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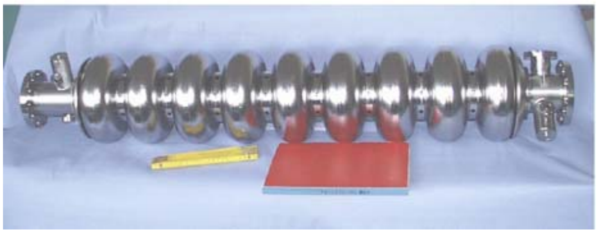

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Conclude on comparison of SQUID scanner vs. Flux gate detector

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Abstract

An electromagnetic non invasive and contact-less technique using a Flux-Gate 1st order electrical gradiometer has been investigated to detect the magnetic field distribution during electropolishing of Copper and Niobium cavities. Local information of the dissolved metal surface during the electropolishing process has been obtained. The electropolishing of the copper surface employed in superconducting RF cavities has been monitored using magnetic field sensors. An electromagnetic inversion of the magnetic field imaging has been implemented in order to better understand the effect of the cathode geometry on the electropolishing process. An electrolytic cell has the possibility to test different cathode shapes. By a tomography of the electrolytic cell section it will be seen that a re-entrant cathode is definitely beneficial for electropolishing uniformity. In addition to that the same flux gate gradiometer has been used for distinguishing Niobium with different RRR Values

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INTRODUCTION

In order to obtain the required performance in a superconducting RF cavity, such as high acceleration gradients with low power losses, an extremely high quality Nb superconducting surface is required.

The Nb surface quality plays an important role in the RF cavity performance. In fact, some limitations could be caused by defects or imperfections on the Nb surface. The latter could produce thermal instability due to a niobium particle with bad thermal contact to the copper surface or a Niobium tip, where the field enhancement leads to a breakdown of superconductivity. Moreover, field emissions could take place because the high surface electric fields lead to electron emission from scratches or particles located on the surface via electron tunneling. This process dissipates power and therefore can cause an exponential decrease of the quality factor with the accelerating field.

For this reason the quality control of the Niobium surface requires electropolished surfaces. A possible Electropolishing technique foresees that the electrolytic cell is driven in current regime. An alternative approach requires a voltage driven operation, on the basis of the I-V characteristics of the electrochemical cell. This method however does not give spatial information on the electropolishing surface. To overcome this limitation, an electromagnetic technique based on magnetic sensors could be considered. The advantage due to the application of the magnetic field monitoring compared to the current-voltage technique is the possibility to obtain local information, in a non invasive way, of the ongoing polishing process over the cavity wall surface even when its shape could be very complex.

In other words, while the standard control of current and voltage during the Electropolishing gives an integral measurement of the electrolytic process, the magnetometric approach can give a point by point a local measurement of the magnetic field associated to Electropolishing phenomenon just in the point detected.

In order to achieve the ongoing monitoring of the quality control of the niobium surface, it is necessary to choose the most suitable sensor with respect to its magnetic field sensitivity, spatial resolution and frequency bandwidth. Examples of magnetic sensors that can be applied in electromagnetic inspections are Flux Gate, Hall probe, GMR (Giant-Magneto Resistance), and low and high temperature SQUIDs (Superconducting Quantum Interference Device) that are the most sensitive but also the most expensive sensors. Instead Flux-Gate Magnetometers work at room temperature and are cheaper than SQUIDs; have a higher magnetic field sensitivity (10 pT/√Hz at 1Hz) than the Hall probes and the GMR. The latter is an emerging magnetic sensor that potentially could be more sensitive than Flux-Gate [1], characterized by a sensitivity less than 1 μT, a good spatial resolution less than 100 μm, and a wide frequency bandwidth (300-400 kHz).

