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Letter to the Editor

Dependence of the outgassing rate of a "vacuum fired" 316LN stainless steel chamber on bake-out temperature

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It was found that the outgassing rate of a vacuum fired 316 LN stainless steel chamber increased *with bake-out temperature in the range of 100°C to 180°C.* © *1998 Elsevier Science Ltd. All rights reserved*

Since the publication of Calder and Lewin in 1967[1] it is common to assume that the process of outgassing of molecules from the inner surfaces of stainless steel vacuum chambers is determined by diffusion. To reduce outgassing and to deplete the bulk in the near-surface region from diffusing atoms, mainly hydrogen, it is recommended to bake out the chamber at relatively high temperature under vacuum. The exponential increase of the diffusion constant with absolute temperature leads to a high migration rate of hydrogen atoms to the surface where instantly recombination and desorption takes place and the hydrogen atoms will finally be pumped away. This model predicts that the higher the bake-out temperature the lower the succeeding outgassing rate. Therefore "vacuum firing", i.e. heating an austenitic stainless steel vacuum chamber after manufacturing it at 1000°C for several hours under high vacuum conditions, became a common method for producing low-outgassing UHV chambers.

It is an open question, however, which after "vacuum firing" is the best *in-situ* treatment of a so treated UHV chamber. Since after "vacuum firing" at 1000°C, bake-out temperatures up to 300°C will not significantly change the hydrogen distribution[1] and content in the bulk material, the surface composition of the stainless steel may play the dominant role in determining the outgassing rates. Several publications[2-6] appeared in recent years, which showed the importance of oxide layers on outgassing behaviour. The composition and thickness of oxide layers varies also greatly with temperature and influences their quality as diffusion barrier. Also the number of adsorption sites per area may play an important role in the outgassing behaviour.[7]

We have investigated which is a suitable bake-out temperature for low outgassing rates of a "vacuum fired" 316LN (DIN 1.4429, 16,5%–18,5% Cr, 11,5%–14,5% Ni, 2,5%–3% Mo, < 0,03% C) stainless steel vessel (vol 45 l, inner surface area 13,200 cm^2), which will be used in a ultra-high vacuum primary standard for vacuum gauge calibrations. We limited the bake-out temperature on the upper end to 180°C to maintain a corrosion resistant chromiumoxide layer on the air-exposed side of the vessel,[8] on the lower end to 100°C, because we assumed that below this temperature no effective removal of water from the surface can be accomplished.

The bake-outs were performed in the following manner: Before the first bake-out the vessel was pumped for two weeks and the outgassing rate measured to 10^{-10} Pa m^3/s m^2 (10^{-13} mbar l/s cm^2). The evacuated vessel was pressurised to atmospheric pressure by the vent valve of the turbo pump attached to the chamber with 50% argon and then 50% humid air for 3 h to simulate a future change of test gauges. Then the vessel was pumped down to 10^{-5} Pa, after which the bake-out was started with a 20 K/h temperature increase up to the desired maximum bake-out temperature. This temperature was kept constant for 60 h, after which the temperature was reduced by 20 K/h to 100°C where the heater was switched off. Twenty four hours after the system had reached near room temperature, the measurement of the outgassing rate was started.

For this we used the pressure-rise method[7] which we have previously tested to give correct values of outgassing rate.[7,9] The chamber was isolated from the pump system by all metal valves and the pressure rise was measured with a spinning rotor gauge (SRG) over a period of typically 150 h. The gauge reading was corrected for the rotor frequency dependent offset.[10] We assumed that the outgassing molecules were predominantly hydrogen[7,11] and did not measure the gas composition, since it is known that the gas composition is changed by a residual gas analyser itself.[11,12]

Figure 1 shows the measured outgassing rates q (nitrogen equivalent at 23°C) in dependence of bake-out temperature. The numbers above the symbols indicate the order in which the

