SUSCEPTIBILITY MEASUREMENTS ON SURFACE TREATED NIOBIUM SAMPLES

B. Steffen*, E.-A. Knabbe, L. Lilje, P. Schmüser, DESY, Hamburg
S. Casalbuoni, J. Kötzler, L. von Sawilski, Institut für Angewandte Physik, Univ. Hamburg

Abstract

AC susceptibility measurements have been carried out on samples of the niobium sheets used for the TESLA cavity production. The surface critical field has been determined for chemically etched and electropolished samples. In particular, the influence of the low-temperature bakeout has been investigated for baking temperatures between 100 °C and 144 °C and baking times between 12 and 96 hours. A definite correlation is observed between the surface critical field in samples and the maximum accelerating gradient obtained in the superconducting cavities: both quantities increase when a BCP-treated surface is subjected to electropolishing. An additional increase is observed after the low-temperature bakeout.

INTRODUCTION

In the past few years the surface preparation by electrolytic polishing (EP) has opened the way to extremely high acceleration fields in niobium cavities, more than 40 MV/m in single-cell cavities and 35 MV/m in nine-cell cavities. Electropolished cavities are thus far superior to chemically etched cavities: the nine-cell cavities of the TESLA Test Facility (TTF) linac, which were prepared by buffered chemical polishing (BCP), achieved an average maximum accerating field of 25 ± 2.6 MV/m in the third series of 24 industrially produced cavities. One reason for the better performance of EP-treated cavities is the smoothness of the electropolished surface which prevents magnetic field enhancements at grain boundaries and associated premature breakdown of superconductivity.

A surprising discovery during the CERN-DESY R&D program [1] on the electropolishing of single-cell cavities was the finding that a necessary prerequisite for reaching accelerating fields above 35 MV/m is a low-temperature bakeout of the finished resonator under UHV conditions. This bakeout is applied after the EP-treated cavity has been thoroughly rinsed with ultrapure water and pumped out to a pressure below 10^{-7} mbar. The baking temperature of 100-144 °C is so low that no dissolved gases, not even hydrogen, can diffuse out of the bulk niobium. Hence only a very thin surface layer can be affected by the baking. One idea is that the 5 nm thick niobium pentoxide layer is partly disintegrated and that oxygen penetrates into the underlying niobium, forming suboxides. The physico-chemical processes are not yet fully understood.

Susceptibility measurements in an oscillating magnetic field are particularly well suited to study the surface properties of a superconductor. In a dc background magnetic field exceeding the upper critical field B_{c2} of the superconductor, only a surface layer with a thickness in the order the coherence length ξ remains superconductive. The ac susceptibility of this layer should then be sensitive to various chemical and electrochemical treatments and to the temperature and duration of the UHV bakeout. In the present experiment small cylindrical samples of the niobium used for the TTF cavity production were subjected to the same BCP or EP treatments and the same bakeout as the cavities. The ac susceptibility of these samples was measured at 4.2 Kelvin and a frequency of 10 Hz of the ac magnetic field. The dc background field could be varied between 0 and 1 Tesla.

EXPERIMENTAL SETUP

The complex ac susceptibility $\chi = \chi' - i\chi''$ is measured by the mutual inductance technique. The sample is placed in the center of a set of concentric coils (Fig. 1). The outer coil produces a dc magnetic field $0 \leq B_0 \leq 1$ T, the middle coil applies a small ac field of $5\,\mu T$. Both fields are oriented parallel to the axis of the cylindrical sample. The pickup system consists of a measurement coil and a compensation coil of opposite winding direction. Without sample the sum of the induced voltages vanishes. When the sample is put into the measurement coil a complex voltage U is induced which is related to the complex ac susceptibility

$$\chi = \chi' - i\chi'' \propto -iU = -U'' - iU' \quad .$$

The induction signal is recorded with a lock-in amplifier. The dc susceptibility of the sample corresponds to the low frequency limit of χ', whereas χ'' is a measure of the losses in the sample due to the oscillating field. The detection system works from 1 Hz to 1 kHz in the temperature range 1.5 K to 4.2 K (pumped He cryostat). The data reported here have been taken at 10 Hz and 4.2 K. For a more comprehensive description of the setup see [2].