Magnetometry can be a suitable and useful diagnostic technique to perform the quality control of the materials employed in the RF cavities fabrication. In this paper the electromagnetic techniques based on magnetic sensors used for the quality control of RF cavities are reported. Static and dynamic measurements of the in plane magnetic field component during the electropolishing of both niobium and copper, using a Flux-Gate electronic gradiometer, have been carried out. Moreover, an electromagnetic inversion of the magnetic field distribution based on Fast Fourier Transform (FFT) has been performed to calculate the current density distribution due to the electropolishing process. The knowledge of the current density distribution allows to obtain information about the electrochemical activity with respect to the cathode geometry.

EXPERIMENTAL METHODS

In this work the electropolishing of niobium surface with different geometries has been monitored. The electrolytic cells are shown in figure 1.

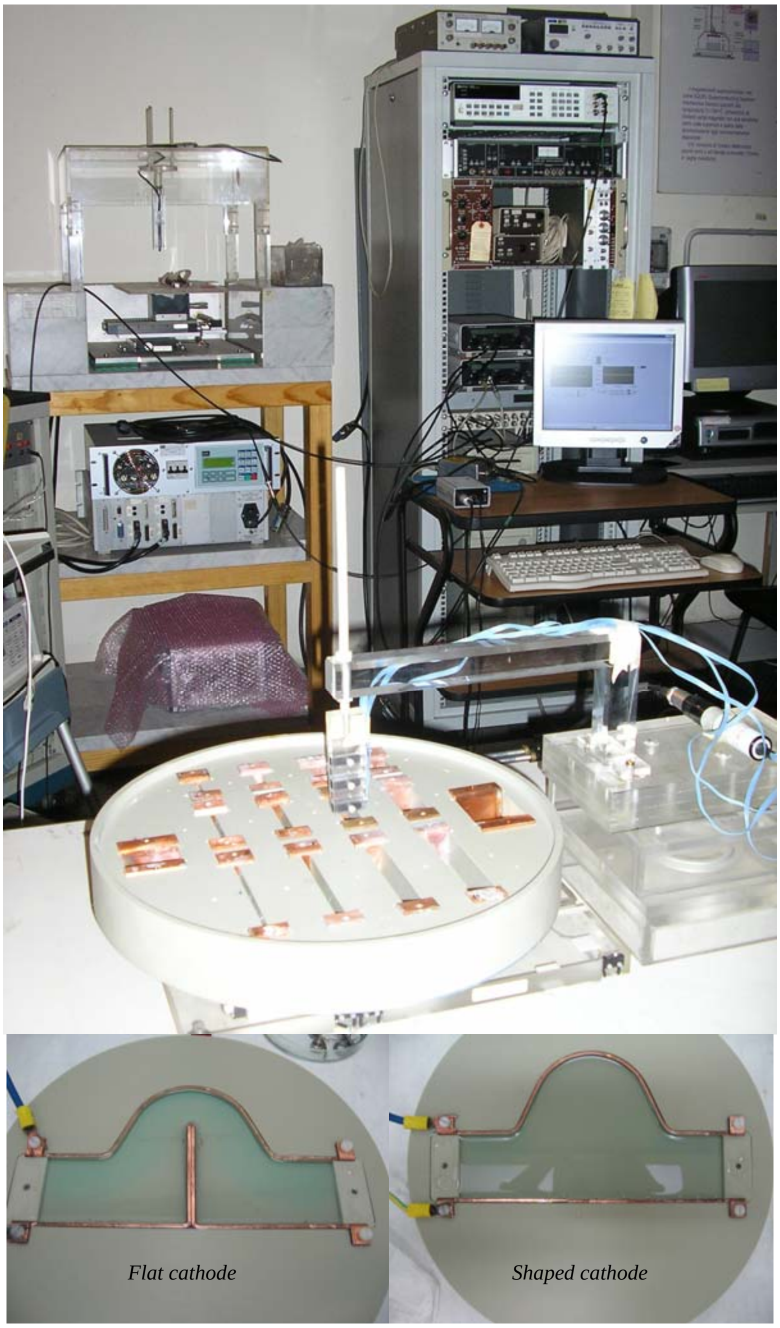

Figure 1 : The electrolytic cells monitored: above are the rectangular cells, below a cavity section with flat and shaped cathode, respectively.

