Proposed Cleaning Procedures for the Vacuum Components for the TESLA Test Facility

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Abstract

The beam line vacuum system of the TESLA Test Facility (TTF) has to preserve the cleanliness of the superconducting cavities' surfaces in order not to degrade the performance of the cavities. Thus contamination by any sort of dust or condensation of gases during assembly and operation of the vacuum system has to be absolutely avoided. During assembly of the first stage of the TTF beam line, a lot of experience was gained in cleaning, installing and operating about 80 m of warm vacuum components. UHV cleaning and particle removing procedures are described, which are based on the experience during this installation. The issues of hydrogen outgassing of stainless steel components and possible consequences for the superconducting cavities are discussed in detail.

1 Introduction

In order to build superconducting cavities with 15-25 MV/m for the TESLA Test Facility (TTF), a big effort is currently put into preparing the cavities and all directly connected components to be "particle free". Therefore they are processed in the TTF clean rooms to class 10 level (i.e., ASTM10 - no more than 10 particles/ft^3 larger than 0.3 µm in diameter). After this procedure, the experience with the TTF cavities tested at DESY shows so far, that field emission is negligible if particle cleanliness is preserved throughout the assembly.

One of the major objectives for the beam vacuum system is to preserve the cleanliness of the superconducting cavities' surfaces and thus the operation at high gradients and high quality factor Q. So contamination by any sort of dust or condensation of gases during assembly and operation has to be absolutely avoided.

Up to now there is no experience with a linac working with cavities at such high accelerating gradients. From simple estimates, particle transport from the warm vacuum chambers into the cold cavities by the electron beam seems to be very unlikely due to the pulsed structure of the TTF beam. However, to minimize the risk of particle propagation into the cavities during pump down and venting, all vacuum components to be installed in the TTF linac should be properly UHV vacuum cleaned first **and** made "particle free" as much as possible before installation into the TTF beam line.

Standard cleaning of UHV-vacuum components includes a degreasing of all components before assembly, super purity (ultra pure) water rinsing, and vacuum drying with warm air. Usually this cleaning is followed by baking the component in a vacuum furnace or an in-situ bakeout of the installed system to typically 250-300 °C. In case of a superconducting linac with high accelerating gradient this treatment is not sufficient. As already mentioned in the introduction, a "particle free" assembly is necessary, and a higher baking temperature has to be con-

sidered because of possible hydrogen contamination of the cold cavities.

2 Hydrogen Contamination of cold Cavities

An estimate of the hydrogen coverage in the capture cavity has been presented by D. Trines [1]. As a typical outgassing rate for stainless steel chambers that have not been vacuum fired (i.e. baked at 950 °C under low vacuum, see sect. 3) he takes a value of $d/dt(dQ/dA) = 10^{-10}$ mbar l / sec cm^2 for hydrogen. Taking into account the geometry of the TTF Linac capture cavity section, a hydrogen coverage of 0.1 monolayer/day was calculated. In a summary paper by H. Padamsee [2] it was claimed that already 1 monolayer (equivalent to 10 days of operation) could cause increased field emission. H. Padamsee also describes a CERN experiment in which a decrease of the maximum surface field has been observed; this measurement was done using a gas mixture (see below). As a conclusion D. Trines suggested to vacuum fire all stainless steel components to 950 °C which reduces the outgassing rate by a factor of 10 to 100 [1]. In addition to this, a reduced aperture of the beam line would be valuable for at least short sections next to the cryostat.

In the following a few remarks are added which should help to find a better understanding of the effects of hydrogen coverage. Questions will be raised and statements be made. Answers and comments are welcome since existing laboratory experience is not well documented.

The hydrogen coverage is viewed as adsorption on the cavity surface, which consists of a multilayer of oxides. This oxide layer mainly consists of Nb_2O_5, but NbO and NbO_2 might also exist. From ref. [3] one probably can exclude absorption, i.e. the inclusion of hydrogen into the niobium. Tests with hydrogen purging at 300 K as well as at 2 K showed no evidence of absorption. Unfortunately a quantitative result has not been given. Small cracks in the niobium have been discussed in the late eighties when people searched for an explanation of the so called Q-disease or 100 K effect.

