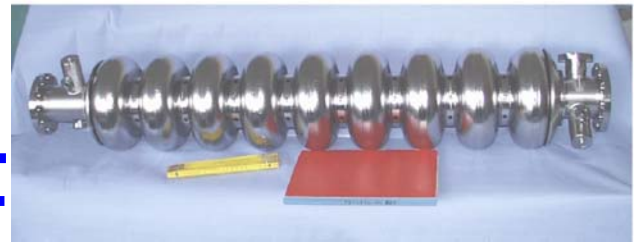
SRF



Deliverable 6.1.5.4:

Final Report on SQUID Scanning

W. Singer, A. Brinkmann

Abstract

Abstract: A SQUID system for eddy current testing of niobium sheets used to fabricate superconducting resonators for particle accelerators is developed at Fa. WSK in collaboration with DESY. Since the fabrication of superconducting resonators from planar niobium sheets is very costly, a measurement procedure is required which can test the niobium sheets before the resonator is made. Our system can detect relevant surface damage or inclusions of foreign material having a volume of as small as 10^{-12} m^3 in a test sample made from high-purity niobium. Due to the relatively high frequency of the eddy currents of up to 100 kHz, the system - although employing a magnetometer - can be operated in an unshielded environment. The SQUID readout uses 4 MHz ac flux modulation; a peak-to-peak dynamic range of ten flux quanta is obtained at 200 kHz.

Acknowledgement

We acknowledge the support of the European Community-Research Infrastructure Activity under the FP6 "Structuring the European Research Area" programme (CARE, contract number RII3-CT-2003-506395).

I. INTRODUCTION

The maximum obtainable electric field strength of superconducting microwave cavities is limited by inclusions of foreign materials, such as tantalum. For the planned superconducting accelerators XFEL and ILC, a few small tantalum inclusions per resonator could already lead to a substantial reduction in the projected maximum electric field strength of the cavities. Since the fabrication of superconducting resonators from planar niobium sheets is very costly, a measurement procedure is required which can test the niobium sheets before the resonator is made. The company WSK Mess - und Datentechnik GmbH (F. Schölz, A. Farr, J. Reuss, E. Wappler, B. Dickel) supported by experiences and knowledge of Institute of Applied Physics, University of Gießen (M. Mück, C. Welzel) developed in a collaboration with DESY an eddy-current NDE system based on a niobium dc SQUID, with which we could detect tantalum inclusions having a volume of as small as 10^{-12} m^3 in a test sample made from high-purity niobium, as well as small defects at the surface of the niobium sheets.

II. MEASUREMENT PRINCIPLE

We have used eddy-current testing to detect Ta inclusions or surface flaws, but other SQUID-based methods, such as the generation of thermo currents by thermal gradients might also be applicable. Fig. 1 shows the principle of eddy current testing a niobium sheet.

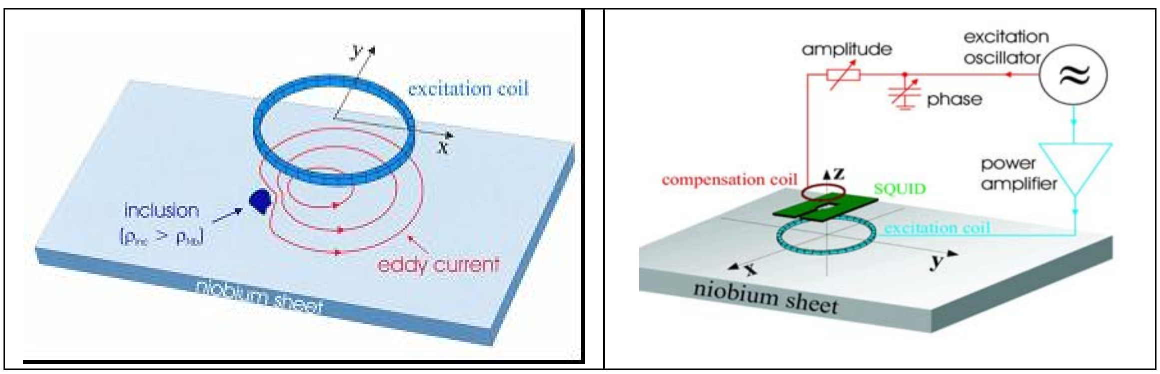


Fig. 1: Principle of eddy current testing on a niobium sheet

A circular coil, usually with a diameter of a few mm generates eddy currents in the niobium sheet. Inhomogeneities, such as cracks or inclusions of materials having a conductivity different from that of niobium lead to a distortion of the eddy current flow, and thus to a change in the eddy current field, which we detect by scanning the sheet with a SQUID. In order to minimize the excitation field at the location of the SQUID, usually a gradiometric excitation coil is used, having the shape of a double D. However, since we expect the Ta inclusions to be only very small, a relatively small double-D coil must be used to maximize the eddy current density at the location of the inclusion. Making small double-D coils with many turns and high symmetry is not easy at all, instead we use an electrical compensation scheme in which the field of the circular excitation coil is compensated electronically at the location of the SQUID by feeding part of the excitation current through the modulation coil used for flux locking the SQUID. By carefully adjusting the amplitude and phase of the compensation current, we can compensate the excitation field at the SQUID by a factor of 1000. An even higher compensation factor is possible, but drift then requires a frequent readjustment of amplitude and phase of the compensation current. Since the obtainable signal-to-noise ratio is directly proportional to the excitation field, the latter should be as large as possible. We use an excitation field of up to 1 mT peak-to-peak; the necessary excitation current then is about 2A peak-to-peak. Dissipation in the coil raises its temperature to about 60°C in this case.