Cells with rectangular electrodes in figure 1 of different width: 8 mm, 16 mm and 24 mm, and cells characterized by iris-like anode and flat or shaped cathodes have been tested. Both Niobium and Copper have been tested. The electrolytic solution was 55% Phosphoric acid plus 45% n-buthanol for Copper and Hydrofluoric acid plus sulphuric acid for Niobium. During the electropolishing process that was operated in constant voltage mode, the Flux-

Gate 1st order electronic gradiometer measured the in-plane component of the magnetic field gradient, G_x, due to the current distribution between the two electrodes.

A magnetic imaging of the ongoing electropolishing process has been carried out moving the sensors over the cell area (within the electrodes), with a speed of 3mm/s and a continuous acquisition mode with 6 data points/mm. In both cell geometries (rectangular and cavity shape) the magnetic field imaging is represented by a matrix in which the columns are the line scans obtained moving the Flux-Gate 1st order electronic gradiometer from the anode to the cathode. To monitor the electropolishing process of niobium surface the Flux-Gate sensors have been applied because of their capability to detect the magnetic field produced in the electropolishing process. The Flux-Gate sensor is a solid state device based on the non linearity of the magnetic characteristic of its sensing ferromagnetic core. It can measure the d.c or the low frequency a.c. magnetic field component, with a field sensitivity ranging from 10^{-11} to 10^{-4} Tesla. A flux-Gate sensor is made of a high permeability cylindrical core around which there are two coaxial coils: bias coil and sensing coil. This sensor detects directly the variation of the magnetic field generally using a Phase Sensitive Detection (PSD), in this way it can work in a bandwidth ranging from d.c. to 5 kHz.

FLUX GATE MAGNETOMETRY APPLIED TO ELECTROPOLISHING

It was already demonstrated [2] that it is possible to control the electrolytic polishing of metal, following the magnetic polarization curve. The experimental measurements were carried out driving the cell in voltage and positioning the Flux-Gate sensors above the electrodes (cathode or anode).

Increasing the current value, the magnetic field versus the voltage is monitored. In this way the H-V polarization curve can be obtained.

Comparing the H-V and I-V polarization curves (Figure 2) it is possible to identify the three different regions that characterize the process: pitting (below the plateau), polishing (at the plateau) and the gas evolution (above the plateau).

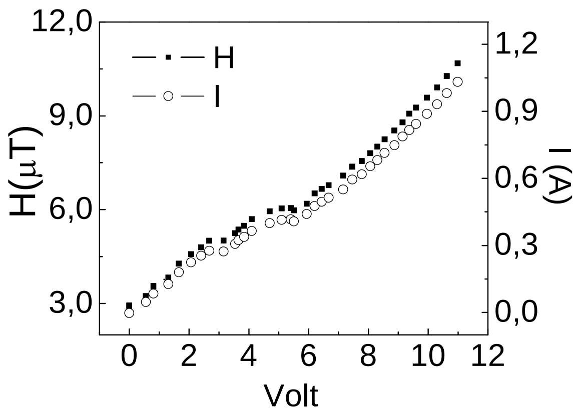

Figure 2 : H-V and I-V polarization curve.

As it can be seen, if renormalized, the Electropolishing I-V Characteristics and the magnetic characteristics with contactless sensors have exactly the same behaviour. The advantage of magnetic measurements is due to the possibility to control the electropolishing process outside of the bath, without intrusive probes, independently of the shape and dimension of the cell surface. Moreover, by monitoring the magnetic field, one can know the exact local I-V characteristics in a determined point.

