

The Recovery of Platinum from Ammonia Oxidation Catalysts

THE CURRENT STATE OF THE CATCHMENT GAUZE PROCESS

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Since the general introduction in the late 1960s of gold-palladium catchment gauzes to recover platinum lost from the catalysts of ammonia oxidation plants (1, 2), and their incorporation into an efficient, reliable process in the mid 1970s (3), their application has become widespread. There are now very few nitric acid manufacturing plants in Europe that do not use them on a regular basis and recoveries in the order of 50 to 60 per cent of the gross losses are being achieved regularly. In the United States of America, a market that has traditionally used filters to recover platinum, their use is also spreading rapidly, the merits of the system having been recognised clearly from both a technical and an economic point of view. It is estimated that on a world wide basis there are in the order of one hundred nitric acid plants regularly using catchment gauzes, and few problems are encountered with their use.

When the most recent article on this topic

appeared in this Journal in 1973 catchment gauze packs were generally less than 2.5 metres in diameter, and the maximum size then in use was 3.4 metres. However, since that time a general change has occurred in the design of nitric acid plants; outputs per burner have increased considerably and as a consequence reactor diameters and hence effective catalyst diameters have, generally, also increased. The largest are now 4.8 metres (4) and even larger reactors may be produced in the future.

The merit of the catchment gauze installation method described previously (5) has been vindicated on many occasions, particularly in large reactors and especially where unscheduled shutdowns are a common occurrence. The ability of the pack to expand and contract freely, illustrated in Figures 1 and 2, without damaging either the catalyst or the gold-palladium catchment gauzes has been, in a large number of cases, the key factor enabling the

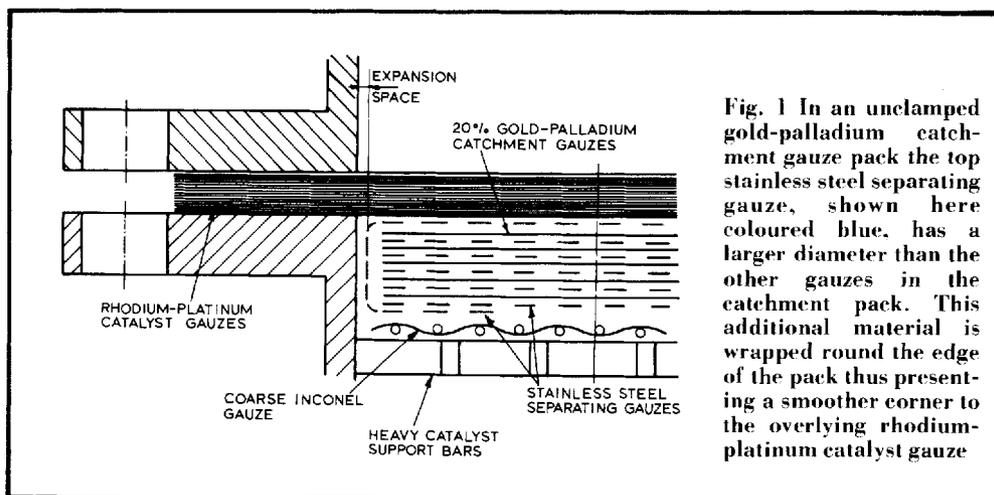
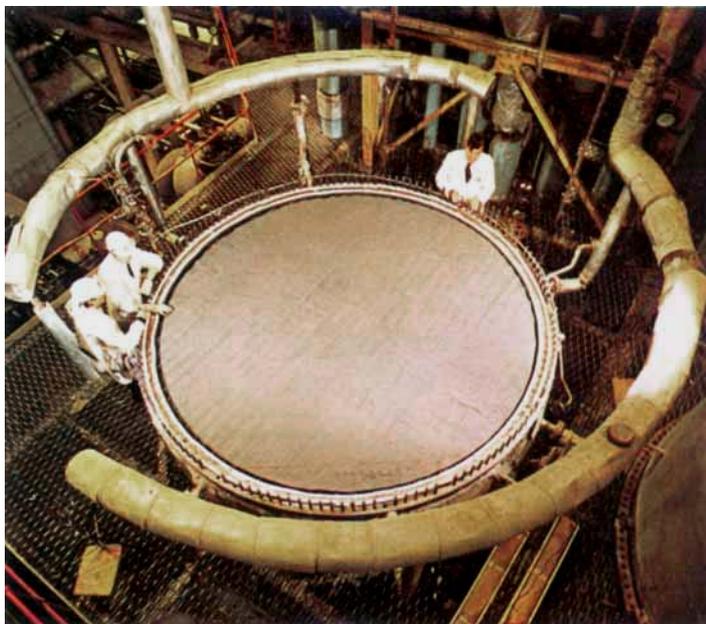


