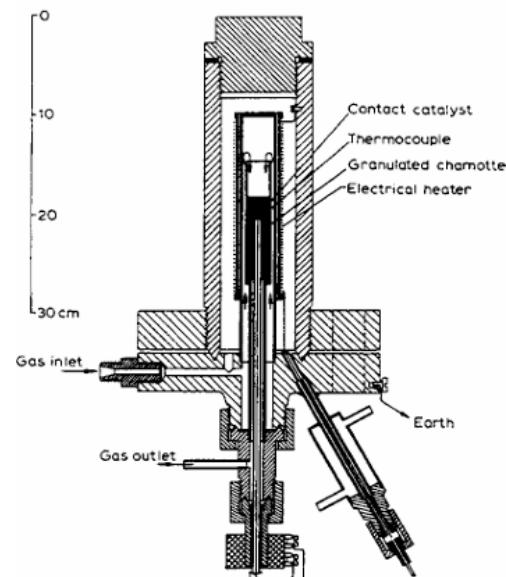


Fig. 2. Laboratory convertor, 1910₄

The two tubes drawn by Mannesmann had each done service for 80 hours, then they burst. If we had charged them with osmium instead of with our phenomena appeared, albeit after a longer period. Tests of materials in the laboratory revealed that at high pressure and quite high temperature, carbon steel is invariably corroded by hydrogen in the manner outlined. It is just achieved. Only refined steels, as we found in subsequent studies, gave better results so that they were a suitable building material and are still in use to-day.³

Meanwhile we had found a better and quite general solution to the problem of making apparatus with unlimited stability to hydrogen at high temperature under all conditions.

The long-sought solution consisted in fitting a pressure-bearing steel jacket internally with a quite thin lining of soft steel in such a way that as the hydrogen, which of course only diffuses, passes through the thin lining it is able to escape without building up pressure before it can attack the outer steel jacket at the high temperature. This is readily achieved by the grooves. The ultimate version of a convertor appeared as in Fig. 30.

The figure illustrates the design of a large convertor of 800 mm inside diameter - an "800 oven" - with gas heating on the inside, a central tube and nitrogen flushing. The inside system of gas heating was retained until 1922. During the more than 10 years that this method was used under production conditions, no explosions occurred in the convertors. Observations of the thermocouples, particularly the one fitted to the burner, showed when it was necessary to apply additional heating to the oven, even below a pressure of 200 atm., by forcing in air.

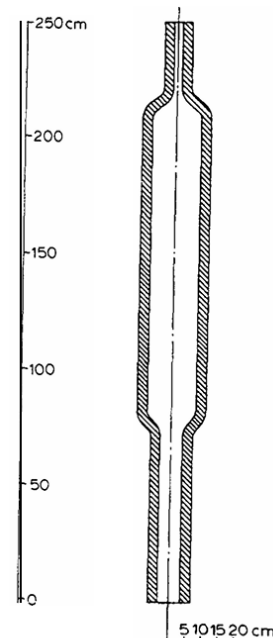


Fig. 3. Contact tube as used in first technical trial, 1910.

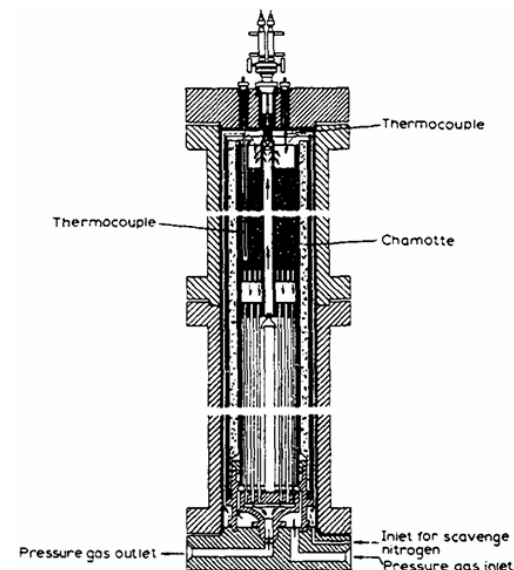


Fig. 30. Convertor with nitrogen flushing, 1916.

apparatus could not stand stoppages. Probably all the designs then built were tried out by us and in years of work, on the basis of the experience which we gained from those small compressors which ran for not more than half a day without stopping, we succeeded in building the gigantic 3,000 horse power units which function reliably and without stoppage for 6 months, after which they receive their regular, periodic clean. At this juncture I should like to stress how important completely smooth running is for the synthesis of ammonia. Every stoppage at a single point affects the entire plant and after a stoppage it takes hours until everything is back to normal. It is no exaggeration for me to say that, technically speaking, it depends entirely on smooth, continuous operation whether the process is economical. It has taken us years to reach that point. Of particular assistance here were the *monitoring instru-*