SAMPLE PREPARATION

The samples are cylinders of 2.6 mm diameter and 2.6 mm height. They were electro-eroded from remainders of the niobium sheets used for cavity production. After the erosion the samples were chemically etched (BCP process) by about 50 μm, annealed at 800 °C and again chemically

* bernd.steffen@desy.de

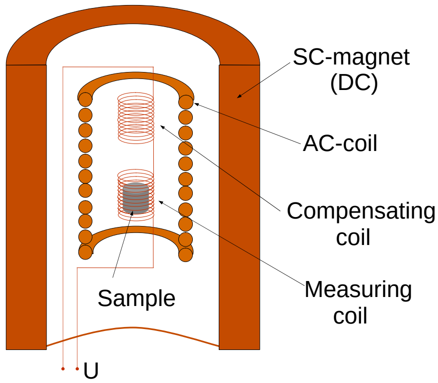

Figure 1: Schematic drawing of the experimental setup.

etched by about 50 μm. After each etching step the samples were rinsed thoroughly in de-ionized water and dried in a clean room. A first series of ac susceptibility measurents was performed on these BCP-treated samples. In the next step the samples were baked in a vacuum of better than 10^{-7} mbar at $100 \pm 1\,°$C, $123 \pm 1\,°$C and $144 \pm 1\,°$C for times between 12 and 96 hours. The second series of measurements was carried out on the BCP-treated and baked samples. Some baked samples were slightly BCP-treated and measured to study the thickness od the surface layer influenced by baking. Another set of samples were electropolished after the second 50 μm BCP treatment. Since the circular end faces were needed to hold the cylinders in the EP-apparatus and to apply the voltage only the cylindrical mantle could be polished[1]. The material removal by EP was between 40 μm and 165 μm. The fourth series of measurents concerned the unbaked EP-treated samples. Finally the bakeout was applied and the baked EP-samples were investigated in the fifth series of measurements.

RESULTS AND DISCUSSION

Chemically Etched Samples

The complex ac susceptibility of BCP-treated samples is plotted in (Fig. 2) as a function of the dc background field[2]. First we discuss the behaviour of the unbaked sample. For $B_0 < 280$ mT one observes perfect diamagnetism: the real part of the susceptibility is $\chi' = -1$, the imaginary part χ'' vanishes. The first deviation from perfect diamagnetism occurs above the upper critical field $B_{c2} = 280$ mT of the bulk niobium. Here the inner part of the sample goes to the normal state and only a thin surface layer remains superconducting. This layer shields the sample volume from the ac magnetic field. With rising dc field the critical shielding current of the superconducting layer decreases and is no longer capable of shielding the ac field completely. Hence χ' rises steeply and reaches 0 (no shielding) at the surface critical field B_{c3}. The steep rise of χ' is accompanied by a maximum in χ''. The corresponding energy dissipation is caused by the magnetic flux moving out of the superconducting surface layer.

A baking at 100°C for 2 days has almost no influence on the complex susceptibility. Higher baking temperatures, however, shift the steep rise of χ' and the maximum in χ'' to considerably higher fields.

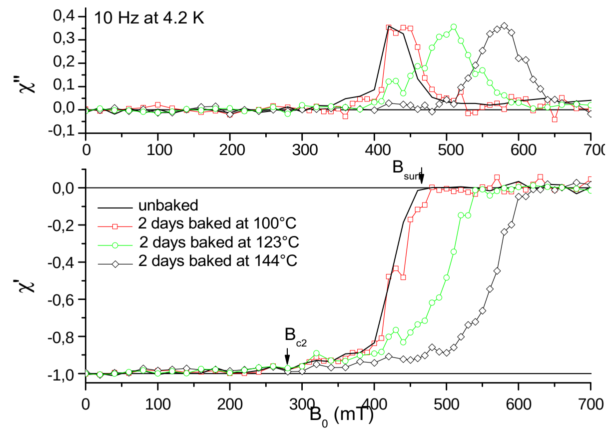

Figure 2: Imaginary (χ'') and real part (χ') of the ac susceptibility of an unbaked sample and three samples baked for 48h at different temperatures. The values B_{c2} of the bulk material and B_{surf} are indicated for the unbaked sample.