During the electrolytic process there is a flow of ions from the anode to the cathode that produce a corresponding magnetic field, which can be monitored by the Flux-Gate sensors. Using the magnetic field produced by the ions a very simple model can be applied to evaluate the anode oxidation. If every ion is approximated by a charge **q** with a speed **v,** the produced magnetic field at distance **r** can be represented as follow:

$$\vec{B} = \frac{\mu_0}{4\pi} q \frac{\vec{v} \times \vec{r}}{\vec{r}^3} \tag{1}$$

where μ_0 ($4\pi \times 10^{-7}$ Tm/A) is the permeability of the free space. Since the Flux-Gate sensors measure the in-plane component of the field B_x, produced by the current j_y orthogonal to the electrode, the module of magnetic field becomes:

$$B_x = \frac{\mu_0}{4\pi} q \frac{v}{r^2} \tag{2}$$

the speed **v** can be obtained considering that the current density **j** is **j = Nqv**, where **N** is the number of ions per volume **N=n/V** (m^{-3}). Then the magnetic field can be written as:

$$B_x = \frac{\mu_0}{4\pi} \frac{jSl}{nr^2} \tag{3}$$

From this equation it is possible to obtain the number of ions **n,** that start from the anode and go into the solution:

$$n = \frac{\mu_0}{4\pi} \frac{jSl}{B_x r^2} \tag{4}$$

Using the Faraday law,

$$w = \frac{jStM}{nF} \tag{5}$$

where **w** is the dissolved copper at the anode, ***S*** is the cross section of the cell, ***t*** is the time during the measurement, **M (**63.456 g/mole) is the copper atomic mass and **F (**96500 As/mole) is the Faraday constant. Considering the equation (4) the Faraday law can be written as

$$w = \frac{4\pi B_x M r^2 t}{\mu_0 l F} \tag{6}$$

which can be used to calculate the etching rate **w/t**.

This simplified model has been checked to estimate the dissolved metal from the anode, for copper cells with length of 50 mm and different width (8 mm, 16 mm, 24 mm) using a static measurement of the magnetic field across the anode.

Table 1: etching rate and dissolved copper at 4V

Cell width [mm]	**w**[g] at 4V by balance	**w**[g] at 4V by B_x
8	0,012±10^{-4}	0,011±7E-3

16	0,023±10^{-4}	0,020±7E-3
24	0,05±10^{-4}	0,032±7E-3

Table 2: etching rate and dissolved copper at 7V

Cell width (mm)	**w**[g] at 7V by balance	**w**[g] at 7V by B_x
8	0,025±10^{-4}	0,048±7E-3
16	0,035±10^{-4}	0,020±7E-3
24	0,13±10^{-4}	0,035±7E-3

The data reported in table 1 and 2 show the dissolved copper (w) at the anode in 5 minutes for the two cases at 4V and at 7V, respectively.

The data calculated using the simple model previously described have been compared with the results obtained weighing the electrode after the magnetic measurement by means of a balance with a sensitivity of 10^{-4}g. It could be noted that in the plateau (4V) the difference between the results of the two different techniques are quite similar, the difference is less than 2%. For a voltage of 7V, instead, the compared data show a difference higher than 2%, probably because of the more chaotic etching process due to the gas evaporation. In this case the in-plane component of the magnetic field is not capable to describe quantitatively the copper dissolution.

ELECTROMAGNETIC INVERSION OF MAGNETIC FIELD IMAGING

Dynamic measurements, moving the magnetic sensors above the cells, monitores the magnetic field distribution due to the flow of current in the solution. Figure 3 shows the magnetic imaging of the rectangular cell, at the potential of 4V.

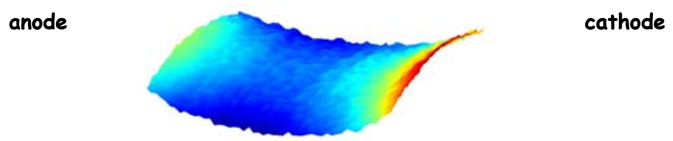

Figure 3: magnetic field distribution of the rectangular cells at the potential of 4 V.

