# WP6. MATERIAL ANALYSIS

## Task 6.3.

# DC field emission studies of Nb samples

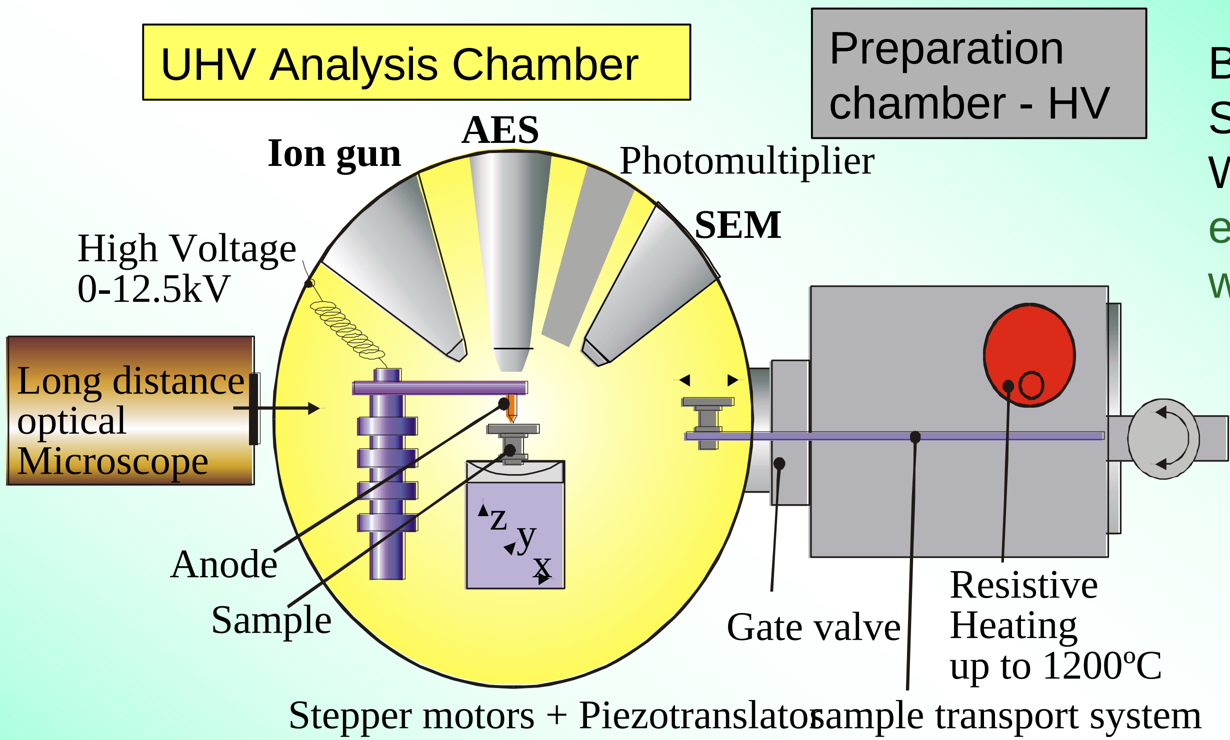
(CARE work started at BUW on July 1st 2004)

**Arti Dangwal**

**Günter Müller**

Hamburg, November 3rd 2004

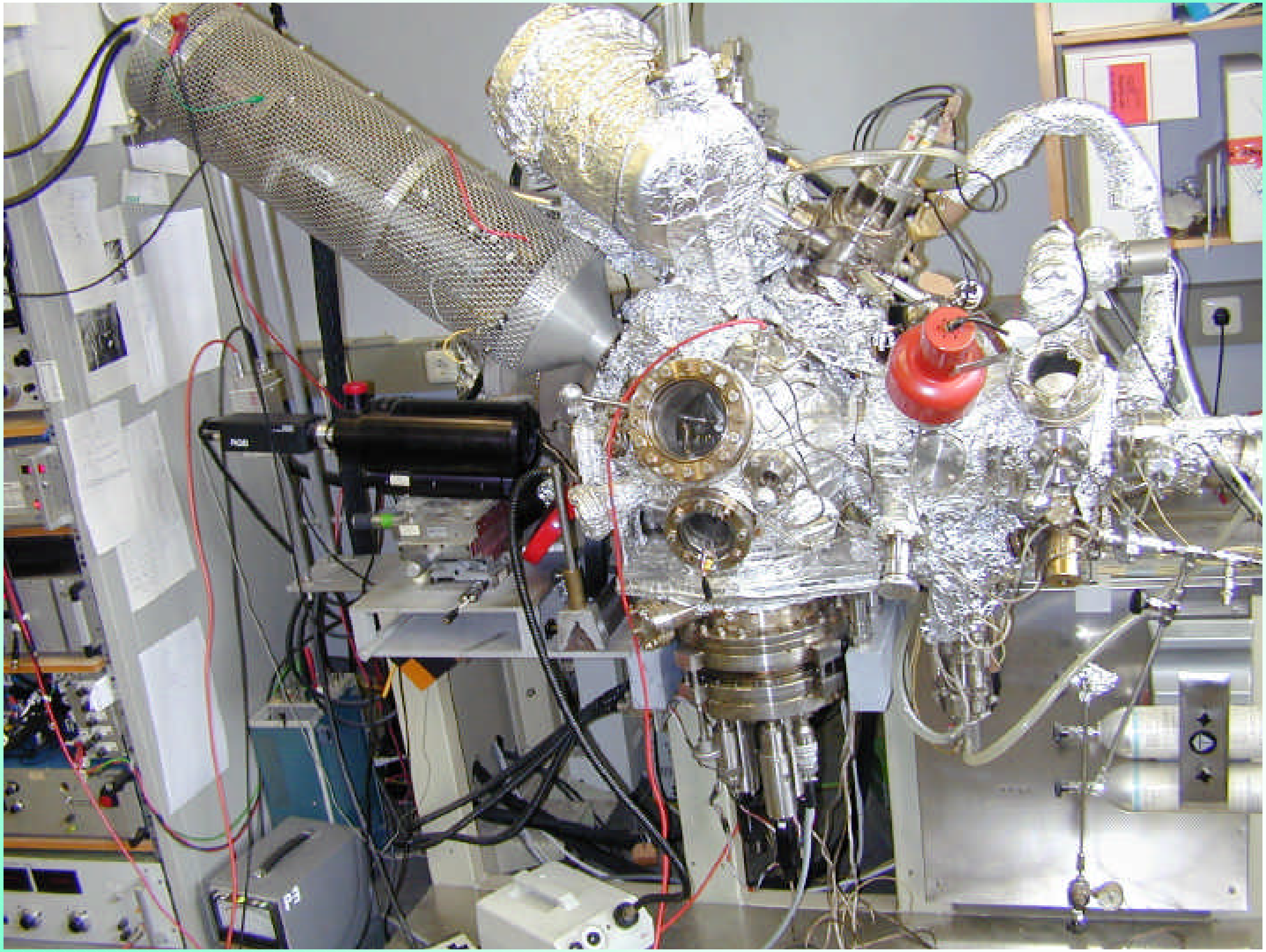
# Field Emission Scanning Microscope and Measurements