We estimate the expected signal from a 100 μm diameter. Ta inclusion to about 30 pT. Although it seems possible to detect this field with a conventional magnetic field sensor, such as a flux gate, the then intolerably long measuring time would make the use of such a sensor impractical. A flux gate with an field noise of 10 pT/√Hz could detect a Ta inclusion with a signal-to-noise ratio SNR ≈ 1 in an integration time of 1 s. Assuming the area covered by the flux gate is 1 mm^2, it would take 300 × 300 s to scan a 30 × 30 cm^2 wide niobium sheet. Not only is this measuring time intolerably long, also the obtainable SNR ≈ 1 is not sufficient to discriminate between noise and real flaws. The desired SNR for the application discussed here is at least 10.

III. MEASUREMENT CONFIGURATION

Since the expected response from a 10^{-12} m^3 size Ta inclusion is very small, high magnetic field sensitivity is required of the sensor measuring the eddy-current field. On the other hand, thermal noise produced by the niobium sheet at room temperature sets a lower limit for the sensitivity to about 50 fT/√Hz. Although this field sensitivity can be achieved by a HTS SQUID, for reasons of reliability we use a niobium dc SQUID in our system.

The SQUID is cooled in a low-noise fibre-glass dewar for biomagnetic measurements which holds one litre of liquid helium for about two days. The stand-off distance between SQUID and sample is about 6 mm. A fast computer controlled x-y translation stage scans the niobium sheet underneath the dewar. The eddy current excitation coil is located in between the dewar and the sample. All important supports are made of granite, to avoid vibrations. A picture of the system is shown in Fig. 2.

The scanner is based on a xy table with ca. 300mm x 300mm travel area. The Nb sheets are fixed by a vacuum sample holder in order to keep them as flat as possible.

The SQUID sensor is electronically controlled by a flux modulation and control loop, in order to keep the magnetic flux through the SQUID constant. Compensation current is controlled by the flux measurement. The amount of compensation current necessary to keep the SQUID's flux constant is then taken as measurement value from the control loop. This signal is then processed by a lock-in amplifier to eliminate noise with a spectral density apart from the excitation frequency. Different filters are implemented into the lock in amplifier to improve the Signal/Noise ratio. The system works in a non-shielded environment.

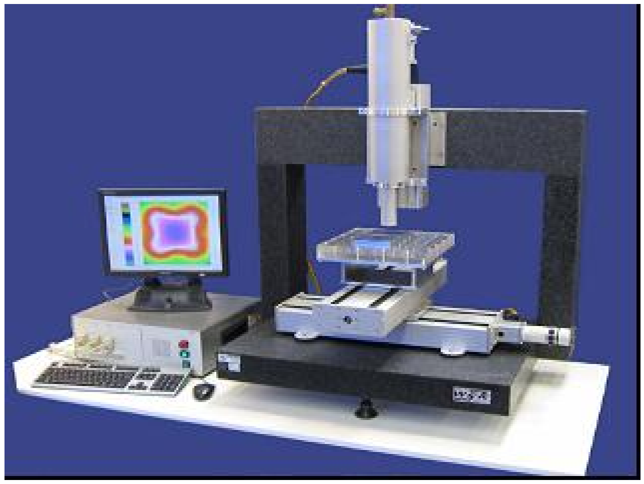
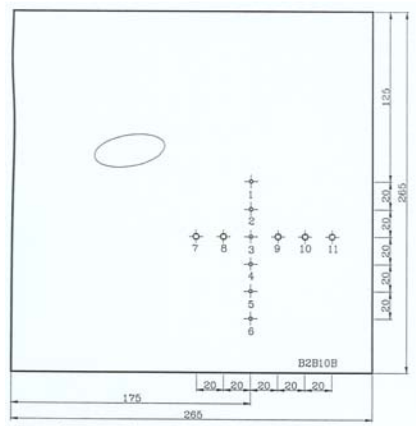


