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MEDIUM TEMPERATURE TREATMENTS OF SUPERCONDUCTING RADIO FREQUENCY CAVITIES AT DESY*

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Abstract

Over the last years several different approaches to increase the performance of superconducting radio frequency (SRF) cavities by heat treatments have been developed and tested. At DESY, the R&D aims for cavities with enlarged quality factors while maintaining high accelerating gradients, since an envisaged upgrade of the European XFEL requires both. For this purpose, medium temperature (mid-T) treatments around 300 °C seem to be very promising. Lately, the furnace infrastructure at DESY was refurbished and now a niobium-retort furnace capable of carrying 1.3 GHz nine-cell cavities can be used for R&D studies. Vertical test results of single-cell cavities treated in this furnace at medium temperatures are presented and compared to four cavities treated similarly in a furnace at the company Zanon Research & Innovation Srl (Zanon). All mentioned cavities show enlarged quality factors but at the same time reduced gradients compared to their reference measurements before the mid-T treatment. The DESY treatments were accompanied by small niobium samples for surface analyses, which are also presented. Furthermore, the influence of post-treatment high pressure water rinsings is studied.

MEDIUM TEMPERATURE TREATMENT

Fundamental in-situ medium temperature (around 300 °C) bake studies [1] showed very high quality factors. Two studies [2, 3] applied then mid-T treatments using commercial UHV furnaces followed by subsequent cleaning and assembly steps under air. This modified process allows a possible future application for accelerator cavities. The typical cavity performance – described by quality factor Q_0 versus accelerating gradient E_{acc} (Q(E)) – is very similar to those of nitrogen doped cavities [4, 5], showing a rise of Q_0 culminating at an E_{acc} of about 16 MV/m, which is often called “anti-Q-slope”. It has to be investigated, whether the mid-T treatment results in similar gradient limitations like specific doping recipes [4,6] or whether it is capable of high gradients above 30 MV/m.

TREATMENTS AT ZANON

Since the furnace infrastructure at DESY was under commissioning, in the year 2021 a cooperation with Zanon was established in order to study the effect of mid-T treatments of SRF cavities. Two sets of two 1.3 GHz single-cell cavities, were treated for three hours at 300 °C. The first set of cavities was treated completely open in one furnace run, while the cavities of the second set were closed by DESY caps. One cavity per set was low temperature treated beforehand [7], while the other one came directly from an electropolishing (EP) procedure. All cavities were vertically tested before the furnace treatment in order to define a baseline performance.

Furnace Infrastructure at Zanon

The furnace chamber is made of stainless steel and actively water cooled, providing a usable space of (0.6 x 0.6 x 1.3) m^3. Thermal shield layers are made of molybdenum, as well as the heaters. The temperature is controlled over three zones achieving a uniformity of ± 5 °C using ten T-sensors, which are not placed directly on the treated cavities. A maximum operation temperature of 1250 °C is allowed and the furnace provides a starting pressure of 1×10^{-8} mbar at room temperature.

Results of Zanon Treatments

After the heat treatment at Zanon and a high pressure rinsing (HPR) at DESY, the vertical performance test took place. Compared to the baseline tests, all quality factors of the 2 K measurements are enhanced, while simultaneously the maximum accelerating gradient is decreased. For three of the four cavities relatively large (enhanced) residual resistances (R_{res}) in the range of $5 - 8$ nΩ have been deduced, which mask the clear reduction of R_{BCS}. The only exception is cavity 1DE9, which shows by far the best result, not only in terms of R_{res}, and hence it will be discussed here in detail. The Q(E)-curves before and after the mid-T treatment are shown in Fig. 1. This cavity underwent no low temperature treatment before and was closed by caps during the treatment. Some field emission was recorded in the test after the mid-T treatment. After an additional HPR, the cavity improved significantly to a $Q_{0,max}$ of 4.6×10^{10} measured at 2 K, the R_{res} is with 2 nΩ very low by DESY standards and the accelerating gradient reaches 24 MV/m.

For all of the curves shown in Fig. 1 – and elsewhere in this contribution – a test-to-test uncertainty of about 10% in E_{acc} and up to 20% in Q_0 can be assumed [8], even if it is for legible reasons not depicted. The uncertainty within curves of one single vertical test is about a factor of two lower.