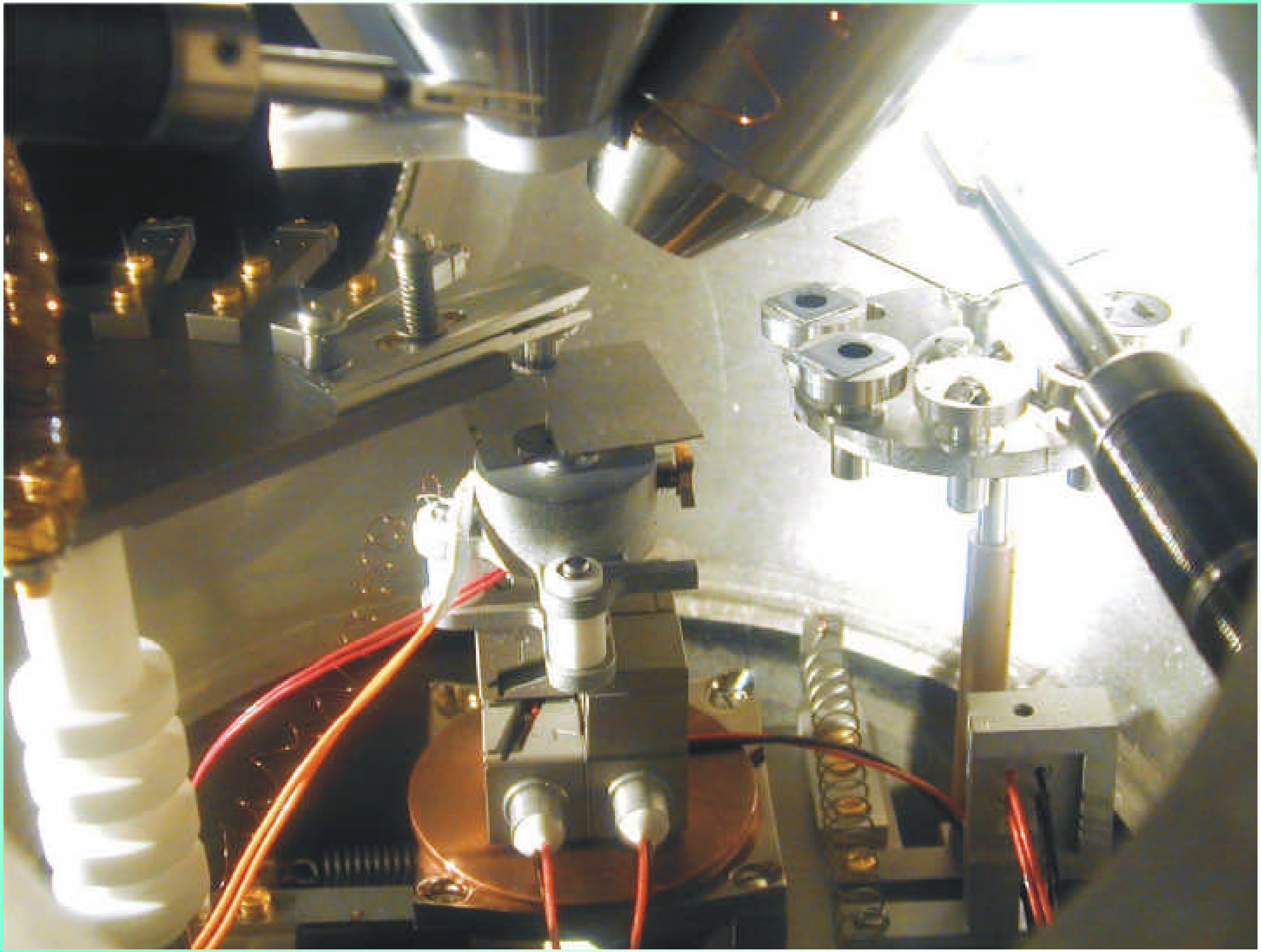

Base pressure $10^{-10}$ mbar
Sample size up to Ø 30 mm
W flat anodes Ø 0.1-1 mm
exchangable W tips
with apex Ø 20 nm - 20 µm

- Voltage scans U(x,y) for a given maximum current at constant electrode distance with fast voltage regulation to prevent discharges
- High resolution measurements of the field emission properties of single emitters (I/V-curves, current limits, stability, fluctuations)
- Emitter identification by means of in-situ SEM (> 0.5 µm), AES and ex-situ SEM
- Emitter conditioning by in-situ Ar-ion gun or resistive furnace up to 1200 ºC

# UHV Surface Analysis System with FESM



# View into the centre of the FESM



# MODIFICATION OF SCANNING APPARATUS

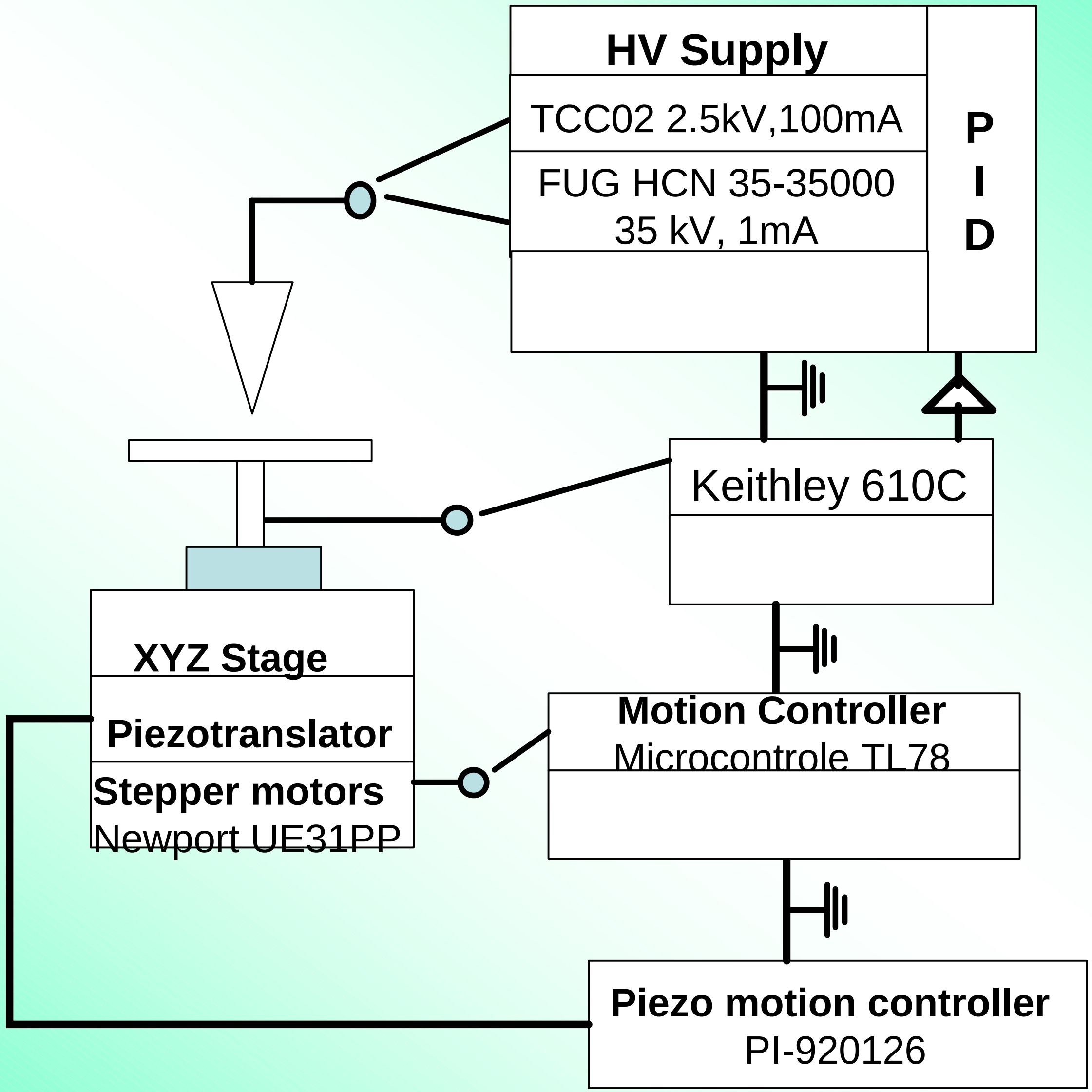
**Motivation:**

- Old motion control too slow for large area (Ø30 mm) samples
- Analog current measurements cause delay and failures
- Two different computers for stepper motor and piezo scans
- Electropolished samples show unflatness up to some 10 µm

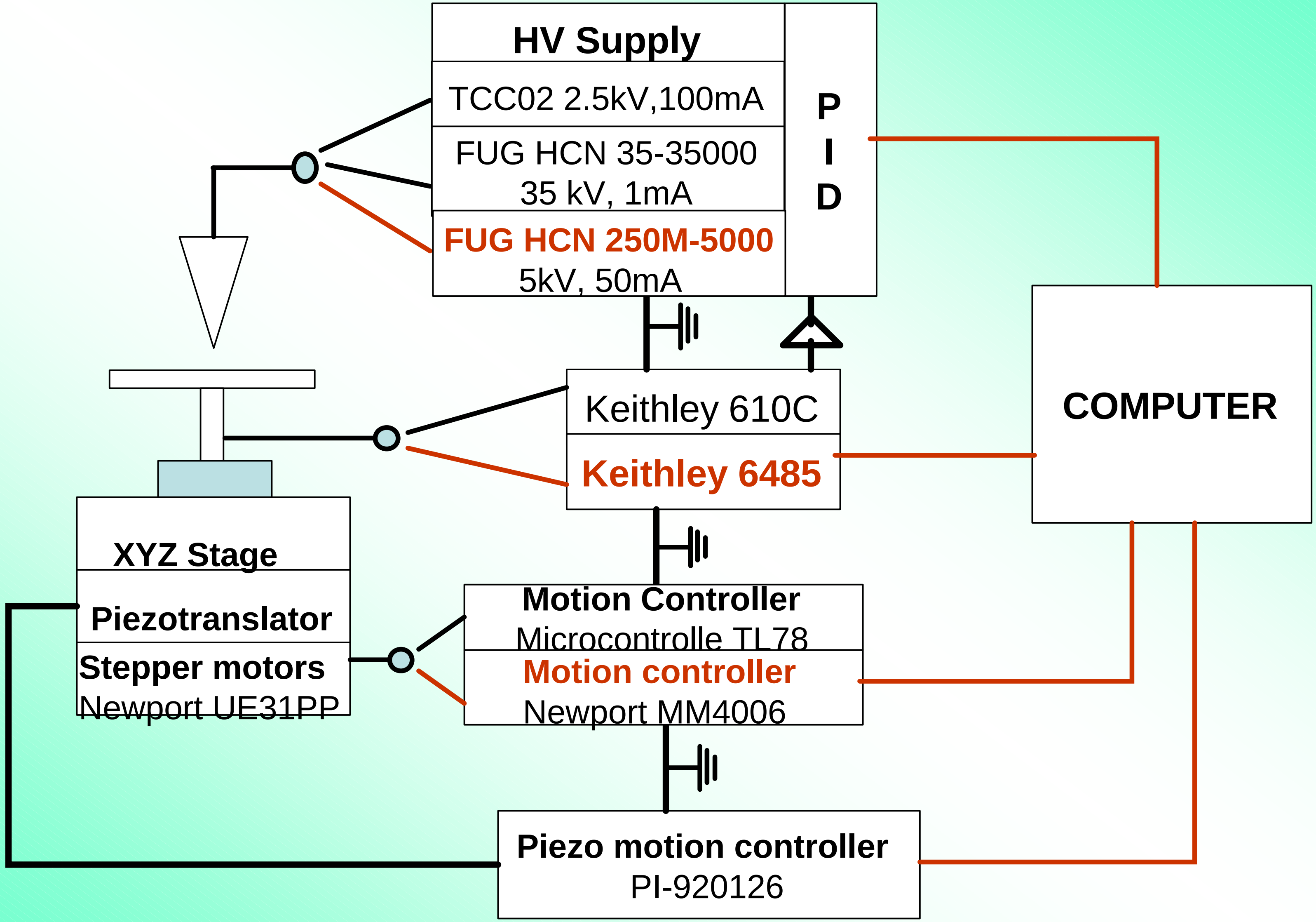
**Ongoing improvements:**

- New pulsed power supply (5 kV in 2 ms) with digital interface
- New 3D motion controller with digital interface (100 steps in 14 s)
- New fast picoamperemeter (1 kHz) with digital interface
- New computer with LabVIEW for fast scans and data recording
- New image analysis software for fast data analysis