Fig.2. SQUID scanner developed at Fa. WSK

The optimum excitation frequency is given by the skin depth in niobium and the expected depth of the inclusions. In a superconducting resonator, rf currents will only flow at the surface of the resonator, so in principle one would only need to find defects in a depth of a few mm. However, during fabrication of the resonator, up to 0.5 mm of niobium are etched away from the surface, so that even defects in this depth might lead to a reduction in the obtainable resonator quality factor. In order to maximize the eddy-current density in a 0.5 mm thick layer at the surface of the sheet, an excitation frequency of about 40 kHz would be required. Although the excitation field at the location of the SQUID is minimized by the compensation coil, a dynamic range of about 20 to 50 flux quanta is still needed at this frequency to prevent unlocking of the loop by scanning across the edges of the sheet. The slew rate required of the flux-locked loop then is about 4 to 10 F0/µs. In order to minimize temperature drift and be less susceptible to rf interference, we use a conventional ac flux modulated flux-locked loop with a modulation frequency of 4 MHz. The voltage noise of the electronics is about 90 pV/√Hz, and we achieve a dynamic range of about 15 flux quanta at 100 kHz. A niobium dc SQUID is used as a magnetometer with a field-to-flux transfer coefficient of 35 nT/F0; its flux noise is about 1.5 µF0/√Hz, and the field sensitivity is about 50 fT/√Hz. Much higher field sensitivities are possible of course, but since the thermal noise of the sample limits the useful sensitivity anyway, we have made the effective area of the SQUID relatively small to achieve a low flux noise and thus a high slew rate. Because of the high excitation frequency of > 10 kHz and using lock-in detection, we can operate the system unshielded.

IV. TEST SAMPLE

In order to make quantitative measurements and reliably compare the experimental results with simulations, a test sample with Ta inclusions of known size, situated at known locations is required. We used the test sample shown in Fig.3. It consists of a high-purity niobium sheet of 2 mm thickness. At eleven locations, arranged in the shape of a cross, small Ta spheres were embedded into the niobium. Holes of different diameters and depth were drilled and filled with tantalum. After that these location were heated by defocused electron beam up to melting. Finally the sheet surface was completely grinded, so that the defect positions were barely seen (Fig. 3).



Fehler	Sackloch ø[mm]	t[mm]	V[mm³]	mit Tantal gefüllt
1	0,12	0,078	0,0009	Ja
2	0,14	0,21	0,0032	Ja
3	0,145	0,298	0,0049	Ja
4	0,145	0,376	0,0062	Ja
5	0,15	0,474	0,0084	Ja
6	0,15	0,469	0,0083	Nein
7	0,22	0,234	0,0089	Ja
8	0,23	0,333	0,0127	Ja
9	0,22	0,435	0,0165	Ja
10	0,22	0,495	0,0188	Ja
11	0,22	0,501	0,0190	Nein

Fig: 3: Nb test sheet wit Ta inclusions

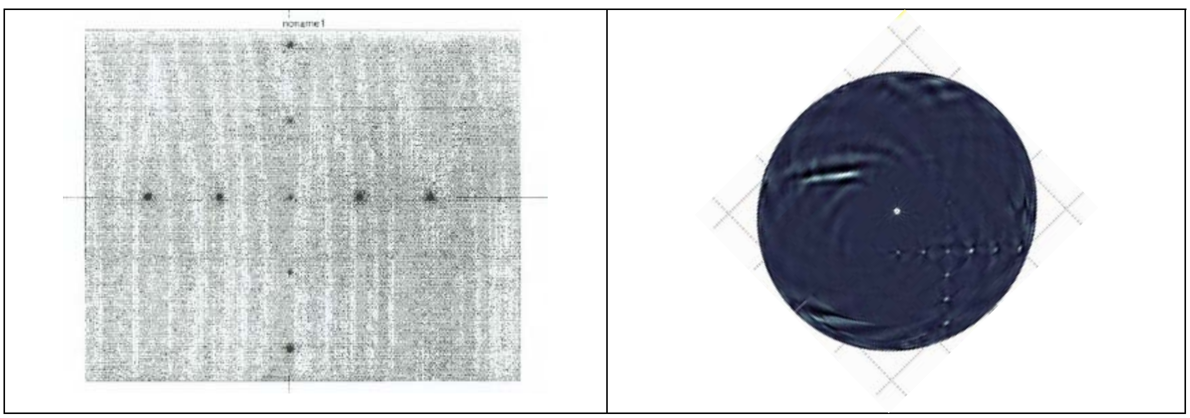


Fig. 4. NAA Neutron activation analysis and eddy current images of the test sheet

The Neutron activation analysis and eddy current images of the test sheet are shown in Fig.4. All artificially produced defects can be located.

V. DETECTION OF TA INCLUSIONS

Although the niobium sheets used for superconducting resonators have a very high purity and so one does not expect the eddy current field to vary across the sample, the signals caused by inclusions can easily be obscured by the lift-off effect. Here, a variation in the stand-off distance between excitation coil and sample leads to a change in the eddy current density in the sample and thus to a change in the eddy current field detected by the SQUID. Already small variations of the order of 10 mm produce field changes larger than the response from a Ta inclusion. Pressing the excitation coil firmly onto the sample

