PURIFICATION OF 6 GHz CAVITIES BY INDUCTION HEATING

A.A. Rossi, A. Battistello, M. Checchin, V. Palmieri, S. Stark, F. Stivanello, R.K. Thakur, G. Yu, Instituto Nazionale di Fisica Nucleare, Laboratori Nazionali di Legnaro, Italy

Abstract

We have developed an innovative technique for purification of bulk-Nb 6 GHz RF cavities under High vacuum (HV) and ultra-high vacuum (UHV) system. The main advantages of 6 GHz bulk-Nb cavities are saving cost, materials and time to collect statistics of surface treatments and RF test at temperature up to 1.8 K.

Induction heating method is used to anneal the cavity at temperatures higher than 2000°C and close to the melting point of Nb for less than a minute while few seconds at maximum temperature. Before RF test and UHV annealing, the surface treatment processes like tumbling, chemical, electro-chemical (such as BCP and EP), ultrasonic cleaning and high pressure rinsing (HPR) have been employed. Cavities are RF tested before and after high temperature treatment.

This kind of Nb 6 GHz cavity purification allow to reduce hydrogen, oxygen and other elemental impurity contents, which effect on cavity Q-factor degradation, by a rapid annealing over 2000°C and a subsequent rapid reduction at room temperature.

INTRODUCTION

Ultra high pure (UHP) bulk Niobium (Nb) is widely used for the fabrication of Superconducting (SC) radio-frequency (RF) cavities to achieve high Quality factor (Q_0) and accelerating fields (E_{acc}), which are the figures of merit. Therefore, rapid High Temperature (HT) purification by means of Induction Heating (IH) helps improve these figures of merit.

The technology of SRF involves the application of superconducting materials for radio frequency devices, where the ultra-low electrical resistivity allows obtaining high Q-factor in RF resonator, which means that the resonator stores energy with very low loss and narrow bandwidth [1]. These properties of RF superconducting cavities have wide applications in Scientific and Industrial research, including the construction of high efficiency Particle Accelerator structures. For example, thousands of SRF cavities are required for the proposed International Linear Collider (ILC) to probe new physics at TeV collisions of heavy ion beams. Nb is the most suited SC for SRF application.

In the present work, 6 GHz cavities are investigated in order to optimize cavity parameters that simulate the real condition with new superconducting materials. A spinning technology is used to create seamless 6 GHz Bulk-Nb cavity [2]. The main advantages of these cavities over a 1.3 or 1.5 GHz SRF cavities are saving time, cost, and materials. One can fabricate such small 4-6 cavities out of the left over Nb sheet material which was used to prepare just one big (i.e., 1.3 or 1.5 GHz) SRF cavity with the same technology, and carryout measurements for various treatments like mechanical tumbling, buffered chemical polishing (BCP), electro-chemical polishing (EP), heat treatment (i.e., induction heating), and triple cavity RF test (one by one) in the same cryostat (cryostat is designed to house 3 cavities at a time) in short time, say in a week. So, we have improved these cavities, for which after all the above mentioned treatments we get about 2 to 3 order of magnitude improvement in terms of Q-factor.

In this article, we discuss the new purification techniques by induction heating method, giving special attention to important new developments that enable easy and quick study and statistic on surface treatments of cavities. Such as, for examples, chemical processes and cryogenic test are simpler. Also reducing the production costs of high quality bulk Nb SRF cavities. We have also compared RF performance of the cavities which are annealed under atmospheric pressure, HV and in UHV system for long and short duration (i.e., 10 sec. up to 5 minutes).

Induction heating technique is described in section 2. Surface Treatment methods are described under Experimental Procedure in section 3. Results and Discussion are given in section 3. Summary of the RF test procedures are given under Conclusion section.

INDUCTION HEATING TECHNOLOGY

Induction heating is a tool, allowing one to rapidly heat only the metal object without contact. It is comprised of three basic factors: electromagnetic induction, the skin effect, and heat transfer. IH is the process of heating an electrically conducting object (usually a magnetic material) by alternating current (AC) electromagnetic induction, where eddy currents are generated within the metal through an inductor and resistance leads to Joule heating of the metal. The frequency of AC used depends on the object size, material type, coupling (between the work coil and the object to be heated) and the penetration depth.

In other words, in an inductor, an AC current of suitable frequency flows and creates an alternating magnetic flux. The flux link with any ideal conducting path in the work-piece to be heated induces an AC current which heat the work piece by Joule effect.

