SYSTEMATIC INVESTIGATION OF MID-T FURNACE BAKING FOR HIGH-Q PERFORMANCE*

H. Ito^{1,†}, H. Arakio¹, K. Takahashio², K. Umemorio^{1, 2} ¹KEK, 305-0801 Tsukuba, Ibaraki, Japan ²SOKENDAI, 305-0801 Tsukuba, Ibaraki, Japan

Abstract

We report on an investigation of the effect of a new baking process called "furnace baking" on the quality factor. Furnace baking is performed as the final step of the cavity surface treatment; the cavities are heated in a vacuum furnace in a temperature range of 200 to 800°C for 3 h, followed by high-pressure rinsing and radio-frequency measurement. We find the anti-Q slope for cavities furnace-baked at a temperature range of 250 to 400°C and a reduction in the residual resistance for all cavities. In particular, an extremely high Q value of 5×10^{10} at 16 MV/m and 2.0 K is obtained for cavities furnace-baked at 300°C.

INTRODUCTION

Decades of continuous research for the improvement of the performance of superconducting radio-frequency (SRF) cavities [1, 2] have resulted in the establishment of various surface treatments; thus SRF cavities with superior performance in terms of the quality factor (Q_0) and accelerating gradient (E_{acc}) have been developed [3–5].

In this study, we investigated the increase of the Q_0 by a new heat-treatment method called "furnace baking" [6] which is a simpler method than the surface treatment methods such as nitrogen doping [7,8], nitrogen infusion [8,9], and two-step baking [10] that have been proposed and investigated in recent years.

EXPERIMENT

In the furnace baking process, a baking process in a vacuum furnace is performed following light electropolishing. After baking, high-pressure ultrapure water rinsing (HPR) is performed to remove any remaining impurities on the inner surface of the cavity and then assembled in a cleanroom. Finally, RF measurement is performed. In this study, these processes are repeated for 1.3 GHz single-cell cavities at different baking temperatures to investigate the relationship between baking temperature and Q-E behavior in furnace baking.

A large vacuum furnace is used for furnace baking [11]. The vacuum system consists entirely of oil-free pumps and can be depressurized to 1×10^{-6} Pa at room temperature using a cryopump. To prevent the inner surface of the cavity from being contaminated by the furnace, the cavities are placed in a large vacuum furnace with the flanges covered

using niobium caps (see Fig. 1). The vacuum furnace is equipped with Quadrupole Mass Spectrometer (Q-mass) to monitor the partial pressure of each element during heat treatment. The main elements during heat treatment are mass 2, 18, 28, 44 which correspond to H_2 , H_2O , N_2 , CO_2 respectively, and the signals of the high mass molecules are small (see Fig. 2).



Figure 1: Photograph of inside of vacuum furnace and single cell cavity with flange covered by niobium cap.



Figure 2: Temperature and Q-mass trends during 300°C 3 h furnace baking and Q-mass data at each moment. Red curve in upper left panel shows temperature trend of control temperature sensor, other curves show Q-mass trend of each element.

Figure 3 shows a photograph of the RF measurement setup including the heater, temperature sensors, and fluxgate sensors. Magnetic shielding inside the cryostat and a solenoid coil mounted at the outside of the cavity keep the magnetic field around the cavity below ~ 1 mG. In addition, flux expulsion is performed using a heater place at the top beam tube to minimize the flux trapping. The magnetic field around the cavity is monitored with fluxgate sensors. A temperature mapping system is equipped to detect the heating point.

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[†] hayato.ito@kek.jp

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Figure 3: Photograph of RF measurement setup.

RESULTS

Q-E Curves

Figure 4 shows a comparison of the Q-E curves measured at 2 K for single-cell cavities that were furnace-baked for 3 h at the various temperatures (200 to 800° C) and a standard treated cavity (120° C 48 h baking).



Figure 4: Comparison of Q-E behaviour measured at 2 K for cavities that were furnace-baked at various temperatures (200 to 800°C) and a standard treated cavity (120°C 48 h baking).

The 200°C 3h furnace-baked cavity (medium purple points) shows generally similar Q-E behavior to that of the standard treated cavity (black point), although slightly different at lower $E_{\rm acc}$. This indicates that the conventional cavity performance can be reached by replacing the 120°C 48 h baking to the 200°C 3 h furnace baking, which would be significantly effective for mass production due to the shorter heat treatment time. Mid-T furnace-baked cavities, baked at 250 to 400°C, have high Q_0 and anti-Q slope, although a lower $E_{\rm acc}$ than that of the standard treated cavity. In particular, 300°C furnace-baked cavities have an extremely high Q_0 of 5 \times 10¹⁰ at 16 MV/m. This high Q_0 is comparable to the nitrogen-doped cavity. These results show that the Mid-T furnace baking has the potential to produce high Q_0 cavities with a very simple surface treatment process compared with nitrogen doping. The high temperature furnace-baked cavi-

field Q slope was observed.

$R_{\rm BCS}$ and $R_{\rm res}$

Figure 5 shows the R_{BCS} behavior at 2 K which is normalized by R_{BCS} at 2 MV/m. The normalized R_{BCS} behavior is classified into three types: one that increases with increasing E_{acc} , one that does not increase as much as the first type, and one that decreases increasing E_{acc} . The 200°C 3 h furnace-baked cavity and the standard treated cavity correspond to the first type. And the high temperature furnacebaked cavities correspond to the second type. Finally, the Mid-T furnace-baked cavities correspond to the third type and the anti-Q slope of the Mid-T furnace-baked cavities originate from this normalized R_{BCS} behavior.

ties baked at 500 to 800°C did not achieve high Q_0 and high



Figure 5: Normalized R_{BCS} behavior at for each furnacebaked cavity and standard treated cavity.