It is not very simple to extrapolate information about the etching process from the magnetic field distribution. For this reason it is necessary to invert the magnetic field distribution to represent the corresponding current distribution which reflects the electrochemical activity in the cell.

In general, the inverse magnetic problem does not have an unique solution but in the case of two dimensions it can be solved uniquely. To obtain the current density distribution during electropolishing of the rectangular cells, the mathematical technique based on the Fast Fourier Transform (FFT) has been applied. The magnetic inverse technique applied in this work to the electropolishing process of rectangular cells has been used successfully in other applications and it is described in detail [3] by B. J. Roth et al.

To calculate the current density from the magnetic field data the rectangular cell has been approximated by a finite short dipole, which generates a magnetic field expressed by Biot-Savart law:

$$B(r) = \frac{\mu_0}{4\pi} \int \frac{J(r') \times (r - r')}{|r - r'|^3} d^3 r'$$

where *J* is the current density that produce the field and *r* the distance where the magnetic field is measured. The configuration of the Flux-Gate sensors allows to measure only the in-plane component of the magnetic field, in this case B_y , so the previous expression becomes:

$$B_y(x, y, z) = \frac{\mu_0}{4\pi} l \cdot z \cdot \iint \frac{J_x(x', y')}{\left[(x - x')^2 + z^2\right]^{3/2}} dx' dy' \qquad (8)$$

In the formula above *l* and *z* are the length of the cell and the distance between the probe and current source, respectively. It should be noted that measuring the y component of the magnetic field, B_y, it is possible to obtain only the corresponding *x* component of the current density, J_x. Moreover, the equation (8) represents the convolution between the current density *J* and Green function *G*, expressed by:

$$G(x - x', y - y', z) = \frac{\mu_0}{4\pi} l \cdot z \cdot \frac{1}{\left[(x - x')^2 + z^2\right]^{3/2}} \quad (9)$$

By using the convolution theorem it is possible to rewrite the equation (8) in the Fourier space as:

$$b_y(k_x, k_y, z) = g(k_x, k_y, z) \cdot j_x(k_x, k_y)$$

where the $b_x(k_x,k_y,z)$, $j_x(k_x,k_y)$ and $g(k_x,k_y,z)$ are the two dimensional Fourier transforms of the magnetic field , the current density and the Green's function, respectively. The variables k_x and k_y are the components of the spatial frequency K. Then the current density in the Fourier space is given dividing the magnetic field by the Green's function:

$$j_x(k_x, k_y) = \frac{b_y(k_x, k_y, z)}{g(k_x, k_y, z)}$$

Finally, the current density distribution J_x is given by the inverse Fourier transform of j_x.

In figure 4 the imaging of the current distributions obtained by applying the FFT technique to the corresponding magnetic field data (figure 3) is shown. It can be seen that the current distribution is uniform along the total length of the cell, because the plateau of the polarization curve (at 4V) guarantees an uniform etching.

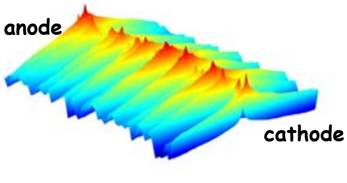

Figure 4: the current distribution for the rectangular cells at the potential of 4 V.

Starting from these results for a very simple cell geometry other electrode configurations very similar to the shape of the RF superconducting cavities have been analyzed.

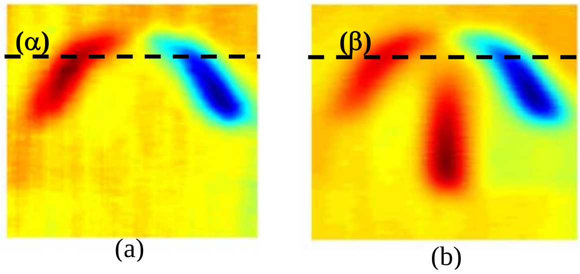

Figure 5: Cell Magnetic field distribution with different cathode shapes: flat (a) and curve (b).