Induction heating offers several inherent advantages. The major advantages of this system are; contactless heating: absence of pollution from the source of heating. High power densities in a wide range of frequency and consequently short heating times: heating speed linked to the possibility of obtaining very high power density, Bulk or surface heating. A process perfectly adapted to industrial medium-sized and mass production requirements. Easy automation of equipment, absence of

ISBN 978-3-95450-143-4

thermal inertia (rapid start-up), and repeatability of operations carried out.

A new technique has been developed for purification of bulk-Nb SRF 6 GHz cavities in order to reduce RF surface resistivity (R_S). Such purification has been accomplished either under HV or UHV system or under cover of He or Ar gas (inert gas) at atmospheric pressure (ATM). Initially HT purification started in HV followed by ATM inside quartz tube and finally in UHV has been done to compare the result of different systems.

One of the most significant characteristic of niobium is its ability to react actively with the gases H_2, O_2, H_2O, N_2, CO, CO_2, Ti, Ta, and oxides of all these elements and compounds [3]. In the solid state, the elements H, N, O and C are dissolved interstitially in niobium. All these impurities greatly reduce RF performance (higher R_s). Also, stresses and dislocations introduced by mechanically rolling, deep drawing, electron beam welding (EBW) and/or spinning deformations of the niobium metal sheet have negative effect on R_s.

We suppose that the new HT-IH technique, presented in this paper, could reduce both, surface contaminations and crystallographic defects by HT evaporation and recrystallization respectively which provides the highest possible purity (RRR) of metal.

EXPERIMENTAL PROCEDURE

High Vacuum (HV) and Ultra-high Vacuum (UHV) Procedure

An induction heating has been done with two different configurations of vacuum chamber. In first configuration (HV setup), the partial pressure is high during and also after the annealing, i.e., 10^{-5} mbar up to few mbar. Several cavities are annealed (Cavity identity number 115 up to 126) using this configuration. In second configuration (UHV setup), the partial pressure is high during the initial stage of annealing, in the range of pressure 10^{-6} mbar up to 1 mbar. Several cavities are annealed (Cavity identity number 127 up to 133) using this configuration.

At first, HV and UHV system along with all other components are cleaned in ultrasonic, dried with N_2 gas then assembled. The cavity is centred against the coil; this in turn is connected to the work head. The work head connected with the power supply where time and voltage are controlled, and approximately 15 kW of maximum power allowed while frequency is 85 KHz in the system. The cavity is electrically insulated from the chamber, tested and finally chamber is closed with CF flange using Cu gasket. Chamber is pumped for some hours to achieve approximately $2*10^{-8}$ mbar pressure using a turbo molecular pump.

Thereafter, chamber is backed at approximately 130 ºC for 60 hours. After the backing vacuum is improved about one order of magnitude (ie, $5*10^{-9}$ mbar), than water cooling system is turned on for cooling induction coil, UHV chamber, work-head and power supply. Soon after power supply is switched on, and power to the coil is increased starting from few watt up to 12 kW quickly (say in 30-40 seconds) which reach temperature of 2000 ºC and more. At maximum temperature cavity is left to heat for some seconds (i.e., 10-60 sec.) then power supply is switched off. Pyrometers (Pyrospot DG 10N and/or DS 10N model) are employed to read the temperatures between 250-1300ºC and 900-3000ºC respectively. Water cooling system is also turned off after 20 minutes.

Once the cavity is at room temperature, chamber is fluxed with extra-pure helium.

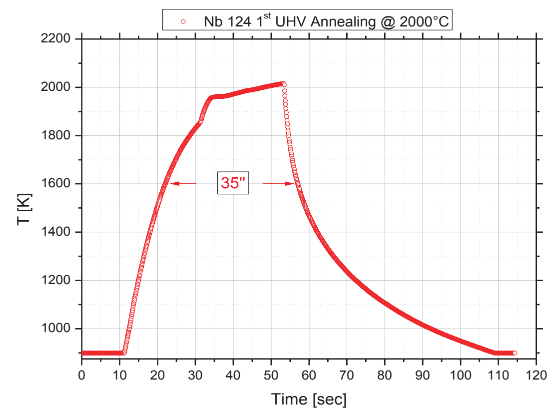

Figure 1: Graph represents the annealing process at UHV.