The slope of the normalized R_{BCS} for each cavity is estimated using the difference between the maximum or minimum value of the normalized R_{BCS} and the value of normalized R_{BCS} at 2 MV/m and the difference in E_{acc} between them. Figure 6 shows the relationship between the slope of the normalized R_{BCS} and baking temperature. While the slope is large in the positive direction below 200°C, it turns negative sharply at 250°C, then reaches a minimum value



Figure 6: Relationship between slope of normalized R_{BCS} and baking temperature.

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at 300°C and slowly turns positive at higher temperatures. This drastically change in the slope from 200 to 300°C may be related to the decomposition of niobium pentoxide (decomposition temperature: 250 to 300°C [12–14]) and the diffusion layer of oxygen obtained from it.

Figure 7 shows the $R_{\rm res}$ behavior for each furnace-baked cavity and standard treated cavity. All of the furnace-baked cavities have a low $R_{\rm res}$ compared with that of the standard treated cavity. Especially, 600°C furnace-baked cavity has an extremely low $R_{\rm res}$ of 0.2 n Ω which corresponds to a Q_0 of 1 × 10¹². This could be a useful surface treatment method for superconducting devices that are operated in the mK temperature range and desire a high Q value.



Figure 7: R_{res} behavior for each furnace-baked cavity and standard treated cavity.

Figure 8 shows the relationship between R_{BCS} , R_{res} , and baking temperature at 10 MV/m. The Black points depict the R_{BCS} which is an absolute value, not normalized R_{BCS} . And the blue open circles depict the R_{res} . It was found that the minimum point is different between them: R_{BCS} has a minimum at 300°C furnace baking and R_{res} has a minimum at 600°C. While the minimum point for R_{BCS} is probably related to the decomposition of the niobium oxide and diffusion of oxygen into bulk niobium from it mentioned above, it is unclear what is the cause for the minimum point of R_{res} .



Figure 8: Relationship between R_{BCS} , R_{res} , and baking temperature at 10 MV/m.

Sensitivity and Frequency Change

The sensitivity to trapped flux for each cavity was estimated by comparing the Q-E curve after cooling down with flux expulsion in <1 mG and Q-E curve after cooling down slowly in a 20 mG field (the magnetic field of 95% was trapped by slow cooling down). The sensitivity S describes the amount of increase of surface resistance R_s (ΔR_s) per unit of trapped magnetic field B_{trap} and is expressed as

$$S = \frac{\Delta R_s}{B_{\text{trap}}},\tag{1}$$

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where R_s is calculated using $R_s = G/Q_0$. *G* is the geometric factor that is independent of material properties [15].

Figure 9 shows the relationship between baking temperature and sensitivity at 2 MV/m. The sensitivity is high at the Mid-T temperature range (250 to 400°C). Especially, the 300°C furnace-baked cavity has higher sensitivity compared with other cavities and there is a structure that has a sharp peak at around 300°C. While the high Q-value of 300°C furnace-baked cavity is very attractive, this sensitivity must be carefully taken into account to install it into a cryomodule and operate it as an accelerator component because it is difficult to realize the ideal magnetic shielding in a cryomodule as in RF measurement system.



Figure 9: Relationship between baking temperature and sensitivity at 2 MV/m.

In order to further understand the characteristics of the inner surface of the cavity, the change in resonance frequency during warm-up was measured with a network analyzer. Since most of the frequency changes start at 8 K, we defined ΔF as the difference between the resonant frequency at 8 K and the resonant frequency at 9.5 K after the transition. Figure 10 shows the relationship between baking temperature and ΔF .

It was found that the 300°C furnace-baked cavity has an abnormally large frequency change compared with other cavities and there is a structure that has a sharp peak at around 300°C similar to the relationship between baking temperature and sensitivity. From these results, 300°C is the most 20th Int. Conf. on RF Superconductivity ISBN: 978-3-95450-233-2

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Figure 10: Relationship between baking temperature and ΔF .

characteristic temperature for frequency change as well as for R_{BCS} and sensitivity, which means the superconducting properties of the cavity surface such as penetration depth and mean free path are most characteristic at 300°C in the furnace baking method.

CONCLUSION

In this study, a new heat treatment method called "furnace baking" has been investigated at several baking temperatures ranging from 200 to 800°C. It is found that the Q-E behavior is sensitive to the baking temperature and a high Q value is obtained at 250 to 400°C furnace baking. In this temperature range, $R_{\rm BCS}$ behaves as decreasing with increasing $E_{\rm acc}$, which leads to a high Q value and anti-Q slope. In particular, 300°C furnace-baked cavities have an extremely high Q_0 of 5×10^{10} at 16 MV/m. Furthermore, 300°C is a characteristic temperature for furnace baking because these cavities are highly sensitive and have a large frequency change. Since this temperature is slightly higher than the decomposition temperature of niobium pentoxide, it is expected that there is a relationship between oxygen diffusion and the high Q value.

Now, sample study is undergoing to clarify the relationship between cavity performance and surface conditions, such as the state of the oxidation layer on the inner surface of the cavity and the diffusion of oxygen in the depth direction. For future work, we further investigation for furnace baking, for example, applying to the 9-cell cavity and LG cavity, and changing the parameter of baking time to achieve both high-Q value and high E_{acc} are planned.

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