Fig. 1 In an unclamped gold-palladium catchment gauze pack the top stainless steel separating gauze, shown here coloured blue, has a larger diameter than the other gauzes in the catchment pack. This additional material is wrapped round the edge of the pack thus presenting a smoother corner to the overlying rhodium-platinum catalyst gauze

Fig. 2 This unclamped catchment gauze pack has served in a large capacity medium-pressure (4 atm) ammonia burner for one acid making campaign. Although the pack has shrunk slightly it is still perfectly operational, and its ability to recover platinum will not be impaired during the next campaign for the pack expands again on reheating

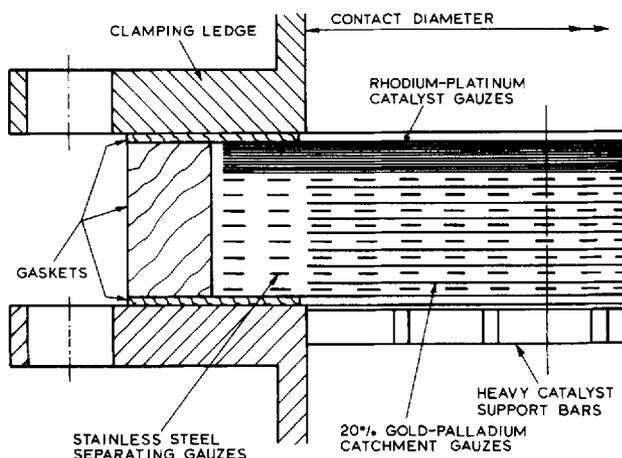


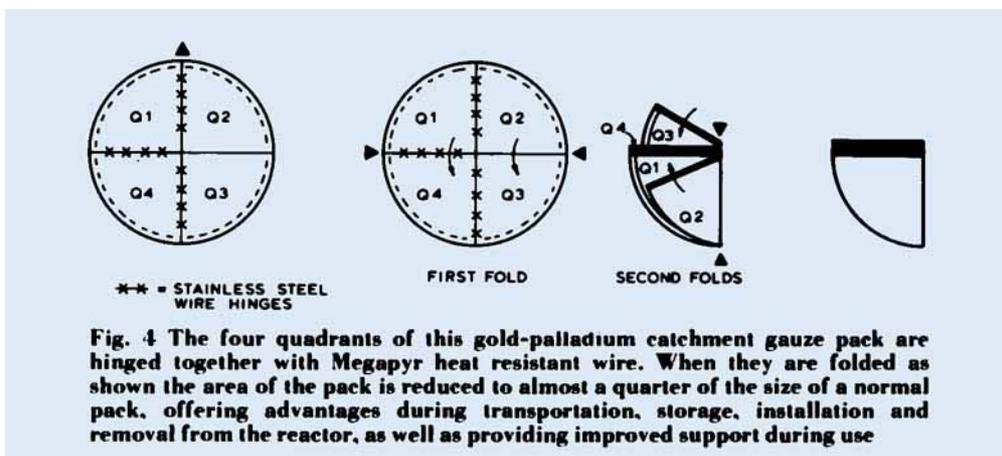
process to be used without operating problems. In addition noble metal losses experienced on removal of the gauzes have been minimised.

In contrast to this arrangement, separate base metal support gauzes, illustrated in Figure 3, inevitably tear the now fragile gold-palladium gauzes on removal, and if this is necessary for an exceptional reason, such as boiler problems, the damage caused when the gauzes are separated can be considerable.