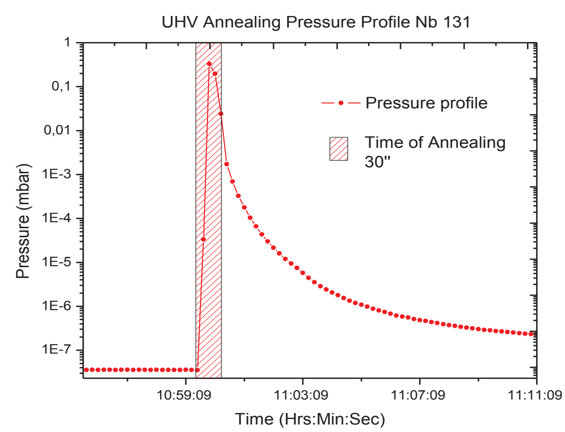

Figure 2: A typical pressure profile during annealing process.

Soon after the heat treatment cavity is cleaned with High Pressure Rinsing (HPR). Thereafter, cavity is mounted on RF stand inside clean room, pumped out for several hours to achieve up to pressure 10^{-8} mbar using roughing/turbo molecular pump and placed inside cryostat for RF test at temperatures from 4.2 K to 1.8 K. A typical annealing profile of cavities using UHV system is shown in Figure 1, while a typical pressure profile during annealing process is depicted in Figure 2.

Atmospheric Pressure (ATM) Procedure

ATM-IH system consists in a Quartz tube of length 1 meter and diameter 8 cm. Quartz tube is sealed with Viton O-ring and aluminium flanges at top and bottom. Cavity is centered against the coil inside a clean quartz tube. Coil is connected to the work head, and work head to induction heating system.

The bottom part of quartz tube is fluxed with extra-pure helium gas or other inert gas for few minutes before heating and flushing continues even after heating is switched off for next few minutes, hence to prevent oxidation by the atmospheric gases in the tube which protects the inner and outer surface of cavity before, during and after the thermal treatment. At maximum temperature cavity is left to heat for some seconds up to few minutes, approximately 15kW of maximum power allowed while frequency is 125 KHz in the system, then power supply is switched off. Cavity needs relatively more time to approch maximum temperature due to the heat exchange at atmospheric pressure. Pyrometer (DS 10N model) is employed to read the temperatures between 900-3000ºC. Annealing profile of cavity is shown in Figure 3.

Cavities are heated to 2000°C–2350°C. In order to capture the oxygen coming out of the Niobium and to prevent oxidation by the residual gas in the quartz tube, extra-pure ^{4}He gas is used (as a flux medium) for few minutes before and after heating to protect the inner and outer surface of cavity. However, we cannot claim that quartz tube is completely clean system. Nevertheless, clean quartz tube has fewer contaminations than UHV chamber.

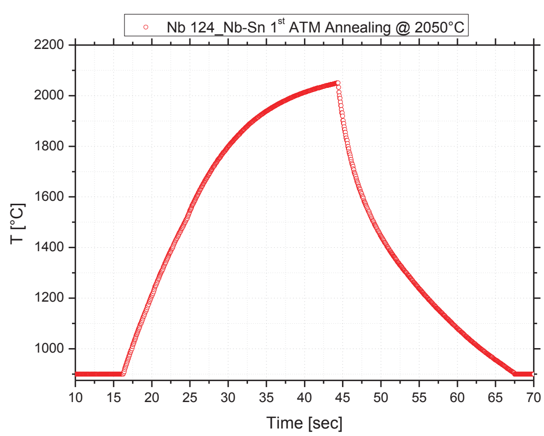

Figure 3: Graphical representation of annealing process at atmospheric pressure.

RESULTS AND DISCUSSION

Several cavities have been annealed with the three different configurations (HV, ATM and UHV) and then RF tested at 1.8 K as shown in Figure 7.

Prior to HT-IH experiments internal surface has been treated (i.e., successive removal of inner materials by vibrating tumbler, CBP and, BCP and/or EP. About 150 μm of inner defected layers are removed during the tumbling, while about 400 μm of inner layers are removed during EP. RF tests data (Q_o vs E_{acc} curve) before and after high temperature annealing between temperatures 1980-2230 °C are shown in Figure 4-6. Cavities which are treated in HV system showed higher Q_o but lower E_{acc}, while cavities treated in UHV showed higher E_{acc}, but lower Q_o.