Summarising the treatments at Zanon, it can be stated that relatively large R_{res} often mask the reduction of R_{BCS}. All 2 K-curves are also shown in the summary section in Fig. 4 marked by a “Z”. Caps are important for mid-T treatments in this furnace, since the two cavities treated without them, show the lowest gradients and higher R_{res}. Also, a prior

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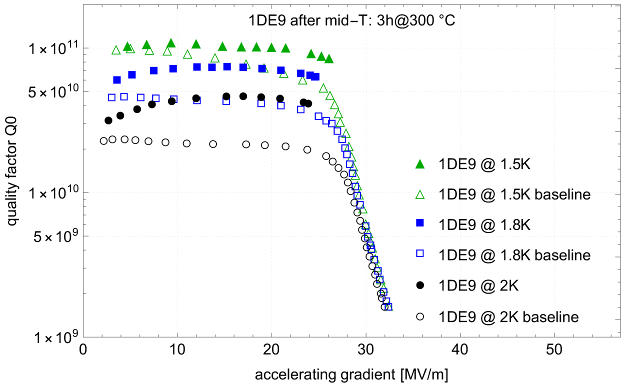

Figure 1: 1DE9 before and after mid-T treatment at Zanon with caps, no low T treatment was applied beforehand, hence the high-field Q_0-slope is visible is in the baseline.

low-T bake seems to be unfavourable (cf. 1DE7 and 1DE5 in Fig. 4) in terms of E_{acc} and Q_0. Hence, it was not applied for the further studies at DESY.

Effect of Additional HPR Treatments

Additional HPR improved the cavity with field emission a lot, while three others showed no difference after HPR. This could be confirmed also with the cavities treated at DESY, which did not change their performance due to an additional HPR applied after the first mid-T test.

Niobium sample studies concerning surface modification caused by different amounts of HPR cycles are in full swing at the moment at the University of Hamburg.

TREATMENTS AT DESY

By the end of 2021, the first heat treatments became possible in the newly refurbished all niobium furnace at DESY. Up to now, the furnace qualification and two successful treatments at 800 °C took place as well as three mid-T treatments. The latter are subject of this section.

Niobium-Retort Furnace

Attached to the ISO 4 area of the cavity assembly clean room, the furnace consists of a niobium-retort with a separate supporting vacuum containing the heaters. The usable diameter is 0.3 m and the depth 1.3 m, so that a 1.3 GHz nine-cell cavity or two single-cell cavities can be treated at once, all hanging in vertical position as shown in Fig. 2. The maximum reachable temperature is 1200 °C. The complete refurbishment consisted of a renewal of the complete vacuum, cooling & control system and the implementation of a partial pressure control & mass spectroscopy. The cryo pumping system is oil-free and provides a base pressure at room temperature of 2×10^{-8} mbar. The pressure during a cavity treatment at 800 °C is about $3 - 4 \times 10^{-7}$ mbar. In parallel to first successful cavity runs, the commissioning of the furnace is not yet fully completed i.e. improving the water cooling, control features, qualification of partial pressure system and establishing reproducible treatment protocols.

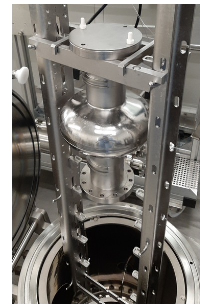

Figure 2: Single-cell cavity with DESY cap mounted on the insert of the niobium-retort furnace (visible at the bottom).

DESY Treatment Results

Three cavities were mid-T treated until now: 1DE19 for 4.5h@335 °C, 1AC2 (a new baseline was established after the Zanon process) 3.25h@325 °C and 1RI2 for 20h@250 °C. The 20 hours run marks the beginning of a study about the impact of the diffusion length and the niobium oxide layers. 1DE19 had caps on both flanges, while the others only had one cap on the top flange in order to prevent particles from falling into the cavity volume. The characteristic signs of mid-T treated cavities were observed and can be found in Fig. 4: Enhanced quality factors at 2 K caused by reduced R_{BCS}, lower accelerating gradients and at least for the first two cavities also a lower R_{res}.

Flux Trapping Sensitivity

Most cavities showed a significant sensitivity for flux trapping after mid-T treatment: The Q_0 after the first breakdown ("quench") at E_{max} dropped, which could be cured by a warm-up to room temperature. For all the subsequent Q(E)-curves, the measurements were stopped at about 25 MV/m on purpose, avoiding a quench with its drop of Q_0.

This observation is in good agreement with the results of other studies [1, 3]. At the moment a sophisticated B-mapping system is being implemented at the DESY vertical test infrastructure [9] and further analyses of this topic will follow, including a thorough check of the magnetic hygiene. In addition, those studies shall reveal why the sensitivity differs for the different cavities and/or recipes for mid-T treatments.

Witness Sample Studies

The DESY treatments were accompanied by niobium samples, which received an EP directly before the furnace runs. Scanning electron microscopy analysis showed no peculiarities and the absence of carbides. Normalised secondary ion mass spectrometry (SIMS) profiles are shown in Fig. 3. The profile for the sample with 1DE19 is very similar to the one shown in Fig. 3(a). Interesting is the high amount of oxygen on the sample witnessing the 20h@250 °C run in Fig. 3(b), which can not be observed for the shorter mid-T runs. It seems that the oxygen has diffused less deep into the bulk than for the shorter and warmer treatments since the other samples do not show the apparent end of an oxygen profile

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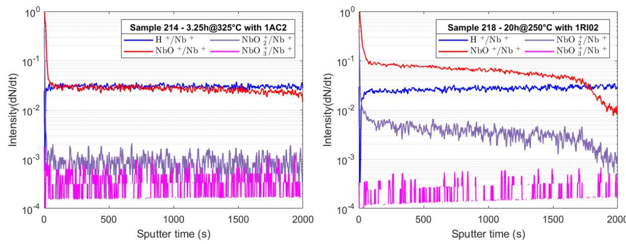

(a) (b)

Figure 3: Normalised SIMS profiles for witness samples of (a) 1AC2 (3.25h@325 °C) and (b) 1RI2 (20h@250 °C). Shown are intensity vs. sputter time.

in the analysed region. Rough estimations of the diffusion lengths support these observations and a more sophisticated modelling will follow in the future.