# Electronics of FESM at present



# Electronics of FESM after modification



# New hardwares and softwares

- **DC Pulsed Power supply** (FUG HCN 250M-5000)
  - Power class 250 W, 50 mA at 5 kV
  - Full height rise time of 2 ms
  - IEEE488 interface
  - Testing in progress
- **3D Motion controller** (Newport MM4006)
  - High-speed stepper motor drive
  - 100x100 scan in about 30 min
  - IEEE488 interface
  - Testing done

# New hardwares and softwares ctnd.

- **Picoamperemeter** (Keithley 6485)
  - 10fA resolution
  - Up to 1000 readings/second
  - Autoranging from 20 fA to 20 mA
  - Instrument just received

- **Computer with LabVIEW**
  - 512 MB RAM, 2 GHz, ~ 100 GB hard disc
  - DESY license for LabVIEW
  - Automatization of stepper motors and piezos with corrections.
  - Programming of FE scans planned

# Outlook

Measurements:

Systematic testing of a series of 10 large Nb samples (Ø 30 mm) planned

First electropolished samples received:

- 2 from CEA Saclay (C-Antoine)
- 2 from DESY prepared in 9 cell structures (A. Matheisen)

Investigation of new preparation steps - Dry-ice cleaning (D. Reschke)

Goals:

Statistically relevant correlations between preparation steps/parameters and field emission properties

Identification of field emitters and enhanced emission mechanisms

Influence of current processing and ion irradiation on field emitters

⇩

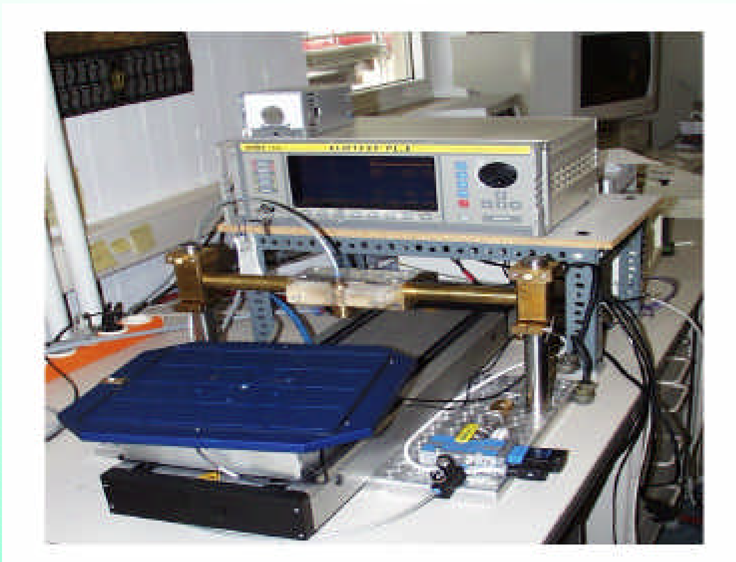
Suggestion for a simplified quality control for standard preparation technique

# WP 6 Task 6.1 SQUID scanning

DESY -- W. Singer

**Deliverables: Reports about technology of SQUID scanning, design, fabrication and operation of a prototype scanner, definition of best operating conditions, design of an industrial SQUID scanning machine**

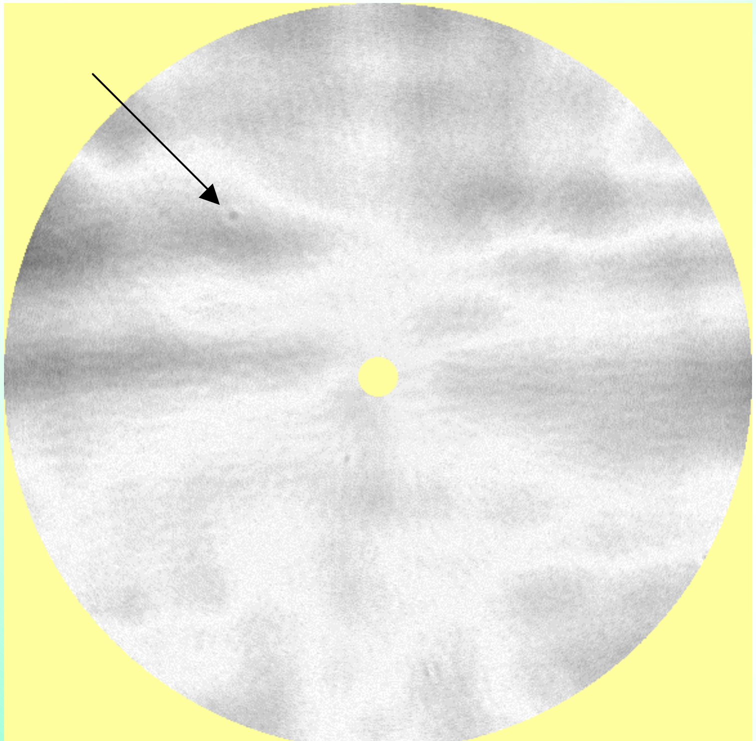
Search for clusters in Nb sheets. An eddy current system for scanning of Nb sheets was developed.



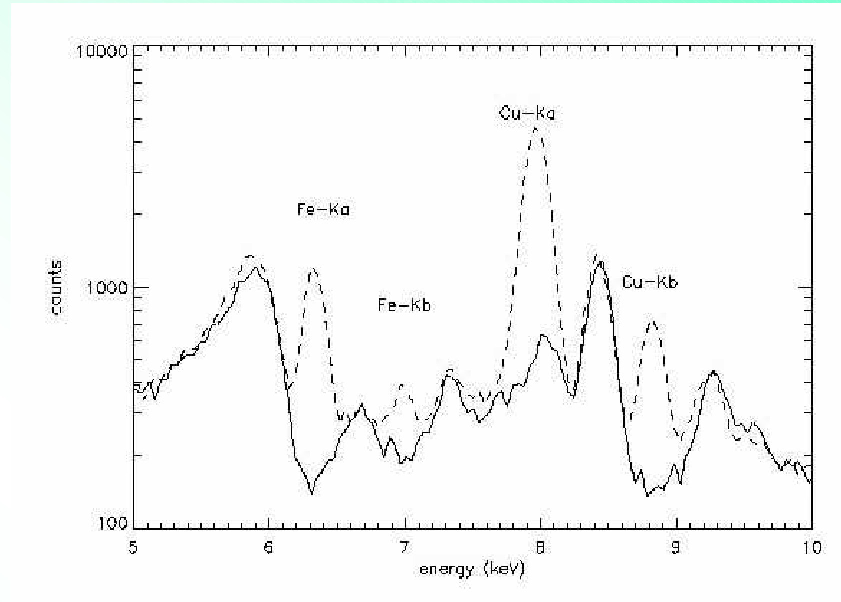
DESY eddy current scanning apparatus for niobium discs. 100% Nb sheets for TTF scanned and sorted out

Eddy current scanning system produced at company FER (Germany) for SNS Nb sheets

Example: A spot observed on the eddy current image was identified as an inclusion of foreign material. Cu and Fe signal has been observed in the SURFA spectrum in the spot area.



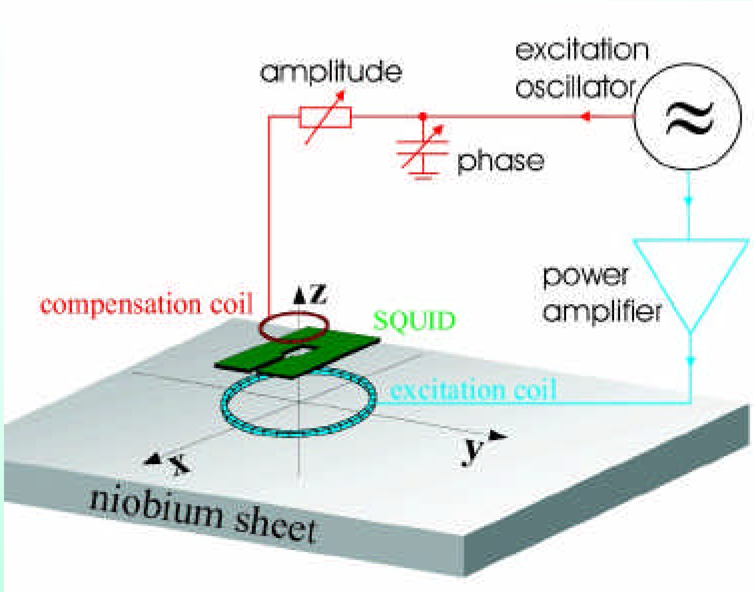
Example of the Nb sheet eddy current scanning test. Arrow indicates the suspicious spot.