RF test (Q_o vs E_{acc}) data obtained before and after IH treatment in HV (configuration 1) are shown in Figure 4. Q_o have been enhanced approximately 3 orders of magnitude after IH treatment. Q_o ($3*10^9$) is measured highest in this configuration while E_{acc} is between 16 up to 22 MV/m measured. R_s is measured about 50μΩ before thermal treatment which reduced up to 70 nΩ at 1.8 K after the HT-IH treatment for cavity Nb 121. Cavities treated in HV system where turbo pump is not connected directly to the process chambers (hence gas pressure is high during the initial stage of annealing and after annealing) shows higher Q_0 but lower E_{acc} in comparison to UHV treatment.

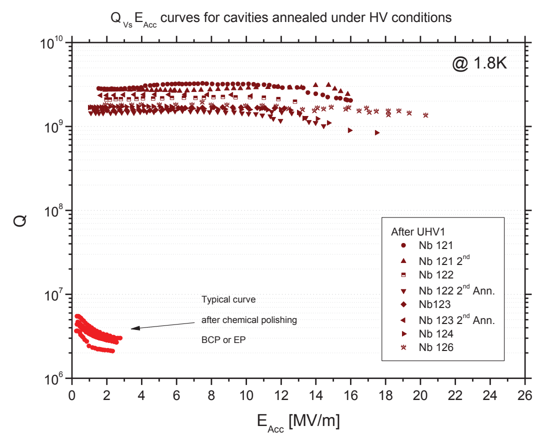

Figure 4: Quality (Q_0) factor vs E_{acc} are shown. RF tests were done before annealing (lower Q_0), while higher Q_0 obtained after HV annealing.

However, one cavity (over 4 produced) having IH treatment in UHV system produced better RF performance in terms of accelerating field. It is shown in Figure 5. Cavity Nb 129 showed best performance. It is EP treated, and IH has been done for 120 seconds at 1900°C. It showed highest accelerating field, > 23 MV/m with Quality factor of ≈ $2*10^9$ and R_s ≈ 140 nΩ among all measured cavities, though X-ray was detected after 18 MV/m. The cavity is subjected to HT under UHV system where turbo pump is directly connected to the main annealing chamber which reduces residual gas pressure during the initial stage of annealing and after annealing, pumping residual volatile contaminations (coming out from cavity).

In Figure 6, RF data are obtained after IH treatment in ATM treatment. Q_o have been increased more than 2 orders of magnitude after IH treatment. Cavity Nb 121 showed better result among all ATM treated cavity. For this cavity, R_s is measured about 50 μΩ before thermal treatment which reduced up to 325 nΩ at 1.8 K after the HT-IH treatment.

ISBN 978-3-95450-143-4

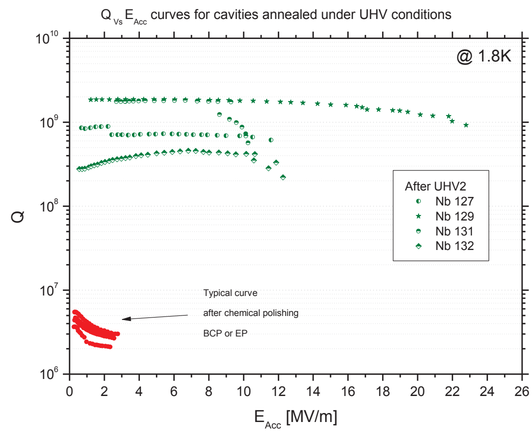

Figure 5: RF tests were obtained before and after UHV annealing (max. E_{acc} measured so far).

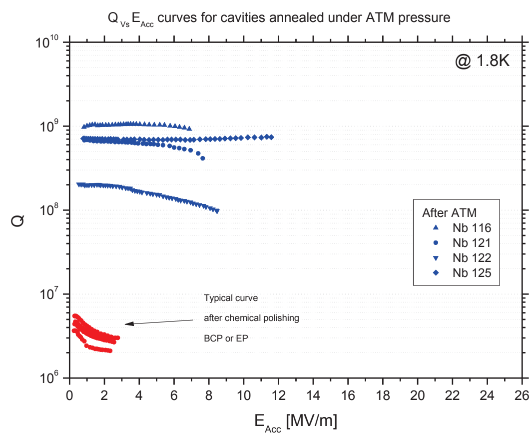

Figure 6: RF tests data, obtained before and after ATM pressure annealing.