H. Padamsee claimed in ref. [2] that a gas inlet produces field emitters which limit the cavity behavior. As adsorbed gas molecules can change the work function, it is likely that existing emitters are switched on (or off after desorption of the gas). It is also possible that particulates were introduced during this procedure. In the documented experiment, oxygen admission was studied two times. The first attempt was with an undefined volume of oxygen, the second with 100 cm^3 x 100 mTorr ($3.5 \cdot 10^{17}$ molecules). Assuming a single cell, 1.5 GHz cavity (approx. 100 cm^2), this small volume corresponds to about one monolayer, if equally distributed. From [2] the experimental conditions are not clear but changes in field emission were observed. The gas admission was done with the cavity beeing cold so that most of the gas could have condensed along the cold beam pipe wall even before reaching the cavity.

Ref. [2] also states that similar experiments at CERN were carried out. Instead of hydrogen, a gas mixture (H_2:H_2O:CO:CO_2 =69%:17%:8%:6%) was used. Again, a cold cavity was exposed. After one monolayer a sudden drop in the surface field from 10 MV/m down to 7 MV/m was observed. A detailed description of the experimental conditions does not exist. Since 1988 further tests have not been done [4].

Ref. [3] claims that residual gas molecules of CO and CO_2 have a clear influence on the field emission activity. For hydrogen it was not clear, for nitrogen it could be excluded. There are no reported results for oxygen.

Since adsorption is a complicated process which very probably is different for metals and oxides, an experiment should be done with well-defined conditions. The problem is to guarantee that the gas is "particle free". All laboratories dealing with superconducting cavities have this experiment in their plans, but probably nobody has done it so far [5].

3 Outgassing of Stainless Steel

Outgassing rates for austenitic steels (300 series) are given in standard vacuum literature as well as in many publications for various cleaning methods and heat treatments. After some hours of pumping, the outgassing is dominated by hydrogen, which comes from diffusion of molecules out of the bulk of the stainless steel material. High concentrations of hydrogen exist because of one step in its production, when the steel is quenched in a hydrogen atmosphere. Ref. [6] gives the number of $1 \cdot 10^{-12}$ Torr l/cm^2 sec (or $1.32 \cdot 10^{-12}$ mbar l/cm^2) upon chemically cleaned stainless steel tubes after performing a 150 °C in-situ bake of the assembled system. DESY assumes $1 \cdot 10^{-10}$ mbar l/cm^2 sec after standard cleaning without any in-situ baking. While the first number seems to be a little bit too optimistic if compared with literature, the second one is certainly an upper limit.

The calculation of time and temperature versus hydrogen degassing is rather straightforward. For an infinite slab of stainless steel of thickness d, which is sitting in a perfect vacuum environment and heated to some temperature T, the degassing can be described by the one dimensional diffusion equation:

$$D\frac{\partial^2 c}{\partial x^2} = \frac{\partial c}{\partial t} \quad . \tag{1}$$

Fig. 1 shows the diffusion coefficient D for austenitic stainless steel as a function of temperatures. Assuming an initial concentration C_0, the solution of the differential equ. (1) can be written in terms of an infinite sum [6]. Aiming for a certain finite concentration of hydrogen (the maximum at half wall thickness has been taken), this solution can be used to determine the baking time as a function of diffusion coefficient. Fig. 2 gives a numerical fit for a reduction to 1% of the initial concentration which is at least valid for material thicknesses between 0.5 and 3.5 mm, and finite concentrations between 10% and 0.1% of the initial value.