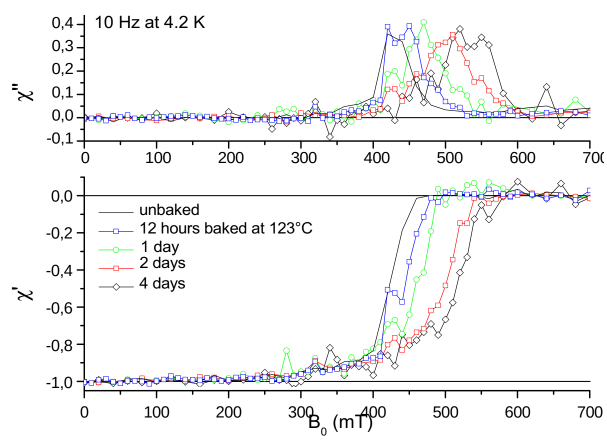

Figure 3: The ac susceptibility of one unbaked BCP-sample and of four BCP-samples baked at 123 °C for times between 12 and 96 hours.

The influence of the baking time is demonstrated in Fig. 3. A clear tendency is seen that the rise in χ' shifts to larger fields when the baking time is extended from 12 to 24 and to 48 hours.

If we make the assumption that the increase in the surface critical field beyond the Ginsburg-Landau value

[1] The unpolished (but BCP-treated) end faces are perpendicular to the applied dc and ac magnetic fields and do not contribute to the measured ac susceptibility.

[2] The data in this and the following figures have been normalized to $\chi = -1$ at $B_0 = 0$ to correct for the demagnetization factor of 1.6 of the cylindrical sample.

($B_{c3} = 1.69 B_{c2}$) is due to diffusion of oxygen from the Nb_2O_5 layer into the niobium, as proposed by several authors [3, 4, 5, 6] as an explanation of the baking effect in EP-cavities, one can refer the baking time t_n at a given temperature T_n to an equivalent baking time t_ref at a reference temperature T_ref. The temperature dependence of the diffusion coefficient D for oyxgen in niobium is given by:

$$D \propto \exp\left(-\frac{A}{RT}\right)$$

with $A = 111530 \,\frac{\mathrm{J}}{\mathrm{mol}}$, T the temperature in Kelvin and R the universal gas constant. From this formula one gets a factor of 8 to normalize the baking time at 100 °C to a reference temperature of 123 °C and a factor of 0.18 for the baking time at 144 °C. The critical surface field B_surf and the field B'' where χ'' assumes its maximum are plotted in figure 4 as functions of the equivalent 123 °C baking time. The surface critical field seems to saturate after baking of more than 32 hours at 144 °C, corresponding to 180 hours at 123 °C. The data obtained at different baking temperatures join smoothly in this plot. This supports the hypothesis that oxygen diffusion from the oxide layer into the superconducting surface layer is responsible for the increase of the surface critical field due to the baking procedure. In Fig. 5 we show that a chemical etching of the baked samples by just a few micrometers is sufficient to restore the results of the unbaked samples. This is further evidence that the low-temperature bakeout affects only a very thin surface layer.

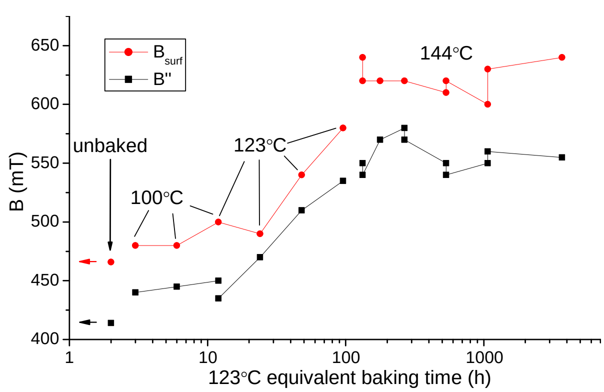

Figure 4: The surface critical field B_surf and the field B'' where χ'' assumes its maximum, plotted against the equivalent baking time at the reference temperature of 123 °C.

