



Science & Technology
Facilities Council

Low Temperature Air Bake of Stainless Steel for Very Low Outgassing Rates

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Surface Conditioning for Ultra-High Vacuum, Brokering Meeting.

Daresbury Laboratory, Warrington. 25 February 2009

I will describe the process used for cleaning and processing stainless steel. As examples I will use our experience in the R&D and building of:

1. ISIS
2. A prototype gravity wave detector

This work was done a long time ago -- 1977-1990.

ISIS

Requirements are for High Vacuum, but built to uhv standards
- no hydrocarbons.

Pressure $\leq 5 \times 10^{-7}$ mbar,

Total Outgassing Rate $\leq 10^{-9}$ mbar l/s/cm² (10 hour),

Hydrocarbons $\leq 10^{-10}$ mbar l/s/cm² (10 hour). (No rubbers or plastics because of ion pumps.)

Material: Austenitic stainless steel e.g. 304, 316L.

Manufacturers of UHV components were, and I assume, are still well aware of the requirements for high quality forged blanks for flanges. After machining using a water based lubricant, the items were cleaned in an alkaline detergent (5% solution of Decon 90) and thoroughly washed with clean water (demineralised water not required). Specified no use of other types of cleaning agent e.g. chlorinated or fluorinated hydrocarbons.

For vessels fabricated from sheet SS we requested bright annealed 316L sheet. The sheet is usually very clean as prepared by the manufacturer - it comes out of a furnace! If the sheet is kept clean there is little need for additional cleaning. Keep clean during the manufacturing process. Cutting and welding should not add any dirt. The final fabrication is washed in alkaline detergent as above.

Final vacuum test and mass spectrometer test to ensure item meets requirements.

I am sure all manufacturers are well aware these processes.

UHV - The Gravity Wave Detector

Requirements:

Pressure $\leq 10^{-8}$ mbar for H_2 ; 10^{-9} mbar all other gases; hydrocarbons as low as possible to reduce possible contamination of the mirrors.

However, to reduce costs of the very large vacuum system (two tubes each 3 km long, 1.4 m diameter), the outgassing rates should be as small as possible.

Total Outgassing $\leq 10^{-12}$ mbar l/s/cm².

Hydrocarbons - as low as possible.

Manufacture - as before.

On-site preparations to reduce hydrogen outgassing:

Bake at 200° C for 1 day in air.

Bake at 150° C for 10 days in vacuum (after reaching 10^{-6} mbar)

Results:

Hydrogen $\leq 4.5 \times 10^{-15}$ mbar l/s/cm².

CH₄ (the only hydrocarbon detected) $\leq 5 \times 10^{-22}$ mbar l/s/cm²

CO $\leq 6 \times 10^{-17}$ mbar l/s/cm²

CO₂ $\leq 3 \times 10^{-18}$ mbar l/s/cm²

H₂O $\leq 3 \times 10^{-17}$ mbar l/s/cm².

The air bake reduces the outgassing rate from $\sim 2 \times 10^{-13}$ mbar l/s/cm² (no air bake).

Provided the item is not contaminated, the air bake need not be repeated again. Tests after 10 years showed that outgassing rates were as good as ever.

Summary of the Cleaning Process for Stainless Steel (e.g. 316 L)

1. Keep the items clean during manufacture.
2. Wash in alkaline detergent, followed by rinsing well in clean water.
3. Bake in air for 1 day at 200°C.
4. Pump down to $\sim 10^{-6}$ mbar and then bake under vacuum for 10 days at 150°C.

Resulting in:

A SUPERB VACUUM!

Comments

1. In general if a vessel has hydrocarbon contamination an air bake, as above, will remove the problem.
2. Ion gauges and ion pumps etc. manufacture hydrocarbons (CH_4), CO and CO_2 . So do not use if you want to keep the pressure as low as possible. But, how do you measure the pressure?
3. Cleaning SS with an alkaline detergent is widely used. A dishwasher can be used for small components. Large components can be sprayed with a commercial high-pressure jet washer that gives very hot water with detergent.

4. The method of using a relatively low temperature air bake followed by a low temperature vacuum bake is an adaptation of the scheme described by: G. Moraw, R. Dobrozemsky, Proceedings of the 6th International Vacuum Congress, 1974; Jpn. J. Appl. Phys. Suppl 2, Part 1 (1974) 261-264.

5. Hydrogen is the main component of the outgassing of stainless steel after baking in vacuum to remove the water. Our measurements of the outgassing rate of hydrogen before the vacuum bake gave 2×10^{-13} mbar l/s/cm².

Finally, in summary, I conclude that:

The specific outgassing of the hydrogen from 316L stainless steel with the low temperature air bake is found to be

$$\leq 4.5 \times 10^{-15} \text{ mbar l s}^{-1} \text{ cm}^{-2}$$

and the total of all other gasses amounts to no more than 10% of this figure. The specific outgassing rate of methane is found to be $\leq 5 \times 10^{-22}$ mbar l s⁻¹ cm⁻².

The water is still problematical and requires further study, but indications are that it is produced by interaction of the hydrogen with the oxygen on the surface of the stainless steel. Hence it is very likely that only hydrogen is outgassed from clean stainless steel, as conjectured recently by Fremery (J.K. Fremery, Vacuum 53: (1999) 197-201).

The process of a low temperature air-bake followed by a vacuum-bake provides hydrocarbon free, very low outgassing rates for SS.