during scanning can reduce the lift-off effect caused by roughness of the sample. Nevertheless, when scanning the surface of the sample using a high excitation frequency (100 kHz), the lift-off effect is still pronounced in this case.
One way to reduce interference from the lift-off effect is to measure the sample from its back side (the side of the sample which later will be the outer wall of the superconducting resonator where no current will flow). The phase of the eddy current with respect to that of the excitation field is a function of the depth in which the eddy current flows. It is possible to maximize the response from eddy currents flowing in a certain depth by choosing the reference phase for the lock-in amplifier following the SQUID electronics appropriately. At the same time, the response from eddy currents flowing at the surface of the sample is reduced, leading to a less pronounced lift-off effect.
Although pressing the excitation coil firmly onto the sample can substantially reduce the lift-off effect, it can at the same time create surface damage which in the end might deteriorate the quality of the superconducting resonator. For this reason we have investigated two further methods for a reduction of the influence of the lift-off effect on the measurement. Under the assumption that all flaws are relatively small (say, 1 mm long or less) and the surface roughness of the niobium sheet is pronounced only over a larger scale (say, several cm), it is possible to reduce the lift-off effect substantially by software filtering. A spatial high-pass filter with a "cut-off frequency" of a few mm will leave the signal of a small Ta inclusion or surface flaw nearly unchanged, but will substantially reduce a variation in the SQUID signal by the lift-off effect, which occurs over a length scale of a few cm - see Fig. 5

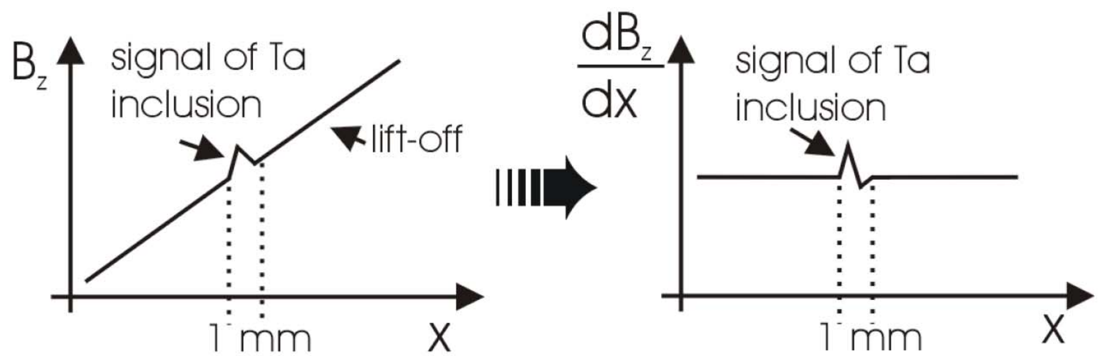


Fig. 5: Basic principle of software filtering was sometimes used to minimize the lift-off effect. Under the assumption that all flaws are relatively small and the surface roughness of the niobium sheet is pronounced only over a larger scale, a spatial high-pass filter with a "cut-off frequency" of a few mm will substantially reduce the lift-off effect.
Using the techniques mentioned above, we were able to detect all Ta inclusions in the test sample with a high signal-to-noise ratio. Fig. 6 shows the two-dimensional distribution of the eddy-current field above the test sample shown in Fig. 3. This measurement was performed by pressing the excitation coil firmly onto the sample and adjusting the phase between excitation current and lock-in reference voltage such that the lift-off effect was minimized. The eddy current frequency was 10 kHz, and the excitation field about 0.6 mT; the scanning speed was 7 cm/s, and the post-detection bandwidth was about 100 Hz. The 11 Ta inclusions are clearly resolved. Some additional structures originating from rolling the sheet can also be seen.

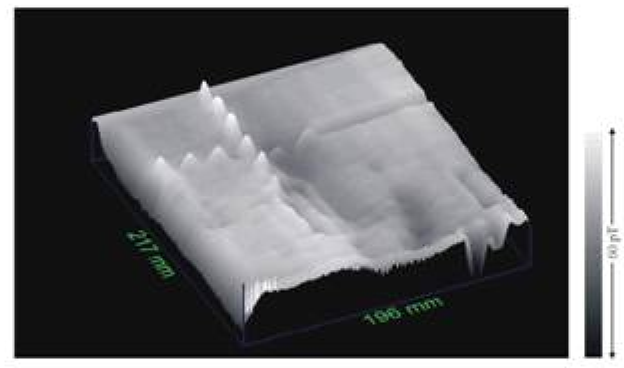


Fig. 6: 3D image of Nb test sheet with artificial Ta defects by SQUID scanning

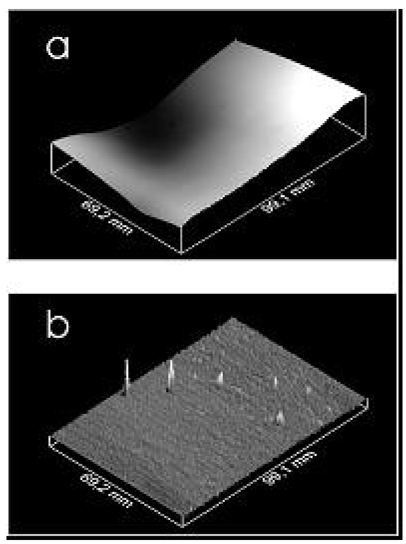


Fig. 7: Unfiltered (a) and filtered (b) data obtained from measuring the test sample with surface Ta defects. In the filtered data we took only into account field changes which occurred over a length scale of less than 2 mm.