Considering the two different cathode geometries, flat and shaped (figure 1), a magnetic imaging of the cells has been carried out. The magnetic field maps are shown in figure 5. The magnetic field images distinguish successfully the different electropolishing activity due to the different cathode shape. Moreover, further information about the efficiency of the electropolishing process, due to the two cathode geometries, can be obtained considering the current distribution. Applying the FFT technique, using as Green's function the equation:

$$G(x-x',y-y',z)=\frac{\mu_0}{4\pi}l\cdot z\cdot\left[\frac{1}{\left[(x+x')^2+z^2\right]^{3/2}}-\frac{1}{\left[(x-x')^2+z^2\right]^{3/2}}\right]$$

the current distribution across the anodes has been calculated. In figure 6 the comparison between the current distribution, related to the lines scan in the magnetic field imaging of figure 5, is reported.

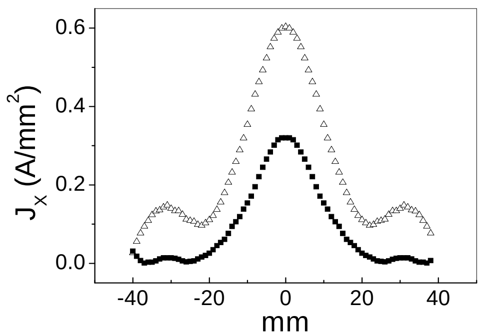

Figure 6: Comparison of the corresponding current density for lines scan extracted from the magnetic maps of fig 4. (squares) flat cathode and (triangles) shaped cathode.

The experimental results demonstrate that the current intensity in the case of a shaped cathode is higher than the corresponding current in the cell with a flat cathode. In other words the shaped cathode assures more current across the anode curvature and as a consequence a more efficient electropolishing of the surface is obtained. This result is confirmed by the visual inspection of the cells. In figure 7 the pictures of the cells with the two cathode geometries are shown.

These pictures are related to the voltage of 4V, where the etching process is uniform. The dot lines confine the viscous layer (the blue area across the anode) that enables the uniform electropolishing of the wall surface. As can be seen in the case of flat cathode the viscous layer is very thin at the equator, while using the shaped cathode the viscous layer is uniform along the whole anode surface.

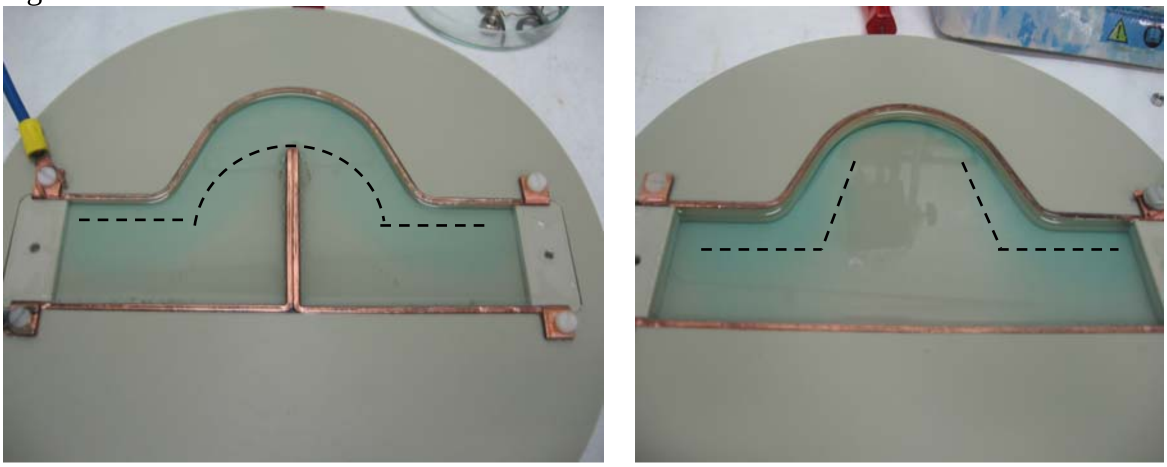

Figure 7: The electrolytic cells analysed: a cavity like electrode with flat and shaped cathode, respectively. The dot lines follow the viscous layer due to uniform electropolishing process.

