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RECENT DEVELOPMENT ON NITROGEN INFUSION WORK TOWARDS HIGH Q AND HIGH GRADIENT*

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Abstract

We report the rf performance of several single-cell superconducting radiofrequency cavities after low temperature baking in nitrogen environment. The cavities are treated at different temperature in the range of (120-160ºC) for extended period of time (~48 hours) with and without nitrogen gas injection in the furnace. The improvement in Q0 with Q-rise in some case was observed when nitrogen gas was injected at elevated temperature (~250-290 ºC) and held at the temperature range 120-200 ºC without any degradation in accelerating gradient over the baseline performance. When nitrogen gas in injected at lower temperature (~120 ºC) the rf performance did not show any improvement on Q0 over the conventional 120 ºC in-situ bake.

INTRODUCTION

Recent advances in the processing of bulk superconducting radio frequency (SRF) niobium cavities via interior surface impurity diffusion have resulted in significant improvements in their quality factor (Q0). The motivation for the development of these processes is to reduce the cryogenic operating cost of current and future accelerators while providing reliable operation [1-3]. Most recently, efforts have been made to preserve high accelerating gradients while also increasing the quality factor of SRF cavities [4-6]. In these new nitrogen "infusion" cavity processing recipes, cavities were heat treated at 800 °C for 3 hours, then the furnace temperature is reduced to 120-200 °C and nitrogen is introduced into the furnace at a partial pressure of ~ 25 mTorr for ~48 h. This process has shown an improvement in Q0 over the baseline measurements, without the need for post-annealing chemical etching. Even though diffusion of the nitrogen into the bulk of the SRF cavity is limited in depth at these low temperatures (120-200 °C) [7], the introduction of nitrogen is sufficient to modify the cavity surface within the rf penetration depth as seen from rf results, which are similar to those previously reported for high-temperature nitrogen doped cavities. Furthermore, while post-doping electropolishing is required to remove coarse nitrides from the surfaces of high-temperature nitrogen doped cavities, no further processing is required for the low-temperature "infusion" recipe showing a clear benefit in reducing processing steps as well as keeping higher gradient with high Q0 values. In this manuscript, we present the results from several rf tests on a single cell cavity treated in low temperature nitrogen environment as well as analyses of sample coupons treated under similar conditions.

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CAVITY SURFACE PREPERATION

The cavities used in this study consists of one low loss shape single cell 1.5 GHz (RDL-02, Low loss shape, Bp/Eacc = 4.19 and geometric factor, G= 277.23) and four 1.3 GHz, TESLA shape (Bp/Eacc = 4.23 and geometric factor, G= 277.85) fabricated in house with high purity fine grain Nb from Tokyo Denkai. Prior to the rf measurements reported in the manuscript, the cavities went through several R&D cycles of heat treatments, chemical polishing and nitrogen doping. The baseline rf measurements reflect the surface reset via electropolishing by removing ~ 30 µm from the inner cavity surface.

Before the heat treatment, the cavity was high pressure rinsed and then dried in an ISO4/5 cleanroom. While in the cleanroom, special caps made from niobium foils were placed to cover the cavity flange openings as shown in Fig. 1. The cavity was then transported to the furnace in a clean sealed plastic bag. The vacuum heat treatment procedure started with the 800 ºC/ 3hours degassing step followed by lowering temperature to (120-200 ºC) range. The furnace is continuously pumped during the cooldown process. Two different gas injection mechanisms were explored; (a) the gas was injected at ~ 250-290 ºC at which the total pressure (also corresponds to nitrogen partial pressure) increased to ~ 25 mTorr by introducing high purity nitrogen. Such pressure was maintained without active pumping of the furnace enclosure. Once the temperature has fallen to the desired value (120-200 ºC), which takes about 2 hours, the temperature was held for 46 hours. (b) The gas was injected in to the furnace when the desired temperature was reached (120-200 ºC) and was held for ~48 hours without active pumping.