As an example for suppressing the lift-off effect by software filtering, we show in Fig. 7a, b the unfiltered and filtered data we obtained from measuring of another test sample shown. In this measurement we took only into account field changes which occurred over a length scale of less than 2 mm. Variations in the eddy current field due to the lift-off effect were then completely eliminated. Finally we discuss an example for reducing the lift-off effect by measuring the sample from its back side and adjusting the phase between the excitation field and the lock in reference voltage such that the response of the system to eddy currents flowing at the surface of the sample is reduced substantially.

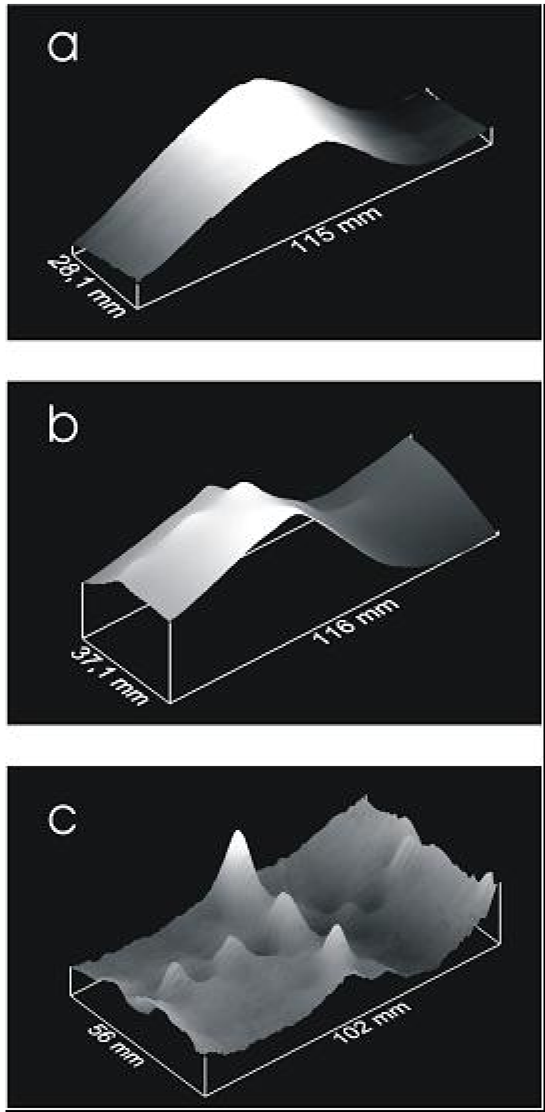


Fig. 8: Two-dimensional distribution of the eddy-current field above the upper left hand part of the niobium test sample

In order to reduce the time needed to scan a niobium sheet, we have tried to increase the separation between the line scans. Since the signal-to-noise ratio is relatively high, one could use a larger excitation coil and thus cover a larger area per line scan. When using a 5-mm diameter excitation coil, we could increase the separation between the line scans to 3 - 5 mm without reducing the probability of detection for the Ta inclusions substantially. An as high as possible scanning speed is advisable, since for the projected accelerator XFEL almost 16000 niobium sheets will have to be tested.

VI. DETECTION OF SURFACE FLAWS

Besides Ta inclusions inside the niobium sheet, surface flaws, such as scratches or residue of grinding or polishing material, can also deteriorate the quality of a superconducting resonator. Although it might be possible to detect such flaws by optical

inspection, it is more convenient to use an electrical testing method. In order to maximize the eddy current density at the surface of the sample, a relatively high excitation frequency of the order of 100 to 200 kHz must be used for these measurements. At such high excitation frequencies the dynamic range of the SQUID system is already quite low. Our flux-locked loop can compensate a maximum flux change of about 10 F0 peak-to-peak (corresponding to 350 nT peak-to-peak) at 150 kHz. We can reliably achieve a compensation factor of the excitation field at the SQUID of about 1000, which limits the maximum excitation field produced by the excitation coil to 350 µT peak-to-peak at most. Since the niobium sheet is very homogenous and its surface is relatively flat, the eddy current field at the SQUID will only slightly change as the sample is scanned. For this reason we could use an excitation field of about 100 µT peak-to-peak even at an excitation frequency of 100 kHz in all measurements performed so far. The "measurement dynamic range" (defined as the ratio between excitation field and field noise in 1 Hz bandwidth) still is higher than 180 dB/√Hz. We have tested a number of niobium sheets for surface flaws with the system described above. Some niobium sheets had artificial surface defects, such as scratches or small indentations. Fig. 9 shows the results of a typical measurement of a niobium sheet with nine artificial surface flaws (indentations about 100 µm in diameter and depth). The eddy current frequency used was 110 kHz, the excitation field about 100 µT peak-to-peak. All nine surface flaws could be detected. For comparison, in Fig. 10 we show a measurement of the same sample using a conventional eddy current NDE system.
Only one or two of the surface flaws could clearly be detected with this system.