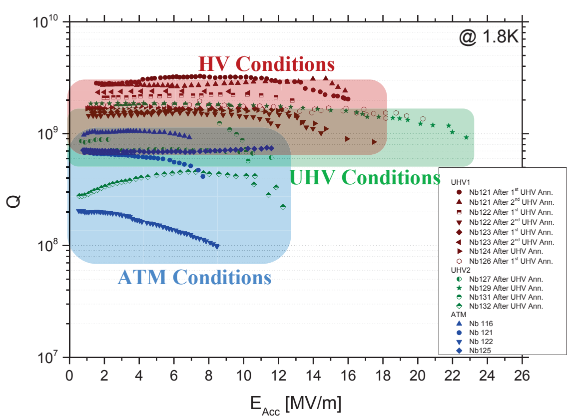

Figure 7: A comparison of RF test at 3 stages is shown. RF tests were done after HV annealing, UHV annealing, and atmospheric pressure annealing.

A comparison has been depicted in Figure 7. In this graph RF test data obtained before and after IH treatment at 3 different systems has been plotted. From the Figure we conclude that HV and UHV system provide better result in two different ways. HV system shows higher Q_0 but lower E_{acc}, while UHV system showed lower Q_0 but higher E_{acc}. In this context, further attention and modification is required toward fast cooling down after IH treatment which could further optimize RF performance.

Furthermore, in UHV system efficiency is higher than the atmospheric pressure treatment inside quartz tube. Efficiency of IH increase between 50% to 95%, and one can reach very high temperature, hence very high power density goes inside the work piece because the induction coil better match the cavity geometry (i.e., coil is much closer and in better shape).

CONCLUSION

An innovative, 6GHz niobium cavity, high temperature purification technique based on induction heating have been presented systematically. Several cavities were purified to corroborate this technique. RF test confirmed that the cavities obtained after different surface treatment procedure especially under HV/UHV system above 2000°C for few seconds, shows values of Quality (Q_o) factor up to $3*10^9$, surface resistance $R_s \approx 70$ nΩ and accelerating field $E_{Acc} > 23$ MV/m (@ 6GHz).

Application of IH with high vacuum technology with respect to atmospheric tecnology increases the quality of niobium purification and lowers residual gas concentrations. Therefore, due to ultra-high vacuum there is much less heat exchange with residual gases between work piece and heating system, hence most of the power density goes to the cavity. That also helps reduce time scale to approach maximum temperature. During the purification process, evaporation of absorbed gasses, metallic impurities and recrystallization takes place which helps to improve SRF resonators.

Further steps on 6 GHz cavities are planned to heat fast (as it was done), but also employing a cryogenic trap in order to perform both a cavity fast cooling and also a fast pumping of residual gasses which could improve figures of merit Q and accelerating fields E_{Acc}.

Moreover, the system of quartz tube has several advantages in comparison to the UHV system, i.e. it is transparent with respect to electromagnetic waves used for IH, it is also perfectly light system. Therefore, the combination of quartz tube like vacuum chamber with a turbo pump directly connected to it using IH technique in UHV system could result in cost effective and fast system, with respect a classical stainless steel UVH vacuum chamber, making affordable the rapid purification treatment of 1.3 GHz Nb cavity.

ACKNOWLEDGMENT

Authors extend thanks to Cryogenic laboratory members of INFN-LNL. Many thanks to Dr. Gabriele Puglierin, Dr. Luca Taffarello and Mr. Paolo Modanese, Mr. Andrea Calore, Mr. Alessandro Friso and Mr. Tiziano contran for their efforts to providing consistently and quickly the liquid helium needed for the hundreds of rf tests carried out in the last two years and for helpful discussion and constructive comments.

REFERENCES

[1] R. Russenchuck, G.Vandoni. "Superconductivity and Cryogenics for accelerators and detectors". CERN Accelerator School. (2004).
[2] V. Palmieri, Seamless cavities: the most creative topic in RF superconductivity, Proceedings of the 8th workshop on RF superconductivity (1997), vol. 3, P.553.
[3] Wilfried Rockenbauer, "Production of Niobium Metal and Compounds from Tantalite" in Niobium - Proceedings of the International Symposium, ed. by H. Stuart (Warrendale,PA, The Metallurgical Society of AIME, 1984), 133-152.