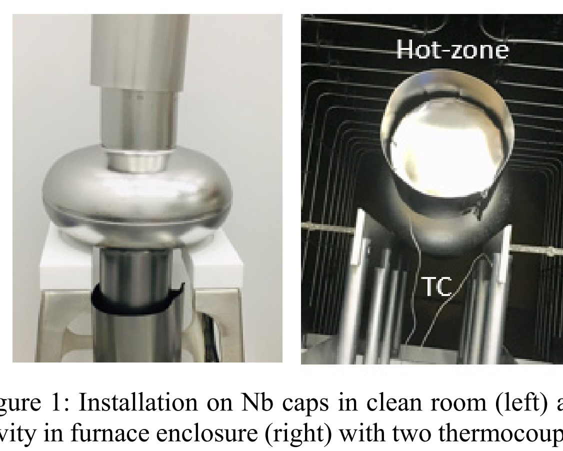

Figure 1: Installation on Nb caps in clean room (left) and cavity in furnace enclosure (right) with two thermocouples attached to cavity surface.

During our earlier cavity heat treatments, we relied on the temperature measured by a thermocouple attached to a panel inside the hot-zone. Later, the modification was

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made such that the thermocouple can be directly attached to the cavity surface to accurately measure the cavity temperature. On average, the cavity temperature is about ~20-30 ºC higher than the hot-zone plate. Furthermore, the fluctuation of temperature is smaller on the cavity compared to hot-zone plate. Figure 2 shows the typical temperature and pressure measured during the heat treatment process. After the heat treatment, the standard cavity cleaning procedure was applied before the rf test. Each cavity's surface was reset by ~10 μm by electropolishing before the cavity goes for next heat treatment cycle.

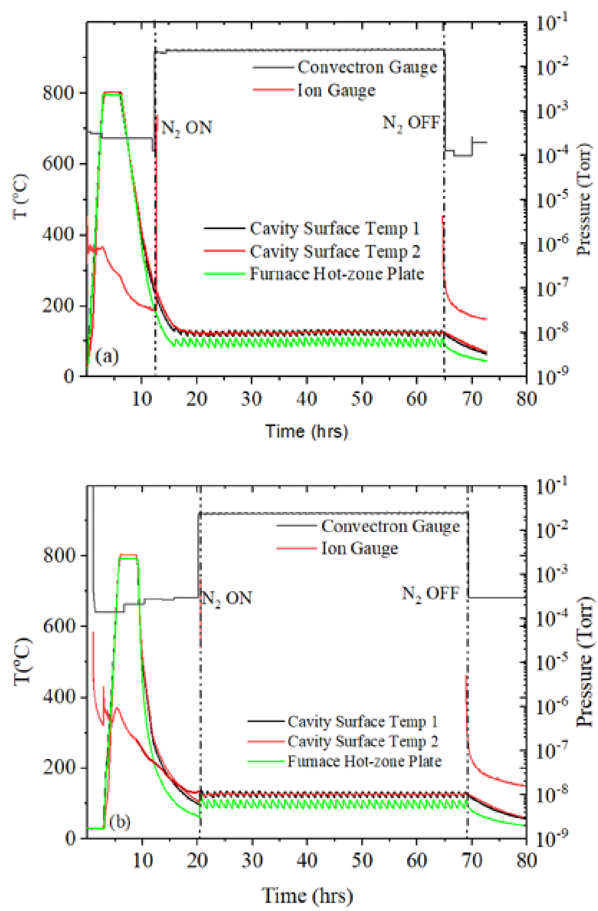

Figure 2: Typical heat treatment cycles. (a) N_2 injection at higher temperature (250-290 ºC) during the cooldown of furnace from 800 ºC. (b) N_2 injection when the cavity surface reaches the target temperature.

CAVITY TEST RESULTS

Standard procedures were followed to clean the cavity surface in preparation for an rf test: degreasing in ultrapure water with a detergent and ultrasonic agitation, high pressure rinsing with ultrapure water, drying in the ISO4/5 cleanroom, assembly of flanges with rf feedthroughs and pump out ports and evacuation. The cavity was inserted in a vertical cryostat and cooled to 4.2 K with liquid helium using the standard Jefferson Lab cooldown procedure in a residual magnetic field of <2 mG. This procedure results in a temperature difference between the two irises ΔT > 4 K when the equator temperature crosses the superconducting transition temperature (~9.2 K), which provides good flux expulsion conditions. Most of the rf measurements were done at 2.0 K acquiring $Q_0(E_{acc})$ curves and in some occasions $Q_0(T)$ from 4.2-1.5 K was obtained at low accelerating gradient in order to extract the residual resistance.