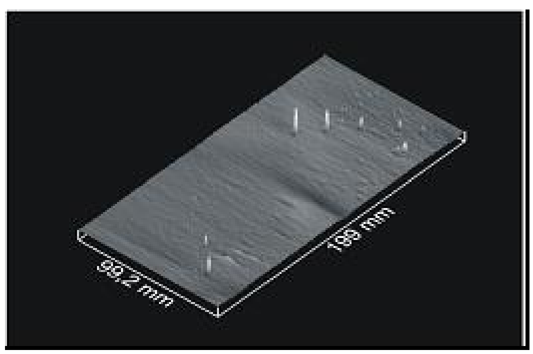


Fig. 9: Two-dimensional distribution of the eddy-current field above a test sample containing a number of surface flaws, measured with our SQUID system. The eddy current frequency was 110 kHz and the diameter of the excitation coil was 3 mm.

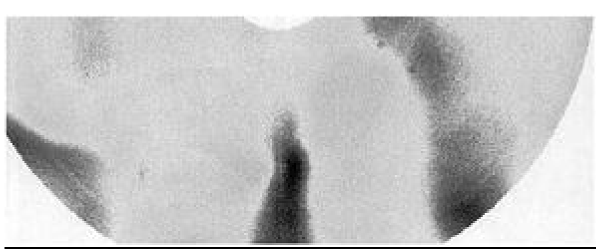


Fig. 10. Same sample as in Fig. 9, measurement was performed with the DESY eddy current system.

VII. COMPARISON OF SQUID AND EDDY CURRENT TESTING RESULTS ON NB SHEETS

Due to the relative complex design of the SQUID electronics, the usable frequency spectrum is limited to around 0-100 kHz. For Measurements on Nb sheets, frequencies from 20 to 90 kHz have been used. In difference to the BAM system used at DESY, only a single frequency is used. One reason for this lies in the very high sensitivity of the SQUID system: Even with 90 kHz scratches and paintings on the back of the sheets (thickness: 2.8 mm) are detected. This means, that not only defects within the skin depth are detected, but up to a multiple of it remain visible.

The following images show testing results on Nb sheets both from the SQUID measurement system and the DESY- BAM test equipment.

Very significant is the difference in measurement expense: DESY-BAM uses two different excitation frequencies on a total of four input channels, the table itself scans with four times the track density and the sensor head used for moving the coils over the sample are special constructions lifted by pressurized air in order not to touch the surface of the sample.

The images are placed side-by-side, so a comparison of conventionally taken images and the SQUID measurements can easily be made. For example figure 11a: Scan of sheet 41 with SQUID sensor system. Figure 11b: Scan of sheet 41 with conventional sensor system. DESY’s default setup was used, with 4.16 l/mm track density. The y-value of channel 2 (at 1 MHz) is shown, which (in this example) delivers the most similar image).

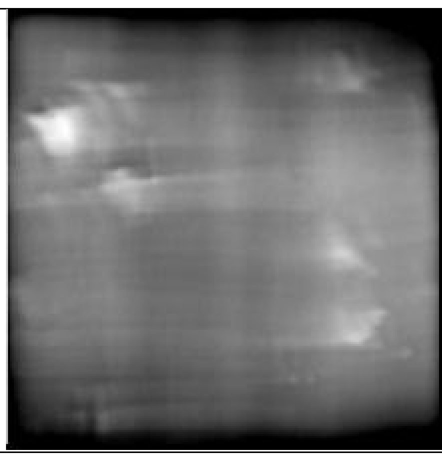
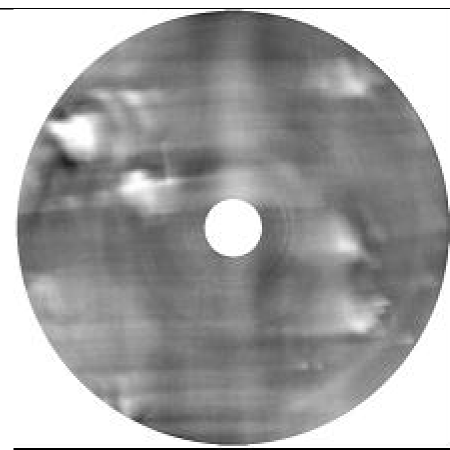
	

Fig. 11a : Scan of sheet 41 with SQUID sensor system. Scanning resolution was set to 300 scan lines, which corresponds to 1 l/mm. The frequency used was 33 kHz, which is closer to DESY’s measurement channel 1 (170 kHz).	Figure 11b: Scan of sheet 41 with conventional sensor system. DESYs default setup was used, with 4.16 l/mm track density. The y-val. of channel 2 (at 1 MHz) is shown.

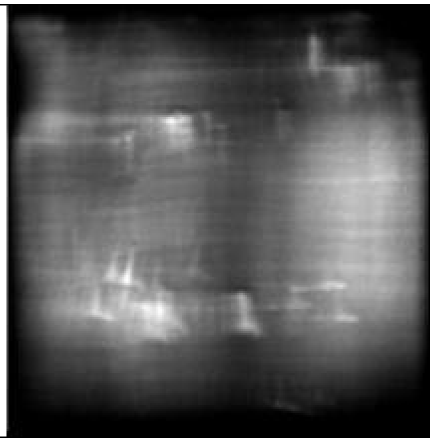


Figure 12a: SQUID-scan of sheet 45. Detailed reproduction without artefacts caused by lift-off. No circular artefacts from the scanning process.