SUMMARY OF MID-T CAMPAIGN

In Fig. 4, the Q(E)-curves measured at 2 K for all treatments are shown. The filled symbols give the final status of the respective vertical tests including the maximum gradient. The additional curves with empty markers were taken in order to explore the maximum Q_0 and hence stopped before the cavities quenched.

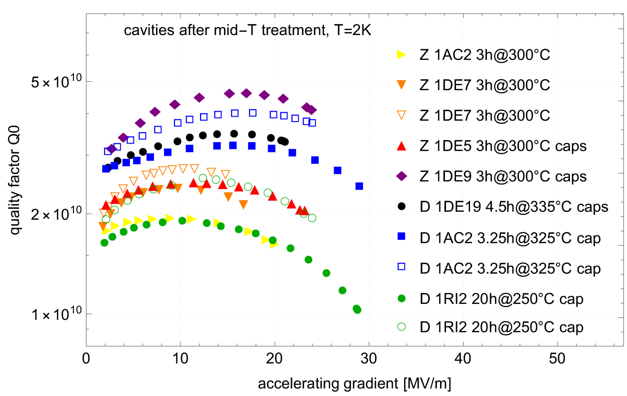

Figure 4: Q(E) for all mid-T treatments. "Z" stands for treatments at Zanon, while "D" abbreviates DESY. Temperature and duration of the treatments are given and whether the cavity was closed by caps. Empty markers depict curves, which were stopped before reaching the quench.

Using Q(E)-curves taken at 2 K and 1.5 K and assuming that the temperature dependent part of the surface resistance is negligible at 1.5 K, a measure which can be assumed to be close to the R_{BCS} at 2 K can be deduced and is shown in Fig. 5. At medium gradients of about 16 MV/m the estimated R_{BCS} of $3-5$ nΩ is significantly lower after all mid-T treatments compared to the baseline test after EP (9 – 12 nΩ). Moreover, a characteristic concave curvature can be observed which causes the anti-Q-slope. Both hold also true for mid-T treatments which turned out to be less successful in measures of E_{acc} and Q_0 at 2 K, like e.g. "Z 1AC2" or "D 1RI2". Very similar performance characteristics have been observed in [3] after mid-T treatments, but also for N-doped cavities [10], indicating that the impurities N and O have a comparable effect on the cavity performance.

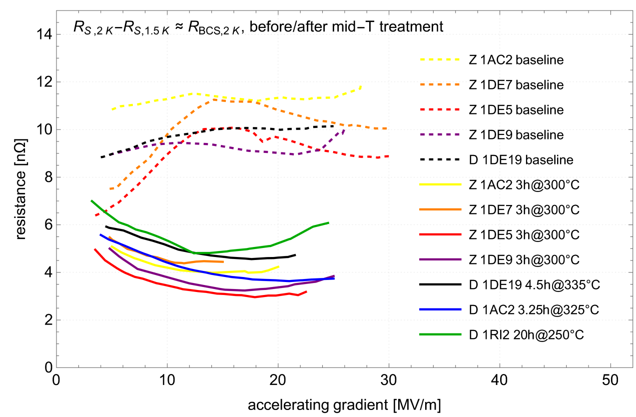

Figure 5: R_{BCS} estimation for all mid-T treatments and corresponding baseline measurements (dashed). "Z" stands for treatments at Zanon, while "D" abbreviates DESY. Temperature and duration of the treatments are given.

CONCLUSION & OUTLOOK

The easy applicable mid-T treatments as chosen path towards cavities with high quality factors seem to be promising. First steps in direction of maintaining large accelerating gradients are made. Clean furnace conditions and the usage of caps seem to be mandatory, while additional HPR cycles do not yield better performance results for the cavities presented here. All cavities show characteristic Q(E)-curves and a reduced R_{BCS}, most of them with large Q_0 and two with gradients of about 30 MV/m. In addition, an enhanced flux trapping sensitivity was observed for most of the mid-T treated cavities.

SIMS studies show significant differences of oxygen concentrations for the different treatment recipes, detailed analyses of this effect are ongoing.

Future studies will aim towards an optimised recipe in terms of temperature and duration of the treatment. In addition, the flux trapping behaviour will be analysed in detail, as well as the magnetic hygiene and influence of the grain structure of the niobium. The frequency behaviour of mid-T treated cavities around T_c seems to reveal also more information, hence data analyses of frequency versus temperature measurements have been started recently.

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