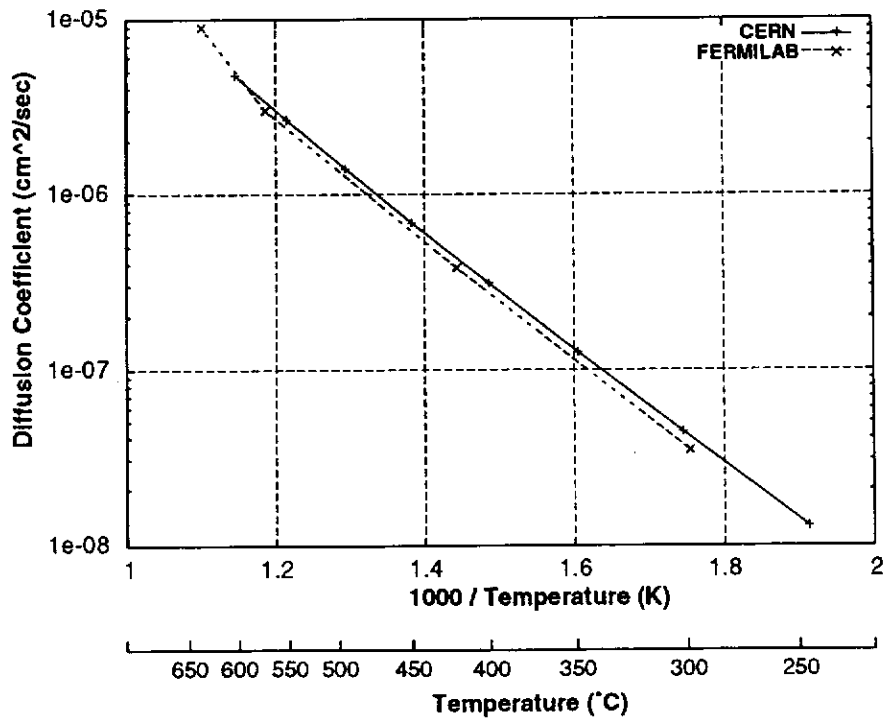

Figure 1: Hydrogen diffusion rate as a function of temperature in austenitic stainless steel. The two different references agree quite well.