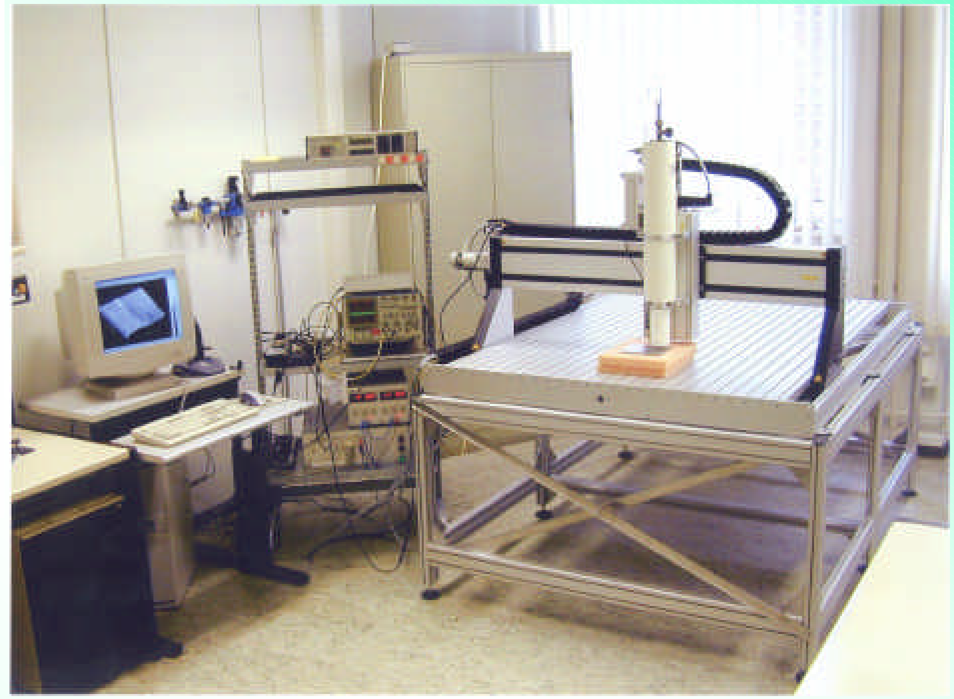

SURFA (Synchrotron Radiation Fluorescence Analysis). Spectrum of K-lines at the spot area (dashed line) in comparison with spot free area (full line).

The ratio signal/noise is rather low. We need a more sensitive system, especially if we will cancel the post purification with Ti (possibly a SQUID Scanner)

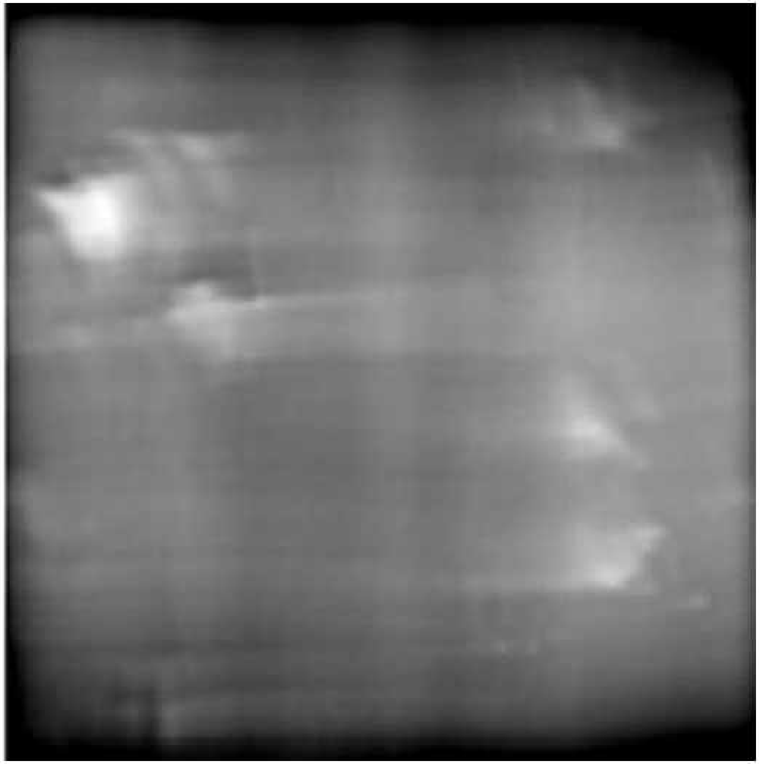
# Aim: SQUID system for eddy current testing of niobium sheets




An excitation coil produces eddy currents in the sample, whose magnetic field is detected by the SQUID. A compensation coil close to the SQUID cancels the excitation field at the SQUID.

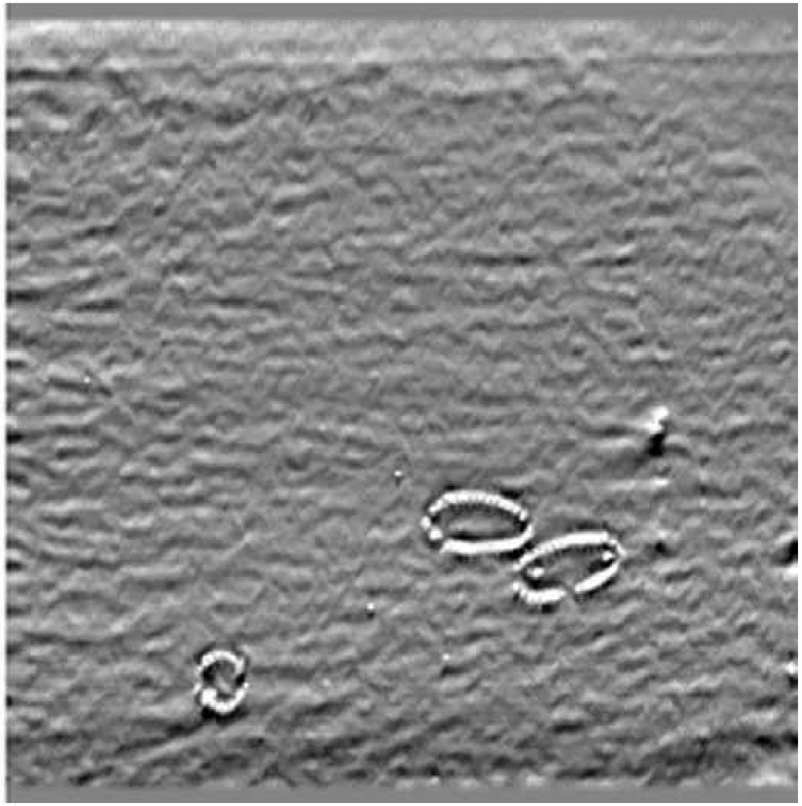


SQUID-based scanning system developed at company WSK and University Gießen (Germany) for big parts 1.5 x 1.5 m
Scanning of Nb sheets possible ( as it can be seen on picture)



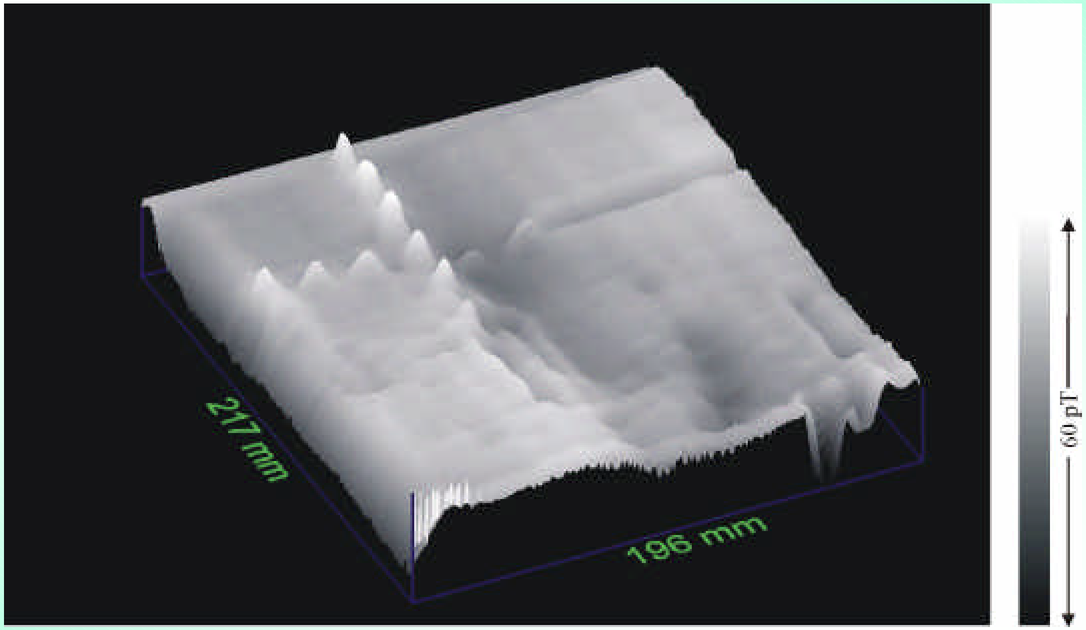
Scan of sheet 41 with SQUID sensor system (left). Scanning line density is 1 line/mm, excitation frequency 33 kHz.
Scan of sheet 41 with DESY eddy current system (right). Scanning line density ca. 5 lines/mm, excitation frequency 170 kHz.

Scanning time of 5 min for the sheet 265x265mm by SQUID system is realistic

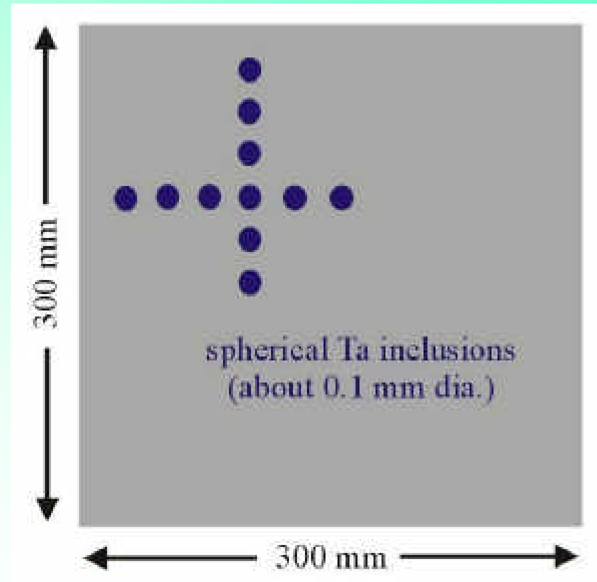


Scan of sheet 9 with SQUID sensor system and excitation frequency 90kHz (left). The three ellipses shown are pen markings for scratches (within the ellipses) on the BACK of the sheet. Some more dots are visible.
Scan of sheet 9 with DESY eddy current system and excitation frequency 170 kHz (right). None of the scratches is visible on the back of the sheet.