The gauzes may even become unfit for further use, to the detriment of operating economics. Furthermore handling and storing a partly used pack of gauzes is a difficult, if not impossible, task. Therefore on anything other than a completely reliable plant it is advantageous to use a composite or 'biscuit' pack. This consists of the normal arrangement of gold-palladium catchment gauzes and stainless steel separating gauzes stacked alternately, but with one larger

Fig. 3 Here the catchment gauzes have a diameter equal to the contact diameter of the catalyst gauzes. This clamping method has been used to secure gold-palladium catchment gauze packs during commercial plant trials but it can cause severe distortion problems, and on removal from the reactor the Megapay support gauzes tend to separate from the others in the pack, damaging the gold-palladium gauzes





than usual stainless steel gauze, coloured blue in Figure 1, wrapped round all the others making them into an integral unit.

For small plants a 'biscuit' pack can be delivered already made up by the manufacturer. Thus the advantages of such packs are fourfold;



Fig. 5 A folded catchment gauze pack can be delivered in a case not much larger than one quarter of the area of a normal pack, thus facilitating easier storage and transportation, particularly when the latter is undertaken by air

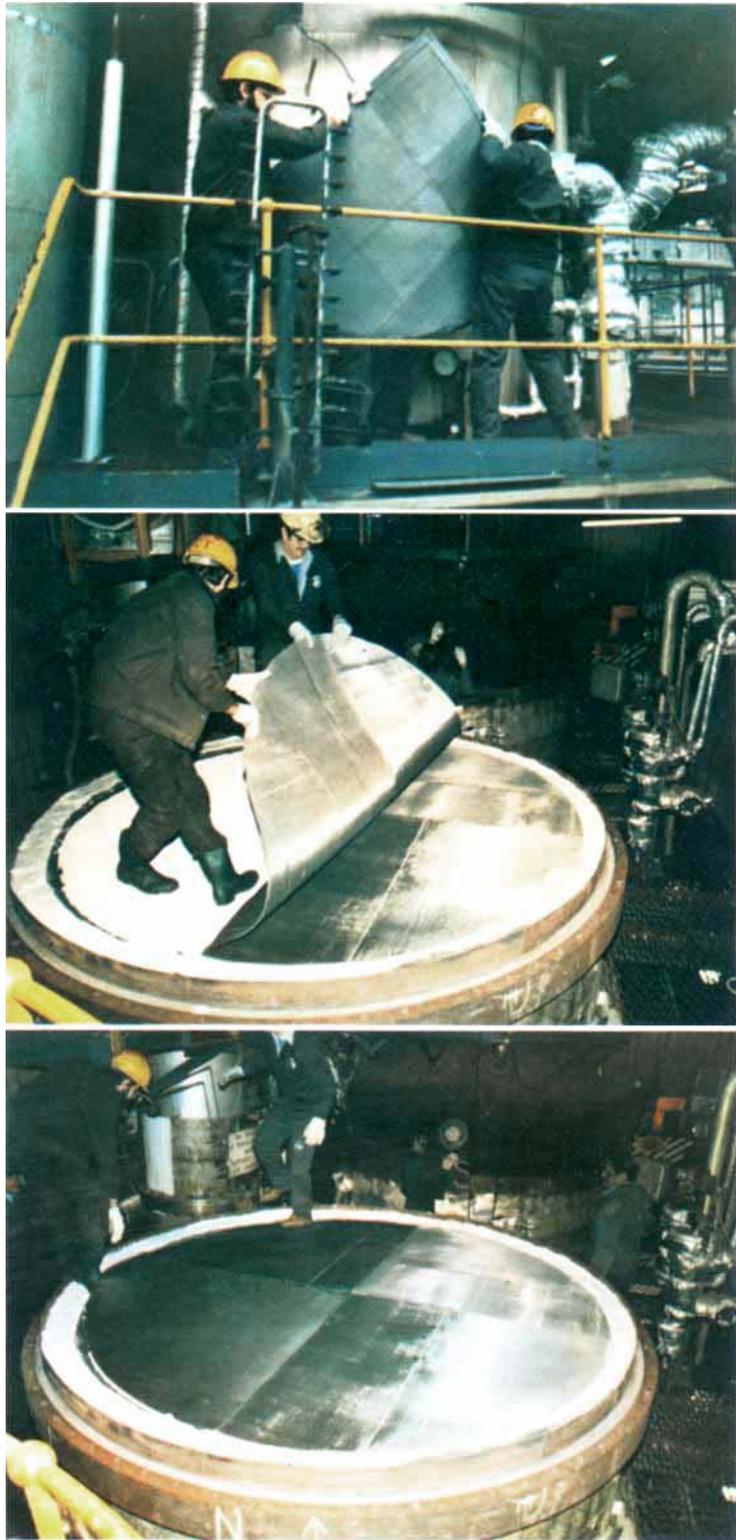


Fig. 6 Handling a gold-palladium alloy gauze catchment pack in the restricted space around an ammonia oxidation reactor is much easier when a folding pack is used, and installation in the reactor is both rapid and simple

fast installation, tolerance of thermal expansion, stability on removal, and they facilitate secure storage during long shutdown periods.

Folding Catchment Gauze Packs

Larger packs inevitably take a significant time to unpack and install, and secure storage can be a problem due to their size. In the search for even higher noble metal recoveries, catchment packs are tending to contain greater amounts of gold-palladium than in the past, necessitating secure storage during shutdowns. Such considerations, coupled with the increasing cost of downtime, led to the concept of using what are effectively a number of small 'biscuit' packs in large reactors (6). A variety of solutions was possible but the preferred method at present is to divide the pack into four separate quadrants hinged together by heat resistant steel wire. By folding the quadrants in a given sequence, shown in Figure 4, it is possible to deliver a large ready made 'biscuit' pack in a case not much larger than a quarter of the area of a normal pack, see Figure 5. Storing and carrying such a pack in a restricted space is now possible, while installation, which is shown in Figure 6, and removal from the reactor is both simple and rapid, and operator reaction to the system has been very good. In addition, the quadrant form is ideal for air cargo transportation since the majority of sizes fit standard cargo hold dimensions.