RDL-02

The cavity RDL-02 was treated with the profile similar to that shown in Fig. 1(a), where the nitrogen gas was injected at elevated temperature. The baseline measurement was limited by high field Q-slope at E_{acc} = 40±2 MV/m and in-situ LTB at 120C for 48 hours extended the high gradient to 46±2 MV/m, which corresponds to a peak magnetic field of 196±8 mT, close to the thermodynamic critical field of Nb at 2.0 K. The summary of rf measurements are presented in Table 1 and also in Fig. 3. The details of the analysis was already presented in Ref. [6].

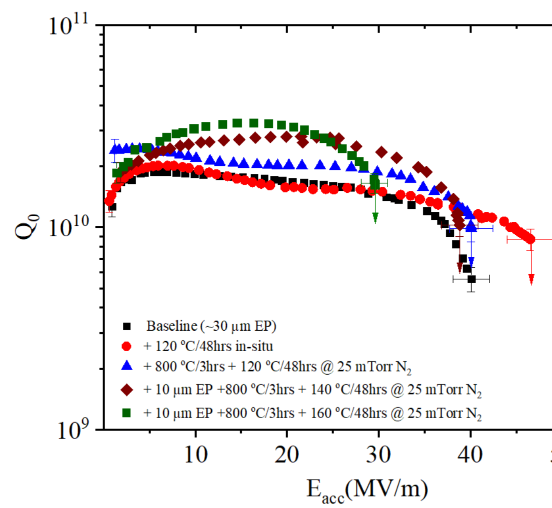

Figure 3: $Q_0(E_{acc})$ at 2.0 K for cavity RDL-02. The arrow represents that the cavity was limited by quench. The baseline measurement was limited by Q-slope. The temperature during heat treatment was measured on the hot-plate zone.

RDT-14

The cavity was limited by high field Q-slope at E_{acc}~31±2 MV/m with $Q_0 = (0.8±0.1)×10^{10}$. After the baseline test, the cavity was subjected to the heat treatment recipe described in Fig. 1(a), where the furnace temperature was maintained at ~140±10 ºC during nitrogen hold which corresponds to cavity surface temperature being ~ 160 ºC. The cavity quenched at 36±2 MV/m with Q_0 = $(1.4±0.2)×10^{10}$. The reduction of Q-slope is consistent with the conventional in-situ baking, however a clear increase in Q_0 was observed over the whole range of accelerating gradient. Figure 4 shows the $Q_0(E_{acc})$ curve at 2.0K.

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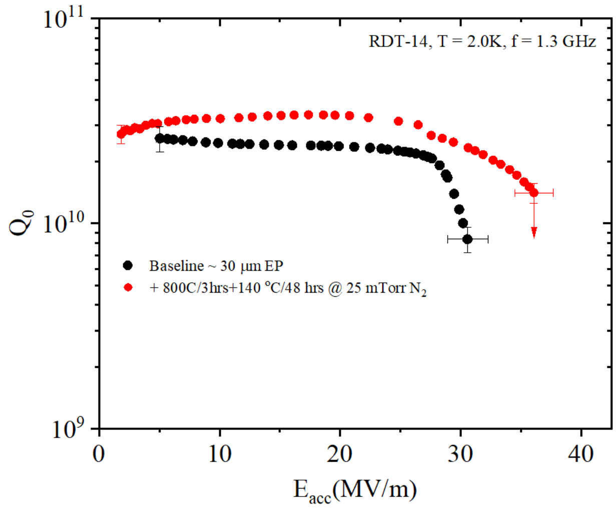

Figure 4. $Q_0(E_{acc})$ at 2.0 K for cavity RDT-14. The arrow represents that the cavity was limited by quench. The baseline measurement was limited by Q-slope. The temperature during heat treatment with nitrogen was measure on hotplate zone.