Electropolished Samples

Figure 6 shows the influence of electropolishing on samples which had been previously treated by BCP. Removing a surface layer of 40 μm by EP shifts the steep rise of χ' and B_surf to slightly higher field values as compared to chemically etched samples. The effect becomes more pronounced after the removal of 80 μm or more. Baking of the electropolished samples leads to an additional shift to higher fields (see Fig. 7).

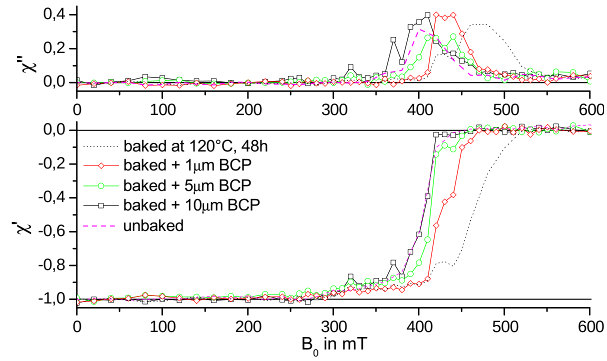

Figure 5: The ac susceptibility of baked BCP-samples after additional chemical etching.

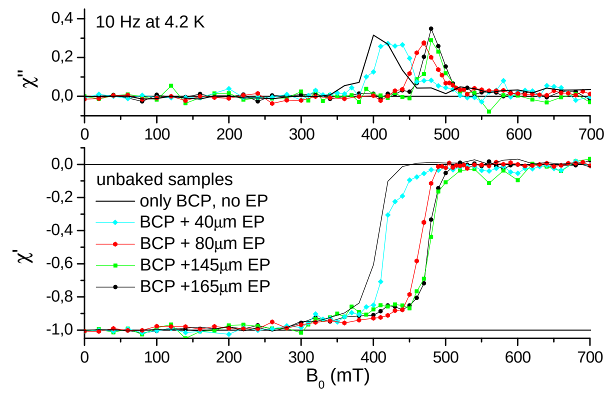

Figure 6: The real and imaginary parts of the ac susceptibility of electropolished samples in comparison with an unbaked BCP-sample.

CONCLUSION

We have shown that the low-temperature baking raises the surface critical field B_surf. The effect is hardly observable at 100°C but becomes pronounced at higher baking temperature. Chemically etched samples reach their maximum B_surf after 32 hours baking at 144 °C; B_surf is about

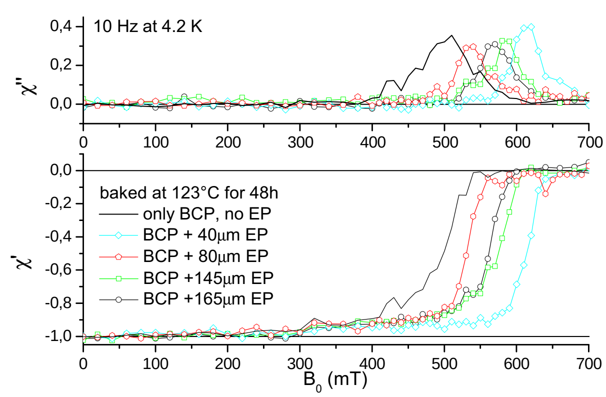

Figure 7: The ac susceptibility of electropolished samples after baking at 123 °C for 48 hours.

20% higher than the B_{c3} of unbaked samples. Baking times beyond 48 hours did not increase B_{surf} any further. Electropolishing raises the surface critical field by about 8% as compared to chemically etched (BCP) samples. A further increase by up to 20% is caused by the baking procedure.

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