# Niobium sheet with artificial bulk defects

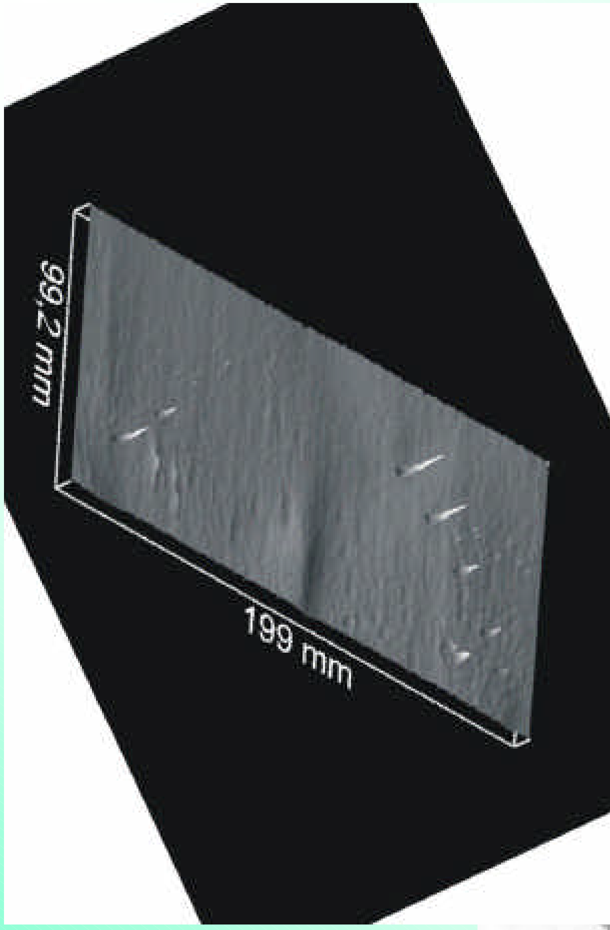



Two-dimensional distribution of the eddy-current field above the niobium test sample. The excitation field generated by a 3-mm diameter coil was about 0.6 mT peak-to-peak; the eddy-current frequency was 10 kHz. The eleven embedded Ta inclusions are clearly detected.

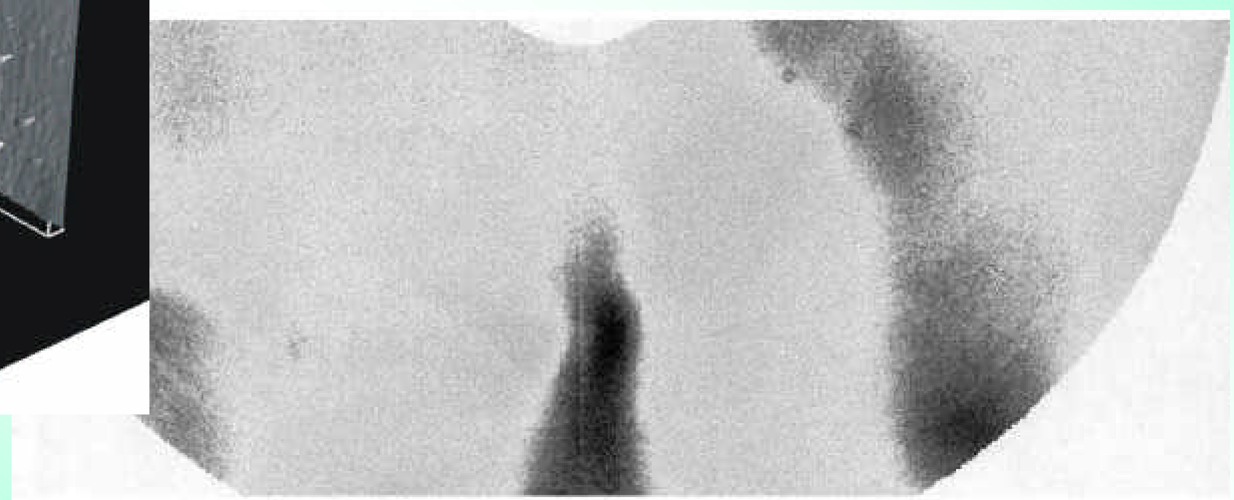



Sketch of the test sample used in measurements. Eleven Ta spheres with diameters of about 0.1 mm were embedded into a 30 x 30 $cm^2$ niobium sheet by electron-beam melting of the surface.

# Niobium sheet with artificial surface defects




**Two-dimensional distribution of the eddy-current field above a test sample containing a number of surface flaws (tantalum inclusions of size 0,1-0,05 mm close to surface) , measured with SQUID system.**
**Eddy current frequency 110 kHz, diameter of the excitation coil 3 mm.**



**Same sample, however, measured by the conventional eddy current system**
**SQUID system promise to be more sensitive**

Task: Creating in cooperation with WSK a SQUID scanning system for Nb sheets of 265x265 mm (prototype for XFEL)

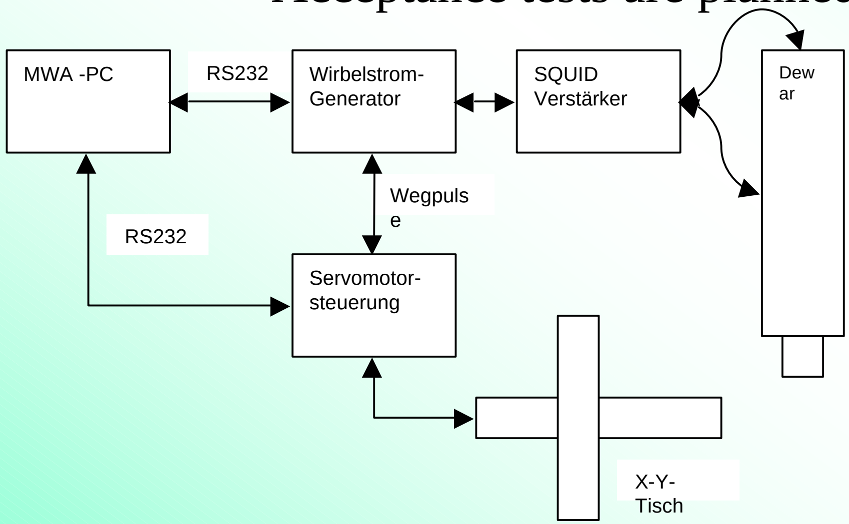
Specification for SQUID scanning system should include:

- rotating table
- high signal/noise ratio
- compensation of lift off effect
- rather fast scanning (5-10 min/sheet)
- vacuum holder of the sheet, keeping it flat

# Status:

## Fa. WSK has got a contract for fabrication of a prototype of a SQUID Scanner for Nb sheets

- Concept and main design of a device is worked out (Low Tc SQUID)
- Acceptance tests are planned for end of 2005




- Drawback: XY scanning table. Attempt: additionally order a rotating table

After finishing of SQUID development a decision should be taken what scanning system is more reasonable for XFEL. SQUID system seems to be more sensitive but more complicate

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# WP6 Task 6.2

## Flux gate magnetometry applied to SC cavities

C.Bonavolontà, V.Palmieri, M. Valentino

Double task:

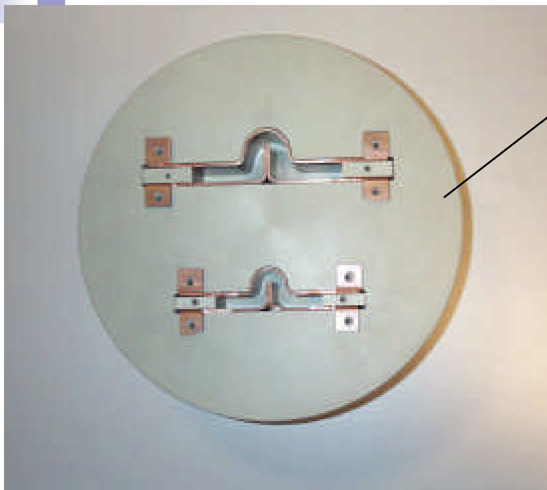
- Defect diagnostics on Niobium surfaces
- Better diagnostics of EP process