EVALUATION OF NIOBIUM RESISTIVITY BY PULSED EDDY CURRENT TECHNIQUE

Since it is very important to use a high purity Niobium surface to guarantee high performances of the rf superconducting cavities, a quick and simple method to detect the purity of the Niobium could be done by means of magnetometry. The resistivity at room temperature of a Niobium sample can be measured in a contactless and not-invasive method applying the Pulsed Eddy Current (PEC) technique.

In figure 8 the comparison of the signals detected by the Hall sensor when there is a Niobium target between the probe and the pulsed source (straight line) and in air, without any sample (dot line), are reported.

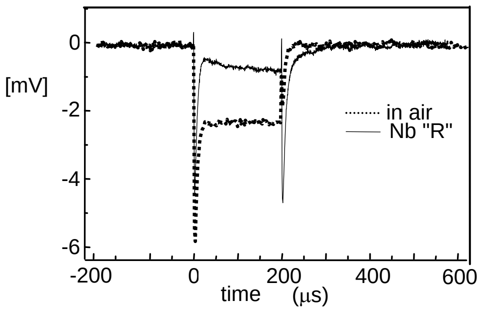

Figure 8 : A magnetic pulse signal detected by the Hall probe in air (dot line) and in presence of a Niobium sheet (straight line).

Table 3: Niobium resistivity for different specimens

Niobium sample	RRR	Δt (μs)	Measured Conductivity $(\Omega\ m)^{-1}$	Measured Resistivity (Ω m)
Reactor Grade	50	251	0.125·108	8·10 -8

Wa Chang	230	267	0.133·108	7.5 ·10 -8
Tokio Denkai	250	271	0.135·108	7.4·10 -8

It can be seen that the magnetic response is sensitive to the presence of the Niobium sample that produces a reduction of the pulsed source signal.
The same PEC technique has been used to detect the magnetic field signals produced by Niobium disks with different resistivity. In figure 9 the magnetic field signals of three different Niobium specimens measured by the Hall magnetometer are shown. Since the conductivity and the thickness of the Niobium target is correlated with the delay time Δt of the transmitted electromagnetic pulse, the electrical conductivity values of the test samples have been estimated and they are summarized in table 3.

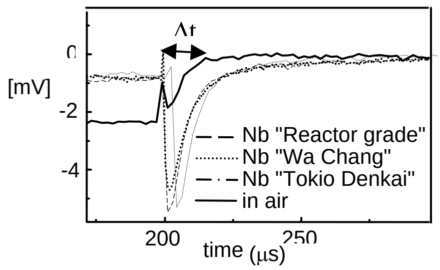

Figure 9: A magnetic pulse signal detected by the Hall probe in air and in presence of three different Niobium test samples.

The agreement between experimental and the nominal values of the resistivity is 1%. These results demonstrate that the PEC method based on Hall probe is a suitable technique to evaluate at room temperature the Niobium electrical conductivity.

CONCLUSIONS

The ongoing etching during the electropolishing of metal surfaces by static and dynamic measurements using a first order Flux-Gate electronic gradiometer has been monitored. An estimate of the metal dissolution can be obtained using a model based on the Faraday law and using the magnetic field measurement. Moreover, a magnetic inversion algorithm on a 2D cavity mock-up has been carried out successfully to obtain the current distribution on the copper surface. This result has demonstrated that the shaped cathode allows a more uniform electropolishing process of the RF superconducting cavities. Moreover, the results demonstrate that the suggested technique is useful to improve the quality process of the electropolishing of copper surfaces for the fabrication of RF superconductive cavities.
In addition we have verified that Magnetometry is a valuable room temperature technique for distinguishing Niobium with different RRR values

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