The folding catchment gauze pack concept was originally conceived to enable rapid installation and removal, and safe storage, without damage to the gold-palladium gauzes. However, operational experience has shown other advantages. The ability of the independent segments to accommodate the slight catalyst bed distortions and minor movements between support gauzes, which generally occur in large reactors as the campaign proceeds, has resulted in far better support for the gold-palladium gauzes. As a result noble metal losses due to attrition appear in a number of cases to have been reduced.

The better support offered by this system will be of even greater value if catchment gauzes of lower gold content are developed. When the technology was first introduced 20 per cent gold-palladium was the optimum material, when considering both strength and recovery efficiency, but the improved support systems now available may permit a reduction to be made in the gold content, to the benefit of the economics of the process.

References

- 1 H. Holzmann, *Chem.-Ing.-Tech.*, 1968, **40**, (24), 1229
- 2 H. Holzmann, *Platinum Metals Rev.*, 1969, **13**, (1), 2
- 3 A. E. Heywood, *Platinum Metals Rev.*, 1973, **17**, (4), 118
- 4 *Nitrogen*, January/February 1980, (123), 34
- 5 *Op. cit.*, (Ref. 3), p. 124
- 6 *British Patent Appl.* 2,050,189; 1981

Tungsten-Osmium Alloys for Improved Cathodes

Progress in cathode research was reported at the second Tri-Service Cathode Workshop held last year at the Rome Air Development Center, New York. Selected papers were later published, and in one of these the authors M. C. Green, H. B. Skinner and R. A. Tuck of EMI-Varian Limited at Hayes, Middlesex, presented studies of tungsten-osmium alloys and their relevance to improved M-type cathodes (*Appl. Surf. Sci.*, 1981, **8**, (1 and 2), 13-35).

When half the emitting area of a B-type cathode was sputtered with osmium the coated area performed as M-type, and the expected difference in the work function of the two parts was observed. However an interfacial band gave

enhanced emission, and was of obvious practical interest. Mixed matrix cathodes were prepared from 80 tungsten-20 osmium sintered compacts impregnated with barium calcium aluminate. Initially these gave disappointing results but after 500 hours the performance became most promising, the zero field emission being some 50 per cent better than M-type.

A definite correlation between the formation of the tungsten-osmium σ intermetallic phase and the improved emission has been demonstrated. As a result an improved dispenser cathode has been constructed.

Experimental work is continuing to determine the mechanism of emission enhancement.