RDT-06

The rf measurement on cavity RDT-06 was done with 5 different conditions. The baseline measurement was done after ~ 30 µm EP and the cavity was limited by high field Q-slope at 31±2 MV/m. During the test of this cavity, a new recipe was reported [8], where the cavity was in-situ baked at lower temperature (~70 ºC) for few hours before ramping up to 120 ºC and held for ~48 hours. This recipe resulted in higher Q_0 compared to conventional 120 baking. After the baseline test the cavity was baked at ~75 ºC for 4 hours and increased the temperature to 120 ºC and held for 110 hours. The malfunction of the controller resulted in the extended baking time. The baking was performed on test stand while the cavity was continuously pumped. The rf test shows the decrease in Q_0 with elimination of Q-slope while the accelerating field increased to 39±2 MV/m (see Fig. 5). Since the baking time and condition wasn't exactly the same as reported in ref. [8], we weren't able to confirm the result presented. Nevertheless, the high field Q-slope was cured as a result of low temperature baking.

The cavity was then subjected to 800 ºC for 3 hours followed by 120 ºC baking for 48 hours in furnace while no gas injection and no active pumping. This step was done with Nb caps being installed on cavity in order to check the quality of the furnace. The rf results showed that the quality factor didn't change in medium field range < 30 MV/m from the previous measurements with similar gradient increase within the measurement error. This confirmed the good quality of furnace without any contaminations.

Next, the cavity was subjected to 800 ºC for 3 hours followed by 120 ºC baking for 48 hours in the furnace while ~25mTorr of nitrogen was injected with the recipe similar to that shown in Fig. 1(a), where the gas injection occurred ~250 ºC, during the cooldown from 800 ºC. The temperature was directly measured on the cavity surface. The rf measurement showed an excellent performance with both increase in Q0 and Eacc over the previous test. The cavity quenched at gradient 44±2 MV/m with Q_0 = $(1.4\pm0.2)\times10^{10}$. The cavity was then subjected to ~8 µm inner surface removal via EP and the performance of the cavity was limited by Q-slope at 41±2 MV/m with high Q0.

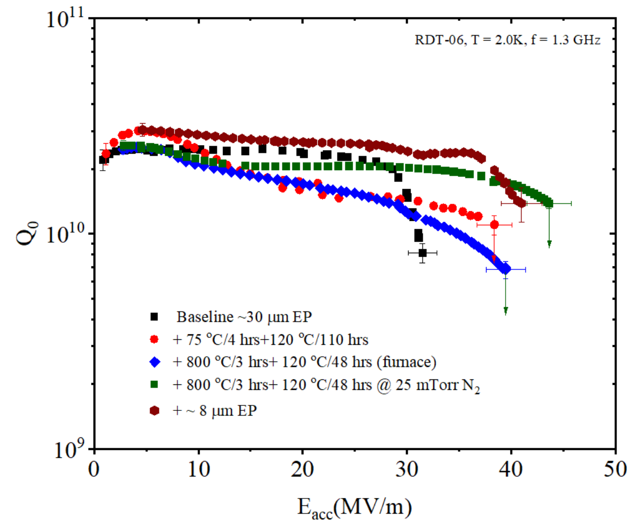

Figure 5. $Q_0(E_{acc})$ at 2.0 K for cavity RDT-06. The arrow represents that the cavity was limited by quench. The baseline measurement was limited by Q-slope. The temperature with nitrogen during heat treatment was measured on cavity surface.

RDTTD-01

The baseline rf rest was limited by high field Q-slope at 32 ± 1 MV/m. The cavity was then subjected to the 120 ºC baking in nitrogen with gas injected ~250 ºC. The cavity was limited by field emission at 33±2 MV/m with FE onset ~21 MV/m. The test results are shown in Fig. 6