## WP 6 Task 6.2 Flux gate magnetometry

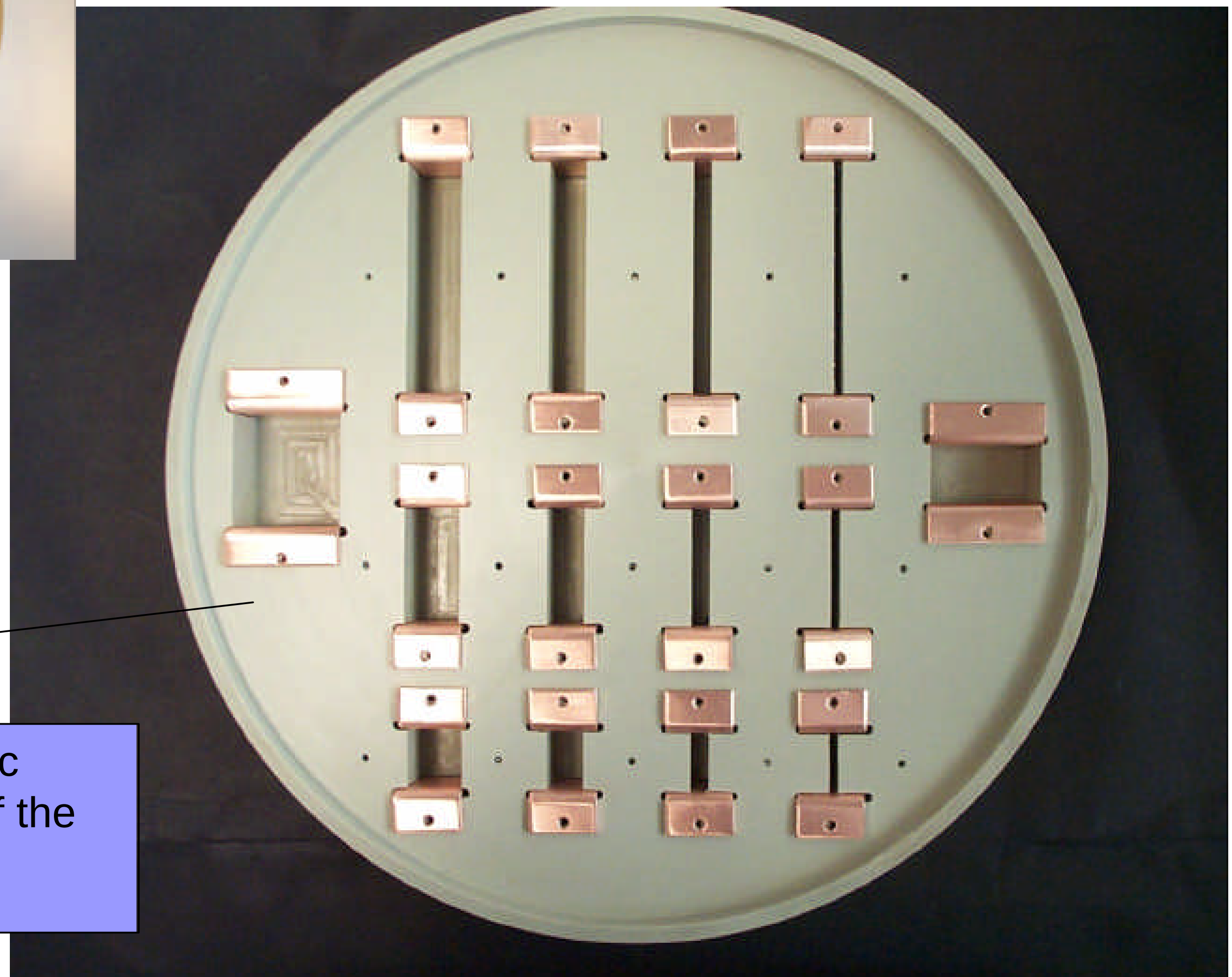
- designing a cavity shaped electrolytic cell having the possibility to test different cathode shapes. The goal is a tomography of the electrolytic cell to figure out the effect of cathode geometry on EP

- Designing the experiment to monitor two different kind of defected samples:
  1. Physical defects like surface scratches and foreign particle embedded onto Niobium
  2. Samples with degraded RRR to distinguish from samples with RRR 300.

## Preliminary investigation with Cu in $H_3PO_4$ bath



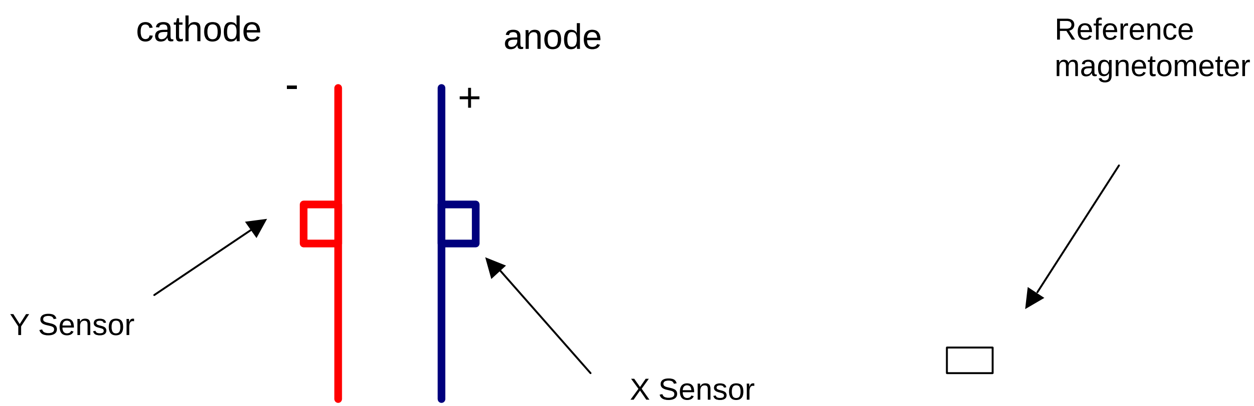
The final goal



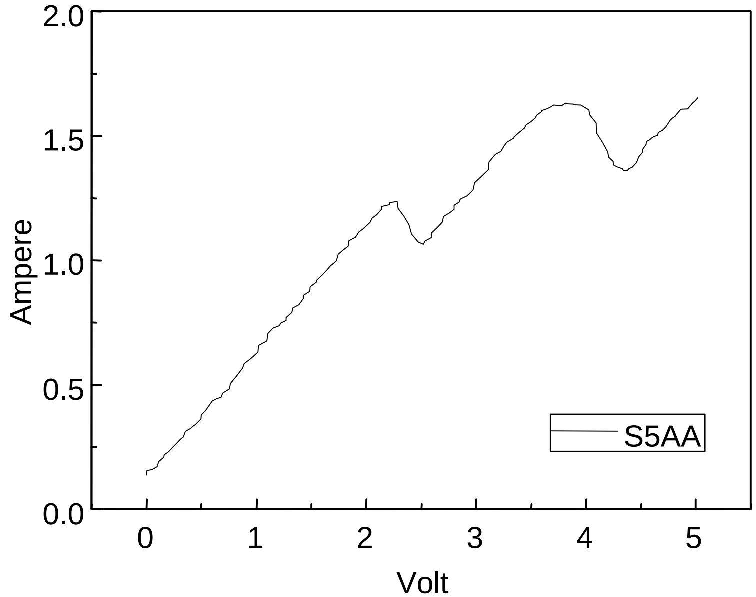
A systematic investigation of the methode

*Planar configuration with three Magnetometers:*

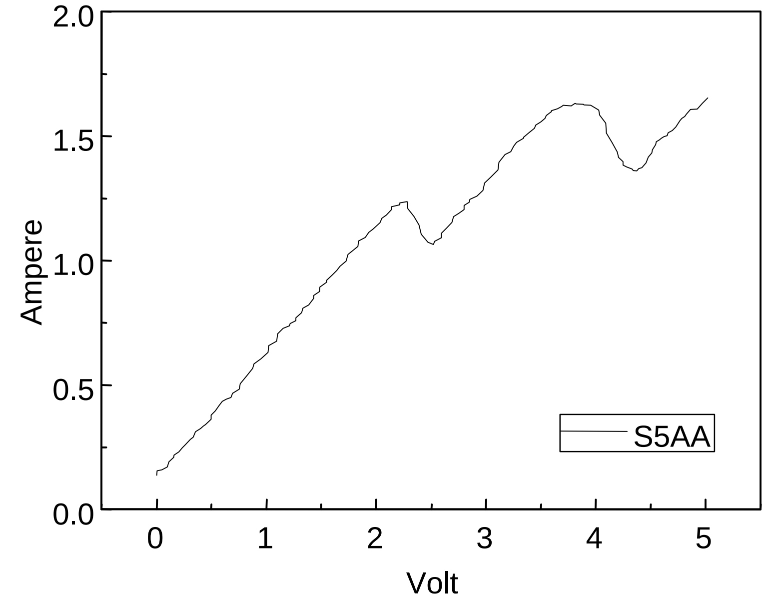
- *Sensor at the anode,*
- *Sensor at the cathode*
- *Sensor at a reference point at 1 mt from the EP cell (10,7 mTesla measured)*