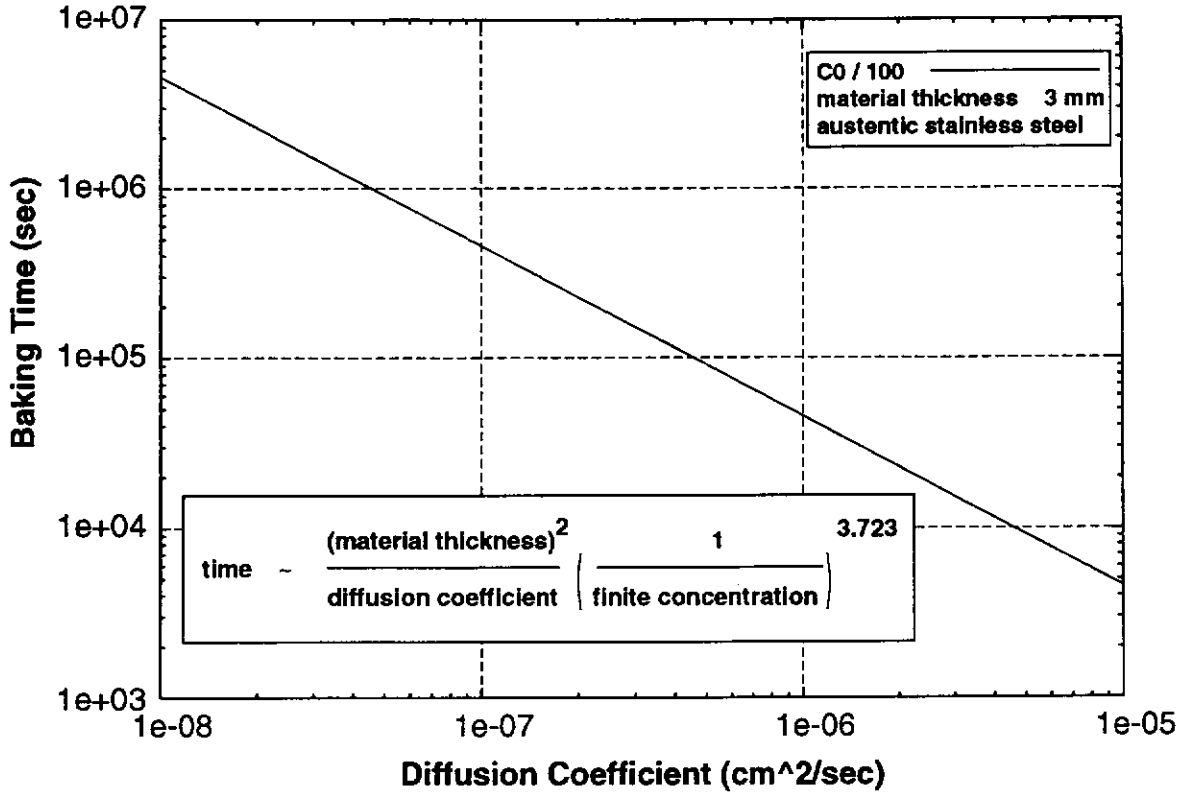

Figure 2: Baking time for 3 mm thick stainless steel to reduce the hydrogen outgassing to 1% of the initial concentration.

At CERN a recipe for vacuum firing has been universally adopted: stainless steel components are baked at 950 °C for 2 hours in a clean oven at very low pressure ($p < 10^{-5}$ mbar) as shown in fig. 3 [7]. This recipe is a result of work carried out in 1965 by R.S. Calder and J. Lewin at CERN. The 2 hour duration at 950 °C results from the difficulty of removing hydrogen from the thick UHV flanges. This means, that if one can neglect the outgassing from the flanges, a much shorter duration would be effective. The procedure takes into account not only the diffusion time, but also the carbides precipitation which in many austenitic steels takes place between 600°C and 800°C. This temperature range has to be avoided and is specified in the bake out cycle to be run through in less than 30 min. during cooldown. (All this information from ref. [5] and [7]).

At FERMILAB the plan for the recycler vacuum system is to bake the components at 500°C for 6 hours. This will guarantee full degassing. From figs. 1 and 2 it is clear, that at 500°C (i.e. a diffusion coefficient of slightly above $1 \cdot 10^{-6}$ cm^2/sec) the reduction to 1% of the initial concentration is achieved after a few thousand seconds, i.e. one hour for materials up to 3 mm thickness.

4 UHV Vacuum Cleaning

The fabrication of the vacuum components for the TTF linac should follow the UHV standard procedures (e.g. as given by CERN) including a careful cleaning of all components after fabrication. In the following some important steps are explicitly mentioned for stainless steel components. The treatment of other material than stainless steel should be discussed individually as needed.