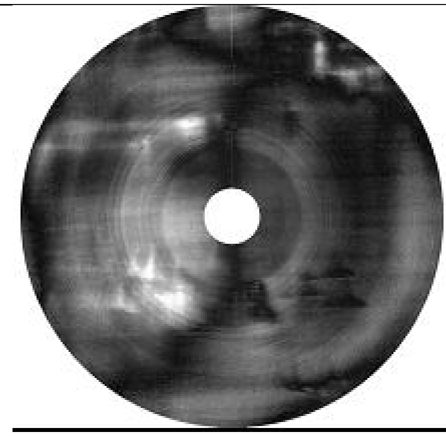


Figure 12b: Eddy current scan of sheet 45 (amplitude of channel 1). The dark shapes are partially artefacts caused by lift-off and the signal is close to noise level.

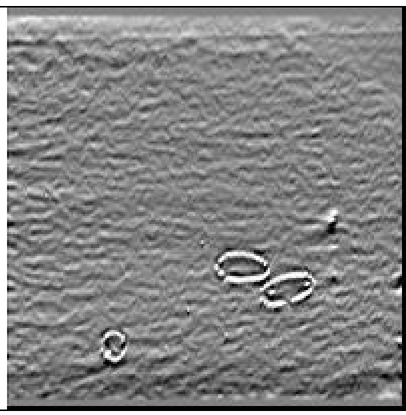


Figure 13a: Scan of sheet 9 with SQUID sensor system. The three ellipses shown are pen markings for scratches (within the ellipses) on the BACK of the sheet. Some more dots and one large signal source (centre left) are visible.

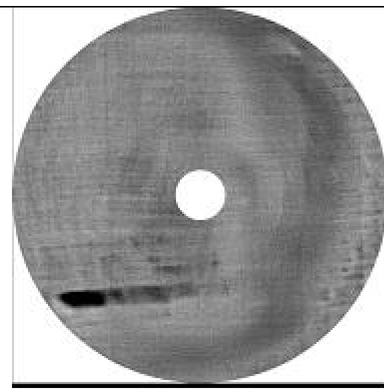


Figure 13b: Scan of sheet 9 with conventional sensor system. The y-val. of channel 1 (at 170 kHz) is shown. None of the scratches is visible, as the back of the sheet is far beyond the skin depth of the frequency used.

VIII. SCANNING OF INDUSTRIALLY PRODUCED NIOBIUM SHEETS FOR 1.3 GHZ RESONATORS

20 niobium sheets of the Fa. Plansee for the cavity AC115 scanned before annealing with WSK SQUID scanner. Excitation frequency was 6.3 kHz.

- Surface structures (increasing of surface roughness) are detected in the sheets No. 14, 24 and 26 is probably caused by the rolling.
- Sheets 12, 17, 18 und 20 demonstrate small density gradients in corners.
- All sheets (excluded sheet No. 28) are defect free. The sheet No. 28 has a delamination in the left down corner penetrating from the surface into the bulk (Fig. 14). This sheet was sorted out and not used for cavity fabrication.

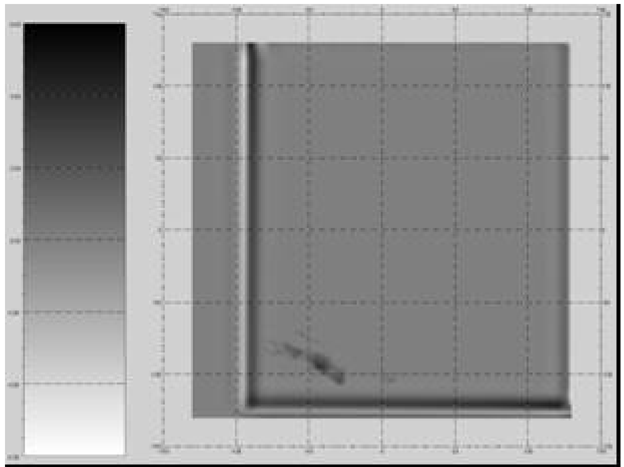
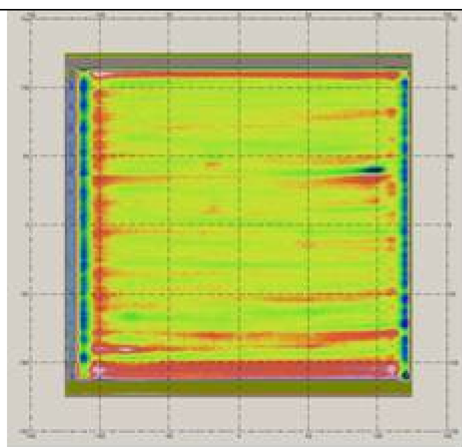
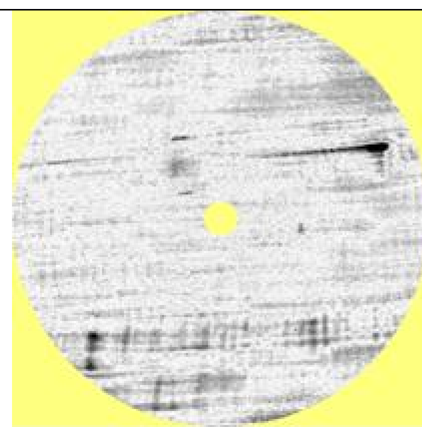