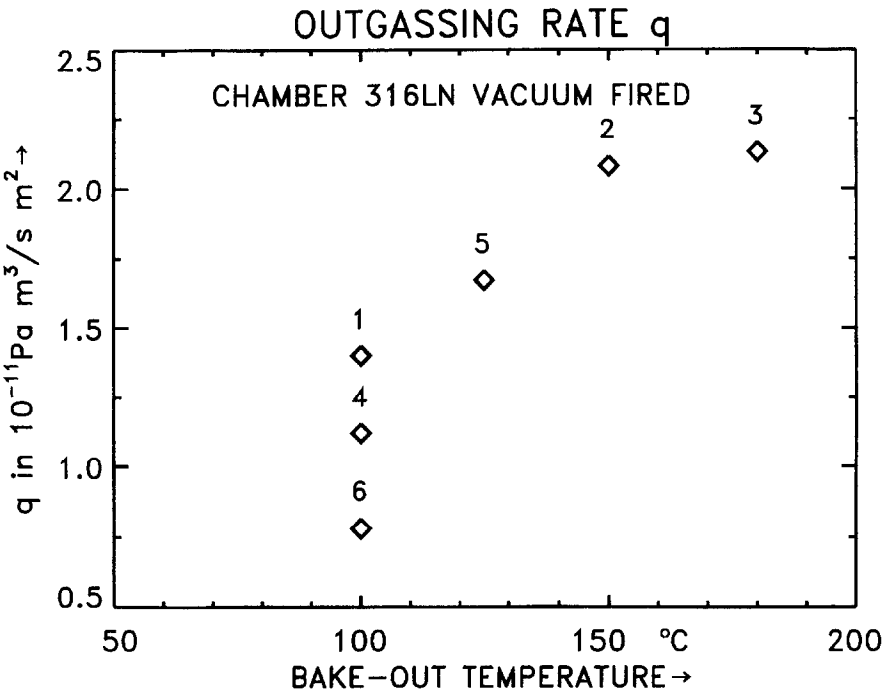

Figure 1. Measured outgassing rate (nitrogen equivalent at 23°C) of the vacuum fired 316 LN stainless steel chamber in dependence on bake-out temperature. The numbers indicate the order of measurements. Between each measurement the chamber volume was exposed to a 50% argon/50% humid air mixture. Relative standard uncertainty is typically 5% (about the size of the symbols).

measurements were performed. The relative standard uncertainty of the obtained values is typically 5%. As described, obtaining each of these points required a period of about two weeks. For this reason no more points could be taken. We have to note here that the total outgassing rate was of the order of 10^{-8} Pa l/s which is about the limit for our leak detector. We can therefore not exclude that leaks $< 10^{-8}$ Pa l/s were present and contributed to the measured outgassing rate. However, it is rather improbable that leaks contributed significantly to the measured rates, because there was a general trend towards lower values at 100°C with repeated bake-outs and no flange was opened in between.

Two effects are apparent from the Figure: (1) The more bake-out cycles at 100°C were performed, the lower q. This was also found by Odaka *et al.*[13] where q reached a steady state only after 4 bake-out/air exposure cycles similar to here; (2) The *lower* the bake-out temperature the *lower* q. The latter is a surprising result when the diffusive outgassing model is applied. As mentioned, in this model higher bake-out temperatures would yield in lower outgassing rates. What might happen instead, is that by exposure to humid air and successive bake-out an oxide layer is formed which serves as effective diffusion barrier. The quality of this oxide layer as barrier seems to be affected by the bake-out temperature. For example, due to different thermal expansion coefficients of the oxide layer and the underlying alloy, the oxide layer may break up at higher temperatures or its chemical composition due to reactions with water may change dependent on temperature.

This investigation strongly indicates that there is a significant influence of the surface of vacuum fired 316LN stainless steel on its outgassing behaviour. A lower bake-out temperature at 100°C appears to be favourable for this type of stainless steel chamber without any built-in parts of other materials.

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