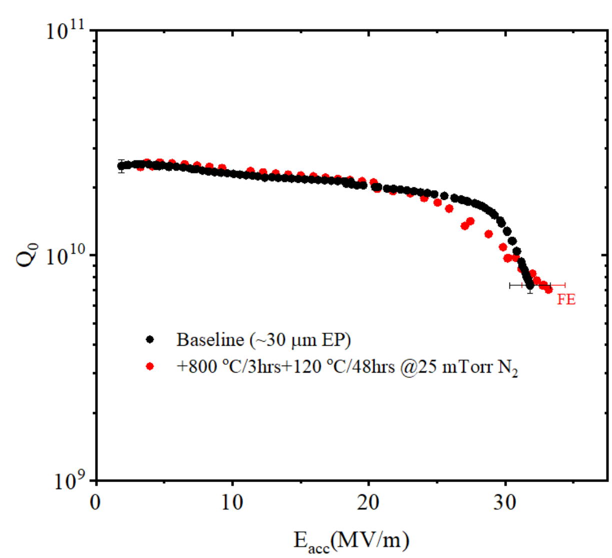

Figure 6: $Q_0(E_{acc})$ at 2.0 K for cavity RDTTD-01.

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RDTTD-02

The baseline measurement was limited by high field Q-slope at 29 ± 1 MV/m. After the baseline test, the cavity was subjected to 75 ºC bake for 4 hours followed by 120ºC bake for 48 hours in test stand. The cavity was limited by quench at 37 ± 2 MV/m with Q_0 = (1.1±0.2) ×10^{10}. After this test, the cavity's inner surface was reset by ~ 30 µm EP to measure the new baseline. The new baseline measurement was also limited by high field *Q*-slope at 33±2 MV/m. As a final test, the cavity was heat treated at 800 ºC for 3 hours followed by 120 ºC for 48 hours while the nitrogen gas being injected into the furnace when the temperature of cavity surface reaches to ~120 ºC (scheme (b) in Fig. 2). The cavity was limited by quenched at 28±1MV/m with no change in Q_0 over the baseline test. The test results for this cavity are shown in Fig. 7.

The summary of rf test results for all cavities is summarized in Table 1.

Table 1: Summary of RF Test Results

Cavity ID	f (GHz)	Cavity Treatment	$E_{acc,max}$ (MV/m)	$Q_0(E_{acc,max})$ 10^{10}	Limitation
		Baseline EP (~30 µm)	40±2	0.56±0.07	Q-slope
		+120 C/ 24 hours in test stand	46±2	0.87±0.09	quench
RDL-02	1.5	+800 C/3hrs+120/48hrs with 25 mtorr N_2#	40±2	1.0±0.1	quench
		+baseline+800 C/3hrs+140 C/48hrs with 25 mtorr N_2#	39±2	1.1±0.1	quench
		+baseline+800 C/3hrs+160 C/48hrs with 25 mtor N_2#	30±1	1.7±0.2	quench
RDT-14	1.3	Baseline EP (~30 µm)	31±2	0.8±0.1	Q-slope
		+baseline+800 C/3hrs+140 C/48hrs with 25 mtorr N_2#	36±1	1.4±0.2	quench
		Baseline EP (~30 µm)	32±1	0.82±0.08	Q-slope
RDT-06	1.3	+75 C /4hrs + 120 C/110 hours in test stand	39±2	1.1±0.1	quench
		+800 C/3hrs+120 C/48hrs in furnace	40±2	0.68±0.06	quench
		+800 C/3hrs+120 C/48hrs with 25 mtorr N_2#	44±2	1.4±0.1	quench
		+ ~ 8 µm EP	41±2	1.4±0.3	Q-slope
RDTTD-01	1.3	Baseline EP (~30 µm)	32±1	0.74±0.05	Q-slope
		+800 C/3hrs+120 C/48hrs with 25 mtorr N_2#	33±2	0.72±0.04	FE
		Baseline 1 EP (~30 µm)	29±1	0.8±0.1	Q-slope
RDTTD-02	1.3	Baseline 1 +75 C/4hrs+120 C/48hrs in test stand	37±2	1.1±0.2	quench
		Baseline 2 EP (~30 µm)	33±2	0.94±0.07	Q-slope
		Baseline 2+ 800 C/3hrs+120C/48hrs with 25 mtorr N_2	28±1	1.6±0.1	quench

#gas injected at elevated temperature (250-290ºC)