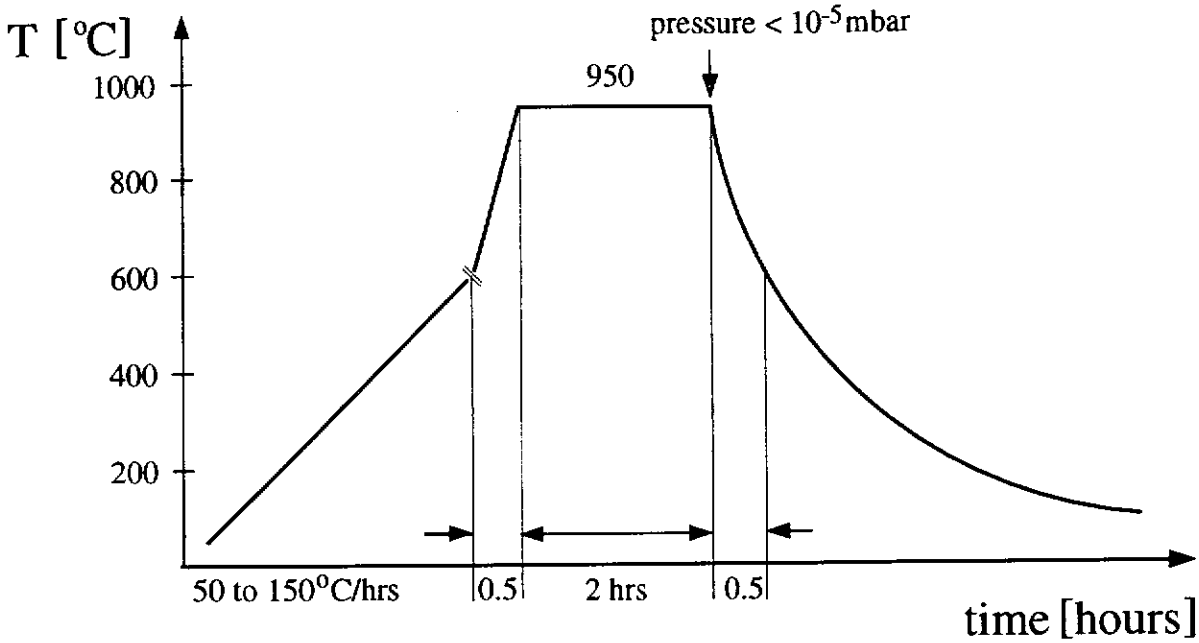

Figure 3: Heating cycle for vacuum firing of stainless steel components as developed at CERN [7].

For welding of stainless steel components, the following steps should be taken into account:

- All components must be thoroughly degreased before welding.
- The welds should be of top quality, i.e. fully penetrating, ...
- The welding areas must be very well protected with inert gas.
- The welds should not be ground, but brushed with clean stainless steel brushes.

To reduce the above mentioned outgassing of the enclosed hydrogen molecules from stainless steel components, they should be baked for 2 h at 950 °C in a vacuum oven with $p < 10^{-5}$ mbar (vacuum firing) after fabrication and cleaning. In order to bake stainless steel components at this high temperature without losing its mechanical strength, all tubes should be made from SS 316 L (1.4435/1.4404) and flanges from SS 316 LN (1.4429). If vacuum chambers or components can not be vacuum fired after final fabrication due to other materials used, e.g. ceramics, the vacuum firing should be done during the fabrication process before connecting them with other components.

After the vacuum firing all vacuum components should be taken into the clean room to make them "particle free" and to do the assembly. If this is not possible, the assembly of components (e.g. diagnostic elements) should be done under clean conditions after cleaning properly all individual parts. Clean gloves should be used for the assembly work.

5 Cleaning Procedures to remove Particles

To avoid contamination of the cavity surfaces by dust, all vacuum components should be made "particle free" by the following procedures:

- The **degreased** components are taken into a clean room, class 10,000.

- Whenever possible all components should be placed in an ultrasonic bath with alkaline solution (e.g. ultra pure water with 1‰-1% Tickopur [8]) for several minutes followed by water rinsing with ultra pure water untill a residual restistance of 12 MΩ/m and drying in class 10-100. This might be continued by vacuum drying starting with a conventional pump stand for pressures down to 10^{-4} mbar followed by pumping with oilfree pumps.
- Fragile components (e.g. grids) or components which can not be washed should be placed in a clean room for several hours prior to assembly. The constant air flow reduces the number of possible dust particles.
- The assembly of components (e.g. diagnostic elements) should be done in a class 10 or class 100 area after cleaning properly all individual parts even before assembling sub-units.
- For chambers and assembled units, where water rinsing is not possible (either due to the materials used or due to partial assembly at another place), the bigger particles will be removed by blowing clean gas through the chambers (purging) followed immediately by pumping. This procedure has be repeated several (5) times. Particle control is done by a particle counter during this procedure. This procedure is usually done using local clean rooms, i.e. laminar flow, at the gas inlet and at the position of the particle counter.
- If testing of partially or completely assembled units like monitors etc. outside the clean room can not be avoided after cleaning, clean gloves should be used when working with previously cleaned components. The pump and purge procedure should be applied afterwards.
- After vacuum drying or pump and purge, the vacuum chambers will be vented with clean argon gas, blank flanged and all flanges, that will be opened later for further installations, will be covered with plastic bags.
- Mounting of all components in the TTF beam line is done using local clean rooms with tools, screws, bolts and gaskets all cleaned in the clean room before and bagged for transportation.
- All flanges should be permanently covered such that collection of dust particles in the leak check channels of the flanges is avoided.
- Well defined procedures for the later handling of each component (venting, pumping, changing parts, etc.) have to be worked out in detail to avoid particle flow in the direction of the cavities.