I am now coming to the end of my account. The development of the ammonia manufacturing process which partly as a result of Germany's isolation owing to the war, was perfected in a relatively short space of time by harnessing all our efforts and resources, has also of course stimulated the development of other reactions which proceed markedly better under high pressure than under atmospheric pressure. Experience has shown that in general it is

I have to-day been able to inform you about only a small section of the work which has been carried out in our firm since 1908 in the field under discussion. It scarcely need be added that this achievement has only been made possible by a large staff of colleagues. It is probably true to assert that such numbers have never before been engaged on one single problem. I sincerely wish to mention in particular two colleagues present here to-day who threw themselves heart and soul into our work from the very outset, namely Dr. Mitasch, head of the scientific laboratory, and Dr. Lappe who was in charge of technical development. To them I would like to acknowledge now my gratitude for their unstinting, loyal co-operation.

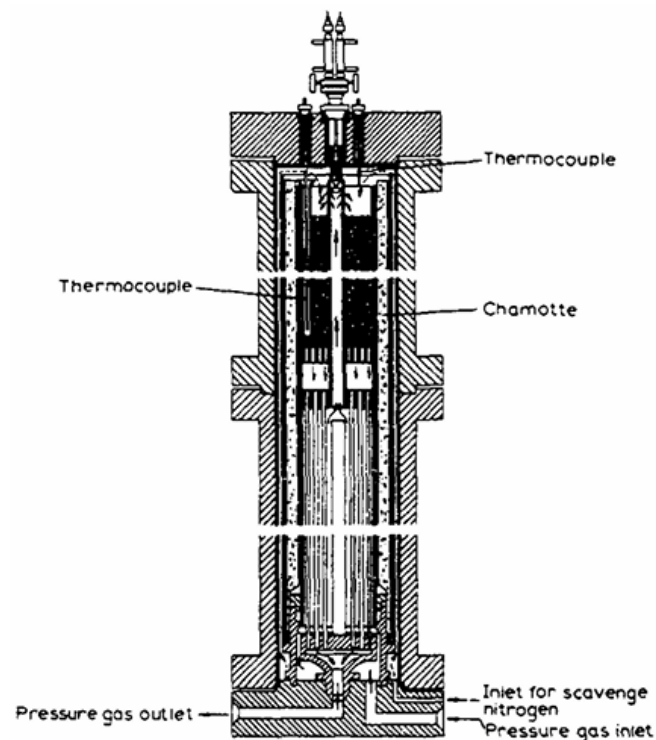
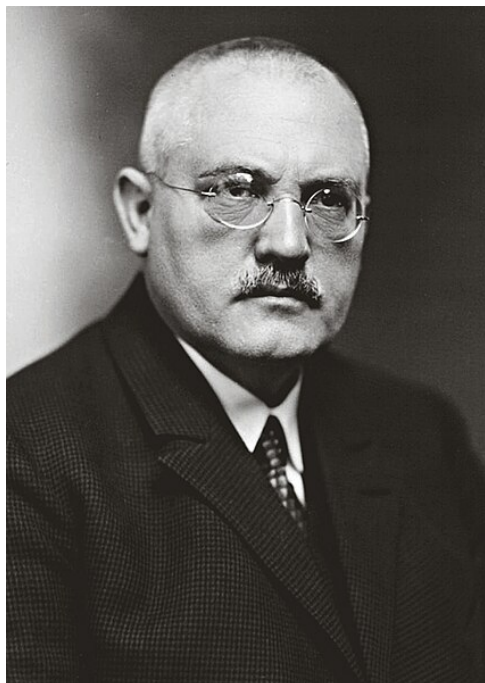


Fig. 30. Converter with nitrogen flushing, 1916.

Biography



Carl Bosch

Born	27 August 1874 Cologne, German Empire
Died	26 April 1940 (aged 65) Heidelberg, Germany
Education	Technical University of Berlin Leipzig University
Known for	Bosch reaction Bosch–Meiser urea process Haber–Bosch process
Awards	Liebig Medal (1919) Werner von Siemens Ring (1924) Nobel Prize in Chemistry (1931) Wilhelm Exner Medal (1932) Goethe Prize (1939)
	Scientific career
Fields	Chemistry
Institutions	BASF , IG Farben
Doctoral advisor	Johannes Wislicenus ^[1]

Signature



Thank you