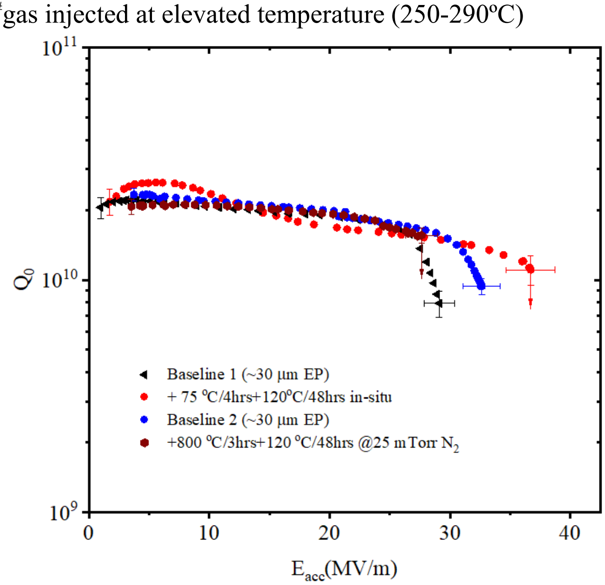

Figure 7 $Q_0(E_{acc})$ at 2.0 K for cavity RDTTD-02.

DISCUSSION

In the past, low temperature baking (100-150 ºC) under UHV has been the standard practice for the final preparation of SRF cavities in order to recover from the high field *Q*-slope. The LTB not only eliminated the high field *Q*-slope; it showed an increase in the achievable gradient, most likely due to the operator stopped the measurements once the *Q*-slope is steeper and rf coupling to the cavity becomes weaker. In fact, recent studies showed that that the cavity can sustain higher field and power dissipation is as high as 200 W for a single cell cavity [9]. Nevertheless, a comprehensive model capable of explaining all of the experimental results related to the high field *Q*-slope and baking effect is still lacking.

In our present study, the introduction of nitrogen during the low temperature baking showed improvement on Q_0 as well as the elimination of high field *Q*-slope similar to that obtained by LTB in UHV environment. The improvement on Q_0 was clearly evident when the N_2 was injected at higher temperature ~ 250-290 ºC during the cooldown of the cavity from 800 ºC. Our earlier studies showed that the presence of the $NbN_{1-x}O_x$ layer between the bulk niobium and top most Nb_2O_5 layer may be responsible for the high Q_0 [6]. The electronic properties of such layer and their influence on the electronic density of states of the adjacent superconducting Nb might explain the difference in the rf performance of "nitrogen infused" cavities compared to those which were subjected to the standard UHV baking.

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The rf measurements and sample surface analysis showed that the heat treatment at lower temperature significantly alters the rf surfaces, mostly driving the superconducting Nb towards the dirty limit where the electronic mean free path is closer to superconducting coherence length with the rf penetration depth. A recent theoretical model proposed by Gurevich extends the zero-field BCS surface resistance to high rf fields, in the dirty limit [10]. Such model calculates $R_s(H)$ from the nonlinear quasiparticle conductivity $\sigma_1(H)$, which requires knowledge of the quasiparticles' distribution function. Qualitatively, the model was able to reproduce the Q-rise phenomenon observed several high temperature impurity doped and low temperature baked cavities in nitrogen environment [6]. The surface modification due to thermal treatments are also evident from the sample coupons study using x-ray photoelectron spectroscopy, magnetization and ac susceptibility measurements done on the samples those were treated along with the cavities [11].

SUMMARY

Improvement in the quality factor of an SRF Nb cavity was observed after annealing at 800 °C/3 h in vacuum followed by baking at 120-160 °C in low partial pressure of nitrogen inside a furnace ("N-infusion") compared to the traditional 120 °C bake in UHV. The improvement in Q_0 was observed only when the gas was injected in the furnace at an elevated temperature during the cool-down from the 800 ºC. During this study, a total of 18 rf tests were performed on 5 different cavities subjected to several surface treatments. The average accelerating gradient $E_{acc} = 37 \pm 5$ MV/m with $Q_0 = (1.1\pm0.3)\times10^{10}$ was observed. Such performance would be of great interest for lowering the cryogenic heat load of high-energy accelerators such as the proposed Linear Collider [12].

As previously reported [6], the near surface impurity management may be responsible for the rf performance on these cavities. Further studies are ongoing to better identify the role of impurities and precipitates on the cavity performance. Explorations of several parameters such as the duration of bake time, optimal temperature and partial pressure of nitrogen is ongoing.

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