In order to collect the information and experience from the TTF beam line for the full TESLA beam line system a written history/check list for each vacuum chamber, pump, valve, diagnosis element, etc. installed in the TTF beam line is made which should contain:

- the materials used,
- the treatment (cleaning, etc.) of the component,
- mass spectrum after cleaning,
- particle control protocol,
- installation date,
- opening/closing of flanges, and
- things that went wrong with that unit.

6 Experience with the TTF Warm Beam Line Vacuum System

About 80 m of the warm TTF beam line vacuum system have been installed and are under vacuum since a few months. The system has been opened several times at various positions to add or change components.

All stainless steel beam pipes, bellow sections and most of the monitor chambers have been vacuum fired after fabrication. All components including the ion getter pumps and gate valves as well as ceramic pieces and membrane bellows have been washed and assembled inside the clean room. Membrane bellows and valves are however difficult to clean due to the large number of narrow slits and corners, and particles might be produced once the bellows or valves are moved. All components have been installed in the beam line using local clean rooms. Both the cleaning and assembly in the clean room as well as the installation procedure with local clean rooms were more time consuming than the standard assembly and required well trained personnel.

The operating pressure of the system is between a few times 10^{-10} and 10^{-11} mbar measured by the ion getter pumps. Short pressure bursts are observed when monitors like the OTR-screens are moved in and out by long membrane bellows.

For the pump down movable oilfree pump stations are connected through manual valves attached to the ion getter pumps far away from the cold sections. Once the pump station is fully operational, the manual valve is opened slowly. However a perfect laminar pump down is certainly not achievable and the risk to transport dust or other particles inside the vacuum chambers during pump down is quite high.

Dry N_2 gas from a liquid N_2 dewar is used for venting. Therefore manual valves are installed close to the cold sections, such that the gas stream is directed away from the cavities. The connection of the vent line to the vent valves is done using local clean rooms. The valves are opened only partially in order to vent in the laminar regime. By this slow venting turbulences of the gas stream and thus the transport of dust or other particles inside the vacuum chambers should be avoided.

The vacuum chambers of the injector have not been vacuum fired and thus have a higher outgassing rate as the chambers of the beam line (see section 3). Once the capture cavity has been cooled down, the gate valves up- and downstream of the cavity have been kept closed most of the time, when no beam was present. This way it should be avoided to pump the neighbouring warm sections by the cold cavity. In addition there have been several times problems with the cryogenic system resulting in warming up of the cavity and thus releasing the adsorbed gases from the cavity walls. From the experience so far it is not yet possible to draw any conclusion about degradation of the cavity performance by hydrogen covarage .

7 Conclusions

About 80 m of warm beam pipes including pumps, gate valves and monitors have been installed in the TTF linac. The system is under vacuum since a few months and has been opened several times to add or change components. The cleaning methods described in this report are a summary of the procedures used during this installation. The detailed procedures for the more complex structures like the monitors have been discussed individually. The quite time consuming methods finally paid off in assembling a very clean system with respect to vacuum properties and contamination by particles.

In order to facilitate the described procedures, components under design should be based on a clean room applicable design.

References

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