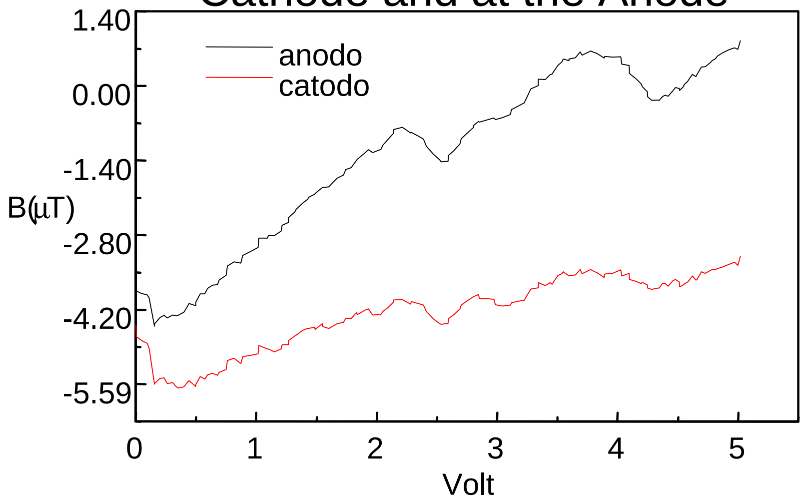
## I-V Polarization curve



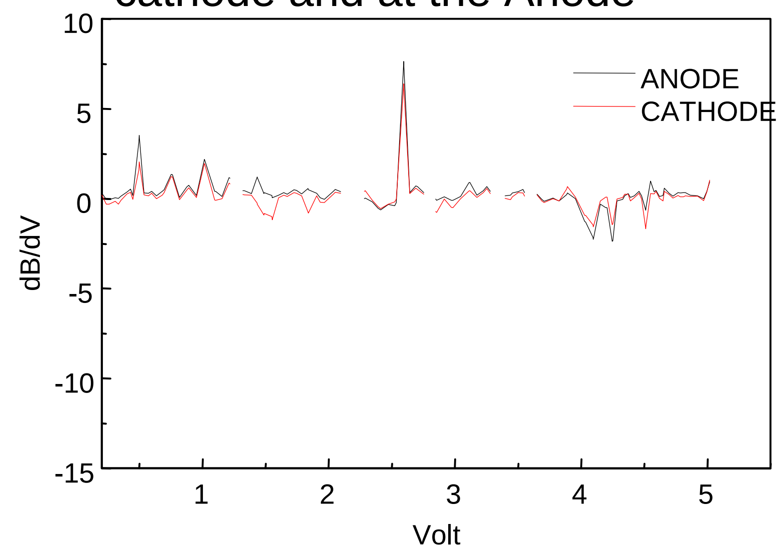

## I-V Polarization curve




## Magnetic Characteristics at Cathode and at the Anode




## Field derivative Vs voltage at cathode and at the Anode

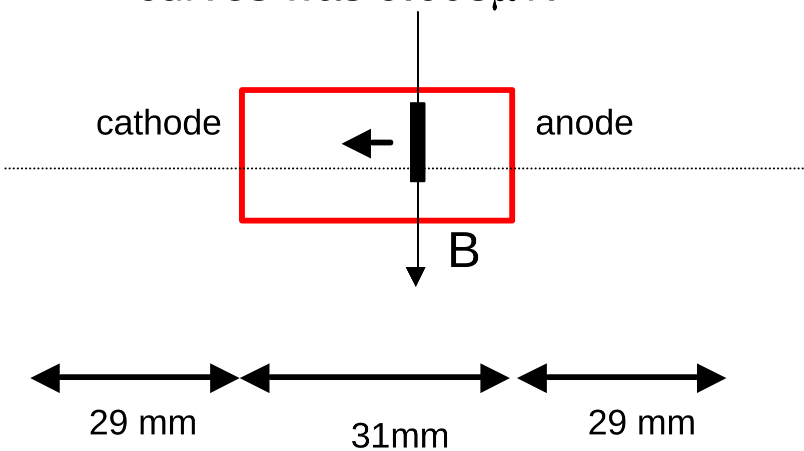


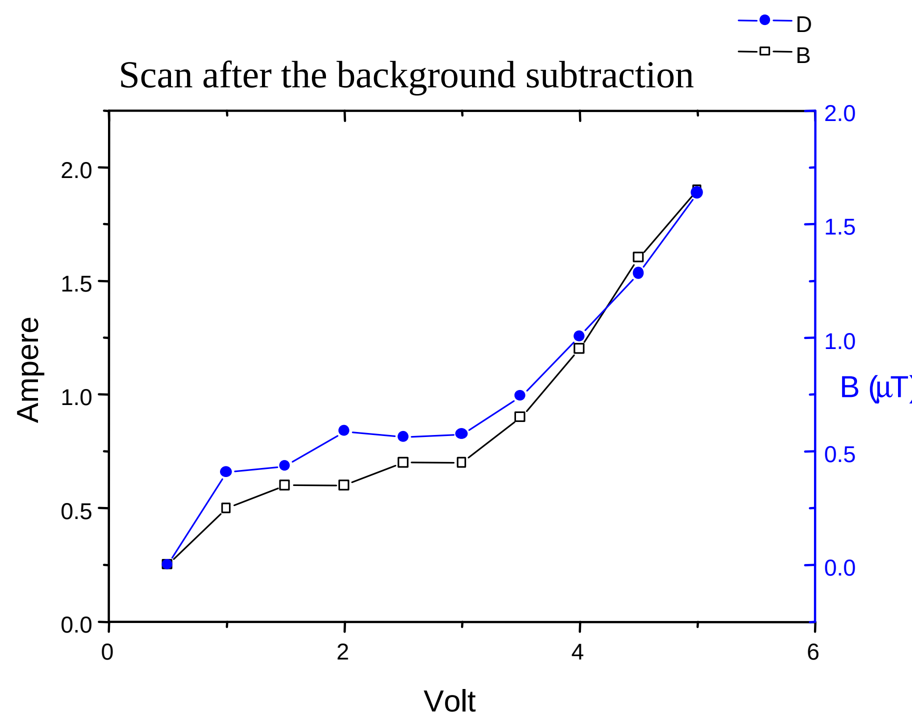
**Scanning the EP cell**

We have scan from anode to cathode at a distance from solution of about 4.5 cm. Every reported value of the magnetic field is the average on 190 points

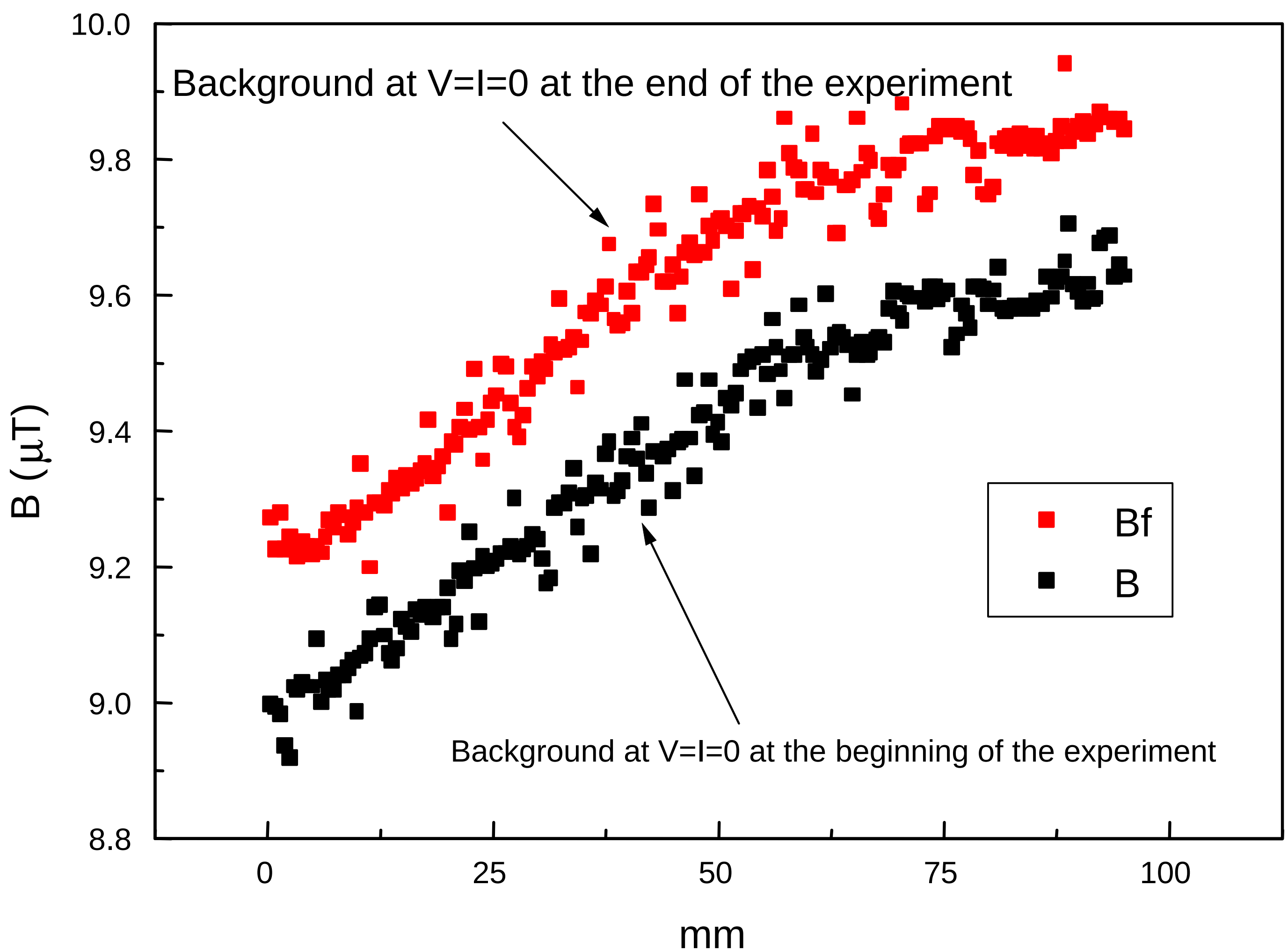
The value of the field when starting the scan at I=V=0 was 9.581μT.

The background that was measured at I=V=0 after having measured all the curves was 9.608μT.

