RAL-93-016

Science and Engineering Research Council
Rutherford Appleton Laboratory
Chilton DIDCOT Oxon OX11 0QX

RAL-93-016

Low Outgassing Rates from Stainless Steel with only an Alkali Detergent Wash

J R J Bennett

March 1993

Low Outgassing Rates from Stainless Steel with only an Alkali Detergent Wash

J R J Bennett, *Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX, UK*

Notes of a presentation given at a meeting:-

IS GREEN CLEAN?

organised by
The Vacuum and Thin Films Surfaces Group of the Institute of Physics

The Institution of Mechanical Engineers
in collaboration with
The Institution of Electrical Engineering
and The Royal Society of Chemistry

on
24 February 1993
at
The Society of Chemical Industry
14/15 Belgrave Square
London SW1 8PS

Low outgassing rates from stainless steel with only an alkali detergent wash

J R J Bennett, *Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX, UK*

Results of measurements of the specific outgassing rates of 304L and 316L stainless steel following only an alkali detergent wash and low temperature bake (≤ 200°C) are presented. Values as low as 3×10^{-15} mbar l s^{-1} cm^{-2} have been recorded.

1. Introduction

Many vacuum components are prepared by long and complicated cleaning procedures. These often entail:- powder or bead blasting; solvent or vapour degreasing, possibly using chlorinated hydrocarbons; ultrasonic cleaning; acid and alkali etching; heat treating at high temperatures (up to ~800°C). Some of these steps are repeated during the cleaning cycle. In addition, the component may then undergo plasma discharge cleaning and bake-out under vacuum.

It was observed that the vacuums obtained by simply washing the stainless steel components in an alkali detergent were as good as those obtained by the more elaborate techniques. This led to the routine cleaning of all stainless steel components in a 5% solution of *Decon 90** followed by thorough rinsing in de-mineralised water. The components were vacuum baked at 150°C for up to 10 days when pressures below 10^{-8} mbar were required. The lowest specific outgassing rates were obtained by an additional bake [1][2] at 200°C in air for 24 hours.

2. Results

Measurements of the specific outgassing rate of 304L stainless steel sheets were made by the throughput method [3]. The size of the sample chamber restricted samples to ~2000 cm^2 surface area and limited the sensitivity of the measurement to ~10^{-12} mbar l s^{-1} cm^{-2}. A larger chamber was used to accommodate many sheets of 0.5 mm thick 316L stainless steel, making up a total area of 1.25×10^5 cm^2. The final series of measurements were of a large chamber, Figure 1, 1.4 m diameter and 5 m long, made from 316L stainless steel sheet. The surface area was 5×10^5 cm^2. Measurements on both these larger surface areas were made by the throughput and isolation pressure rise methods (IPR). The results are summarised in Table 1.

Samples #1 and #2 consisted of 0.5 mm thick bright annealed sheets. The plates were cleaned by immersion in a 5% *Decon 90* solution for ~18 hours, rinsed under running water for ~5 minutes and then rinsed in de-mineralised water. The samples were then baked in air at 200°C for 24 hours. After pumping down, the vacuum equipment, including the samples were baked for 10 days at 150°C. Measurements of the outgassing rates were taken at intervals following cooling to room temperature. The results shown in Table 1 are the "equilibrium", long term, ~10 day, measurements.

Sample #3 was the prototype vacuum vessel [4][5] for an interferometric gravitational wave detector [6]. It consisted of 0.9 mm thick bright annealed sheet welded to form tubes, ~1.2m long, which were convoluted by roll-forming to give the necessary stability to withstand the vacuum load. The tubes were welded (TIG) together to form a 5m long section and the ends closed with 3 mm thick dished plates. The end plates were purchased from a vacuum company and cleaned to their own "uhv specification" using an alkali detergent. The end plates contributed ~ 5% of the total surface area. Each 1.2 m long convoluted section of tube was washed by rolling in a shallow bath of 5% Decon 90 solution for a few minutes, followed by rinsing in 2 shallow baths of de-mineralised water. The surfaces were manually wiped during the processes using industrial paper wipes. Finally the tubes were sprayed by a jet of hot de-mineralised water from a commercial pressure cleaner. The end dishes were similarly treated. Once the tube was welded together, including the end dishes, there was no way to clean the assembly: the vacuum ports in the centres of the end dishes were only 60 mm bore diameter.

---

* Decon 90 is a product of Decon Laboratories Limited, Conway Street, Hove, East Sussex BN3 3LY, UK. It is a phosphate-free, biodegradable, alkali, surface active, cleaning agent in an aqueous base.