Fig.14: Delamination detected at the sheet by SQUID scanning

21 niobium sheets of the Fa. Tokyo Denkai has been scanned with WSK SQUID scanner. SQUID scanning results have been compared with Eddy Current scanning results get for the same sheets earlier. The sensitivity of the SQUID apparatus is at least on the same level as of the EDDY current apparatus. Two examples of the comparison can be seen in the figures 15 - 16.

<table>
<tr><td></td><td></td></tr>
<tr><td>Fig. 15a: SQUID image of the NC-1355-335 Nb-sheet</td><td>Fig. 15b: Eddy Current image of the NC-1355-335 Nb-sheet</td></tr>
</table>

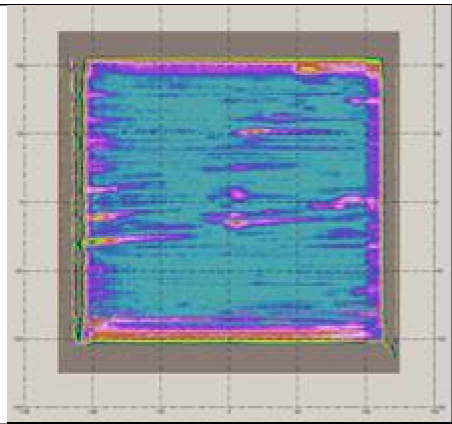


Fig. 16a: SQUID image of the NC-1357-400 Nb-sheet	Fig. 16b: Eddy Current image of the NC-1357-400 Nb-sheet

It can be seen that for the sheet NC-1357-400 the SQUID sensor makes the potential flaws even more visible as the eddy current.

IX ANALYSIS OF THE ADVANTAGES AND DISADVANTAGES IN APPLICATION OF THE SQUID DEVICES FOR THE EUROPEAN X-RAY LASER PROJECT XFEL

Pro	Contra
More sensitive procedure compare to conventional eddy current devices	Not sufficient experiences of industrial application
The lift off effect is less pronounced compare to eddy current devices with conventional pick up coil	The prototype with XY scanning table is build. The rotating table and the software for scanning of the round discs are not available.
	More complicate technique. Using of liquid He is required. Set up of the electronics is laboriously. Providing of the spare parts are critical
	Due to the relative complex design of the SQUID electronics, the usable frequency spectrum is limited to around 0-100 kHz. The signal penetrates completely through the sheet, what produce difficulties to determine the RF side of the sheet

X. SUMMARY

Good progress was achieved in last years in R&D of the SQUID scanning system. It was demonstrated that the developed at Fa. WSK SQUID scanning apparatus is in position to detect material defects of the size > 50 µm in the plane sheets. The time consuming for SQUID scanning is comparable to Eddy Current devices. The SQUID scanning apparatus developed at Fa. WSK is produced and delivered to DESY. The system is at the moment in the testing phase.

XI. LITERATURE

1. R. Casperson, R. Pohl and H.-M. Thomas, "Nachweis von Verunreinigungen in Supraleitern mit dem Wirbelstromverfahren", Berichtsband der DGZFP 68, 735-742 (1999), http://www.ndt.net/article/v04n08/casperson/casperson.htm (in German).
2. H. Weinstock and M. Nisenoff, *Review of Progress in QNDE* 6, 669-704 (1986).
3. Y.P. Ma and J.P. Wikswo Jr., *Review of Progress in QNDE* 13, 303-309 (1994).
4. A.Cochran, J.C.Macfarlane, L.N.C.Morgan, J.Kuznik, R.Weston, L.Hao, R.M.Bowman and G.B.Donaldson, IEEE Trans.Appl.Supercond. AS-4, 128 (1994).
5. J. H. Hinken and Y. Tavrin, *Review of Progress in QNDE* 19, 2085-2092 (2000).
6. Cryoton fibre-glass dewar LH-1.2-TE, http://cryoton.webzone.ru/.
7. W. Singer, A. Brinkmann, DESY F. Schölz, A. Farr, E. Wappler, WSK M. Mück, ez-Squid. *SQUID-BASED SCANNING SYSTEM FOR DETECTING DEFECTS IN NB SHEETS FOR RF CAVITIES.* Proceedings of the 12th International Workshop on RF Superconductivity (SRF2005), Ithaca, NY
8. M. Mück, Ch. Welzel, A. Farr, F. Schölz, and W. Singer. Nondestructive testing of niobium sheets for superconducting resonators. Proceedings of ASC 2002, August 4-9, Houston (USA). - (2002)
9. W. Singer, A. Brinkmann, D. Proch, X. Singer Quality requirements and control of high purity niobium for SRF cavities. Physica C 386 (2003)