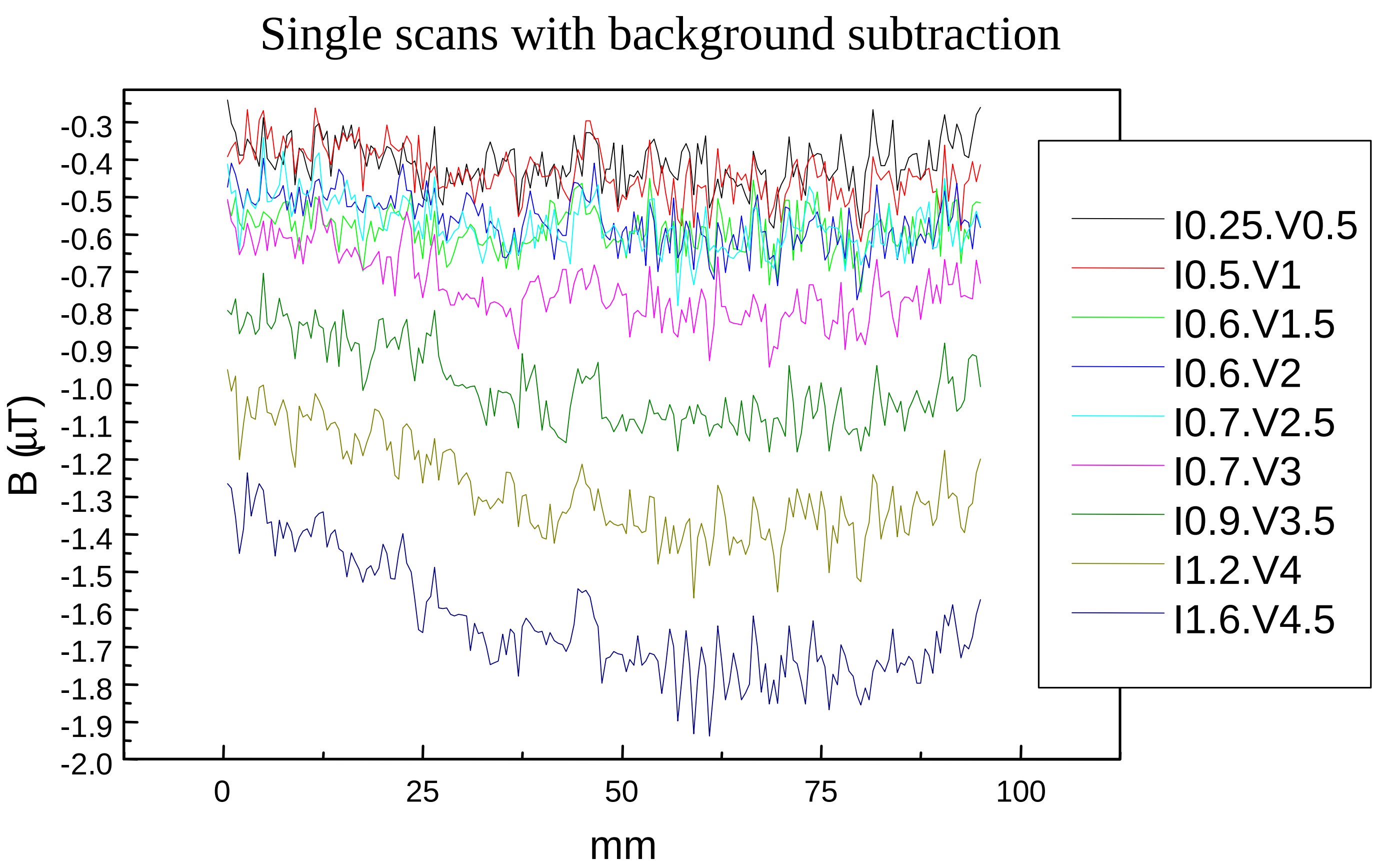


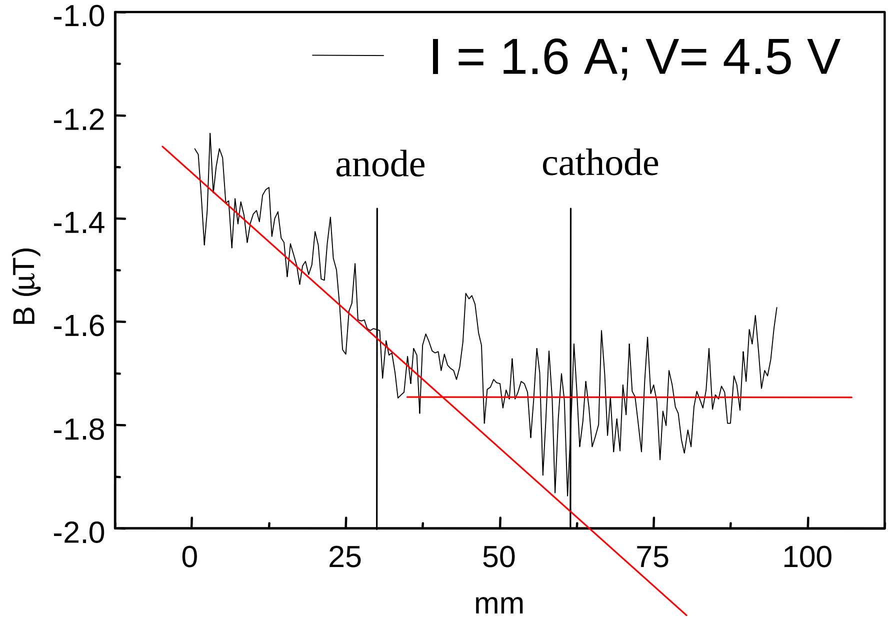
## The difference in background depends on the EP bath self-heating



# Megnetometer scans obtained varying the cell voltage

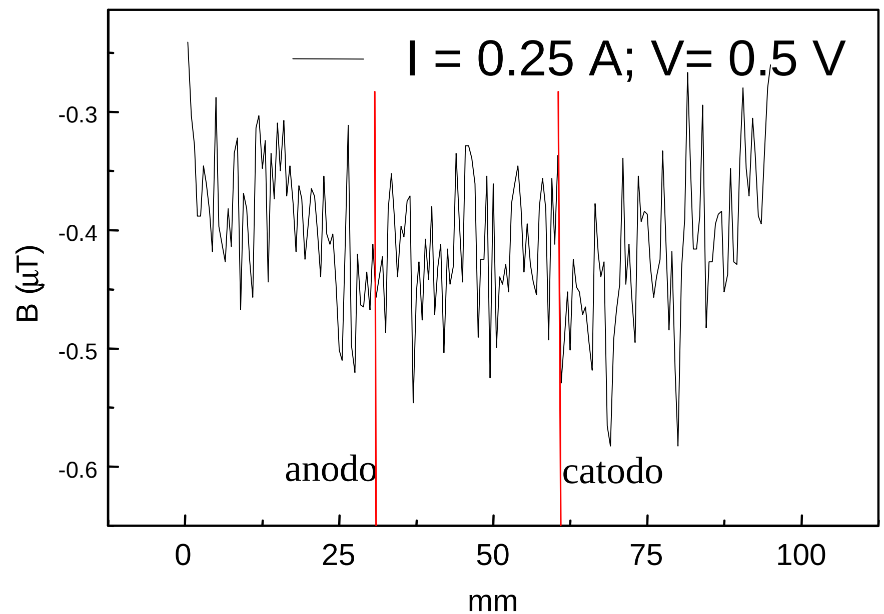


Scan along the cell from anode to cathode just at the working point V = 4.5Volt and I =1.6 A.




Scan along the cell from anode to cathode just at the working point V = 0.25Volt and I = 0.25 A.

But this is a false working point!!!



| Task Name | Milestones | Deliverables |
|---|---|---|
| **WP 6 Materials analysis** | | |
| **6.1 SQUID scanning** | | |
| **6.1.1 Produce calibration defects** | | |
| 6.1.1.1 Production of surface defects | | |
| 6.1.1.2 Production of bulk defects | | |
| **6.1.1.3 Calibration defects finished** | **Report** | |
| **6.1.2 Design components of SQUID scanner** | | |
| 6.1.2.1 Design of the scanning table and support | | |
| 6.1.2.2 Design of the SQUID cooling system | | |
| **6.1.2.3 Design Scanner finished** | **Design report** | |
| **6.1.3 Construction of scanning apparatus** | | |
| 6.1.3.1 Fabrication of the SQUID | | |
| 6.1.3.2 Fabrication and purchase of components for SQUID scanner | | |
| 6.1.3.3 Software for the SQUID scanner | | |
| **6.2 Flux gate magnetometry** | | |
| **6.2.1 Produce calibration defects** | | |
| 6.2.1.1 Production of surface defects | | |
| 6.2.1.2 Production of bulk defects | | |
| **6.2.1.3 Calibration defects finished** | **Report** | |
| **6.2.2 Design components of flux gate head** | | |
| 6.2.2.1 Design electronics | | |
| 6.2.2.2 Design of flux gate head | | |
| 6.2.2.3 Design of operations software | | |
| **MS 6.2.2.4 Design flux gate head finished** | **Design report** | |
| **6.2.3 Fabrication of flux gate detector** | | |
| 6.2.3.1 Fabrication of flux gate head | | |
| 6.2.3.2 Fabrication of mechanics | | |
| 6.2.3.3 Implementation of software | | |
| 6.2.3.4 commissioning of flux gate detector | | |
| 6.2.3.5 Calibration of flux gate detector | | |
| **6.2.3.6 Flux gate detector operational** | **Report, Start operation** | |
| **6.3 DC field emission studies of Nb samples** | | |
| **6.3.1 Quality control scans** | | |
| 6.3.1.1 Modification of Scanning apparatus | | |
| 6.3.1.2 Calibration of Scanning apparatus | | |
| **6.3.1.3 Start scanning activity** | **Start operation** | |
| 6.3.1.4 BCP and HPR samples | | |
| 6.3.1.5 EP and HPR samples | | |
| 6.3.1.6 BCP/EP and DIC samples | | |
| **6.3.1.7 First report on BCP/EP and DIC surface** | **Report** | |