Table 1

#	Sample Material	Surface Area cm^2	Air Bake at 200°C for 1 day	Gas Species	Specific Outgassing Rate mbar l s^{-1} cm^{-2}
1a	304L	2000	no	total	$\leq 1x10^{-12}$
1b			yes	total	$\leq 1x10^{-12}$
2	316L	$1.25x10^5$	yes	total	$3x10^{-15}$
3a	316L	$5x10^5$	no	H_2	$2x10^{-13}$
				CH_4	$\leq 2x10^{-18}$
				H_2O	$\leq 3x10^{-17}$
				CO	$\leq 4x10^{-16}$
				CO_2	$\leq 2x10^{-17}$
3b			yes	H_2O	$\leq 8x10^{-14}$
				CH_4	$\leq 1x10^{-18}$

4. Discussion

The results shown here are as good and in many cases better than those reported previously, for example [1][2][7][8][9][10][11], using more elaborate cleaning processes and generally at much higher baking temperatures. High temperature vacuum bakes, ~800°C, will always produce low outgassing rates. However, high temperature bakes are not always convenient, particularly with large vacuum vessels or where certain components can not withstand the high temperatures. The results illustrate the effectiveness of the simple cleaning technique, which is environmentally "friendly" and does not use any chlorinated or fluorinated hydrocarbons, and employs modest baking temperatures.

The particularly good results of sample #2 were not achieved on the prototype tube, sample #3 . This may have been due to the non-uniform temperatures over the vessel, particularly during the air bake. Small areas of the chamber (near the supports) were at only 140°C, whilst others were at 280°C.

There is some indication that 316L performed better than 304L steel, but this may only be a result of the lower limits of measurement with the larger samples. The reasons for using 316L steel in the latter tests were mainly due to preferences for greater corrosion resistance and better welding characteristics for use on the prototype gravity wave detector.

The air bake is effective in reducing the outgassing rate, probably by forming an oxide layer which inhibits the passage of hydrogen from the underlying material [12][13][14]. Also the air bake alone has been found to be effective in removing light contamination from surfaces by oxidation. Further tests are to be made to assess the effectiveness of only an air bake.

It is interesting to note that the last operation in the manufacture of the bright annealed stainless steel sheet is a controlled atmosphere bake at over 1000°C. If the steel is kept clean and not manually handled then the sheet will be very clean by virtue of this last processing. In this case cleaning with the alkali detergent may be unnecessary or even detrimental.

The results of sample #3 are particularly revealing in showing that over 99.77% of the residual gas is hydrogen. Difficulties were experienced in measuring the true partial pressure fractions due to manufacture of the gases in the gauges [15][16][17]. It is believed that the hydrogen could account for an even larger fraction of the total gas. However, the results show very low levels of hydrocarbon contamination (only methane was detected by the quadrupole mass spectrometer). The results also indicate that even though the tube was fabricated in an industrial environment, not as clean as one would normally expect for uhv work, nevertheless the outgassing rate was low and there was no sign of contamination.

Acknowledgements

The author is pleased to acknowledges the invaluable work of R J Elsey and practical help of T P Parry in the successful execution of this work.

References

1. R L Samuel, Vacuum, **20**, 295 (1970).
2. G Moraw and R Dobrozemsky, Proceedings of 6th International Vacuum Congress, 1975; Jpn J Appl Phys, Suppl 2, Part 1, 261 (1974).
3. R M Zabel, Rev Sci Instrum, **4**, 233 (1933).
4. J R J Bennett, R J Elsey and R W Malton, Vacuum, **43**, 531 (1992).
5. J R J Bennett and R J Elsey, Vacuum, **43**, 35 (1992).
6. Proposal for a Joint German-British Interferometric Gravitational Wave Detector, J Hough, B J Meers, G P Newton, N A Robertson, H Ward, G Leuchs, T M Niebauer, A Rudiger, R Schilling, L Schnupp, H Walther, W Winkler, B F Schutz, J Ehlers, M W Hamilton, I Schutz, H Welling, J R J Bennett, I F Corbett, B W H Edwards, R J S Greenhalgh and V Kose, Max-Plack-Institut fur Quantenoptik Report No MPQ 147 and Rutherford Appleton Report No GWD/137/JH(89), September 1989.
7. R Calder and G Lewin, Brit J Appl Phys, **18**, 1459 (1967).
8. V S Zhilnin L P Zilnina and A A Kuzmin, Proceedings of 4th International Vacuum Congress, 1968.
9. J R Young, J Vac Sci Technol, **6(3)**, 398, (1969).
10. R Nuvolone, J Vac Sci Technol, **14**, 1210 (1977).
11. H C Hseuh and X Cui, J Vac Sci Technol, **A7(3)**, 2418 (1989).
12. Y Ishikawa and K Odaka, Vacuum, **41**, 1995 (1990).
13. C L Huffine and J M Williams, Corrosion, **16**, 430 (1960).
14. R A Strehlow and H C Savage, Nuclear Technol, **22**, 127 (1974).
15. D Alpert, Le Vide, **97**, 19 (1962).
16. H F Dyalla and W R Blanchard, J Vac Sci Technol, **A1**, 1297 (1983).
17. J R J Bennett and R J Elsey, Proceedings of the 8th International Vacuum Congress, 1992; to be published in Vacuum.

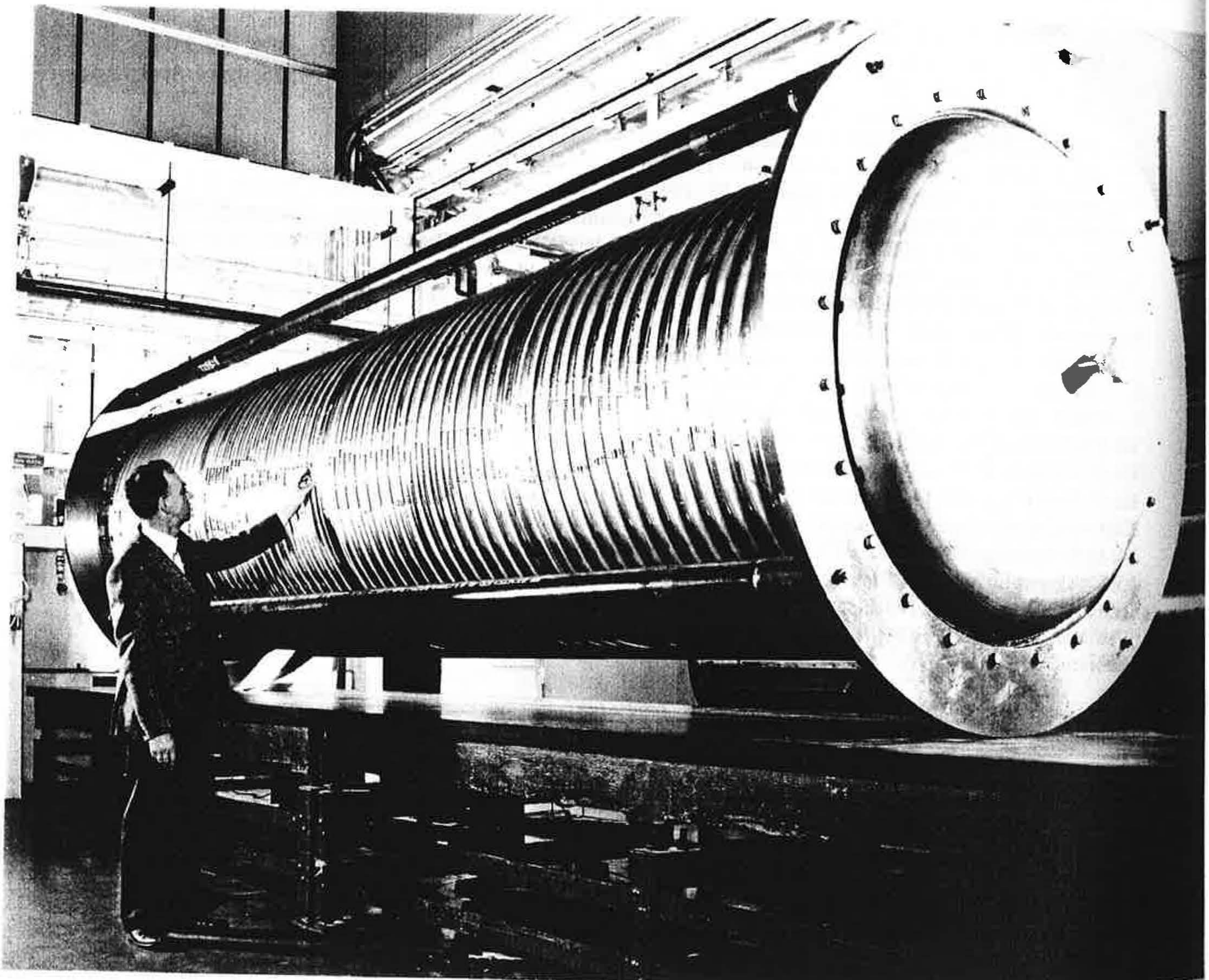

Figure 1. Prototype Convoluted Vacuum Chamber