Tosca final meeting

Laser material processing of niobium for the optimisation of cavity resonators

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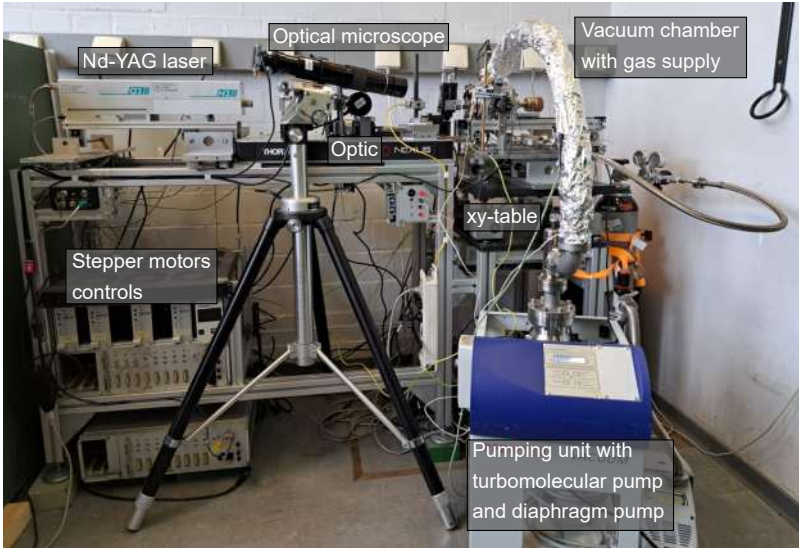
Bergische Universität Wuppertal

Inhalt

1. **Final experimental setup:**
   New optic, new GUI, new vacuum chamber
2. **Investigation of the field emission:**
   Point measurements on a laser-polished fine grain sample
3. **Laser polishing under nitrogen atmospheres:**
   Polishing of niobium foils at various nitrogen partial pressures
4. **Polishing at Fraunhofer Institute:**
   Laser polishing of unpolished niobium samples
5. **Summary and outlook**

Final experimental setup



Nanosecond laser
λ=355 nm, 532 nm, 1064 nm, τ<8ns

xy-Table
Δx≈1µm, Δy≈70 nm

Variable optics
Beam expander, λ/4, energy monitor, motorised focus lens

Vacuum chamber
UHV, gas supply, oil-free

Measurands
pressure, reflected laser energy, optical images of the surface, electrical signals

New GUI

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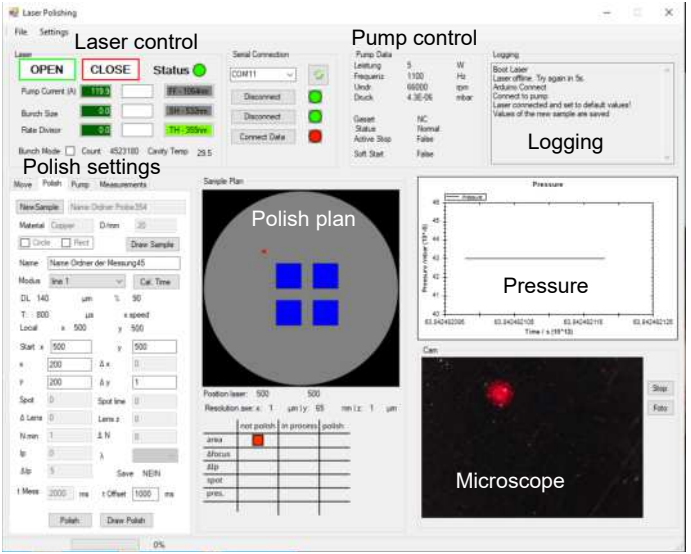
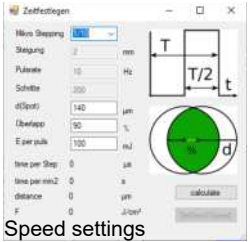




Speed settings

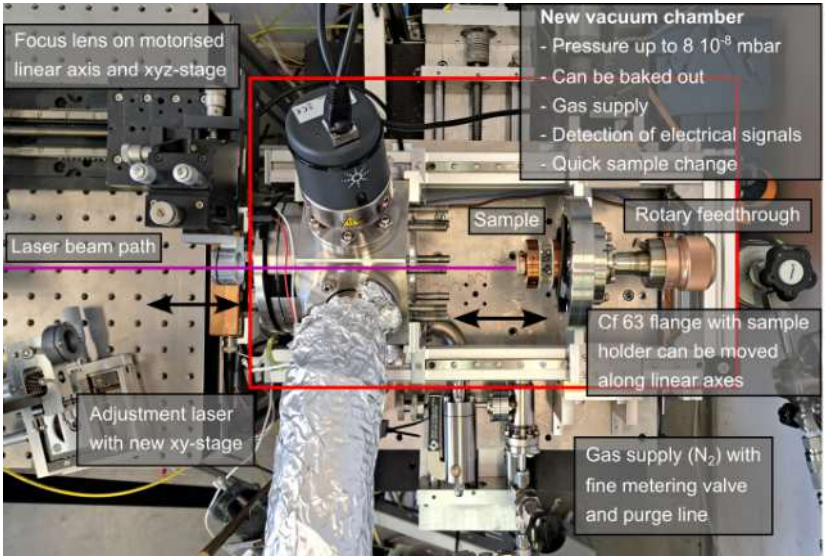
New laser software

- Control of the new pumping station (oil-free)
- Stepper motors are controlled via an Arduino so that the motor speeds can be changed

→ 30 % faster polishing speed, 10 min/mm, limit f=10Hz

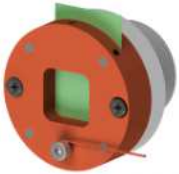
- visualisation of the polished areas

New vacuum chamber



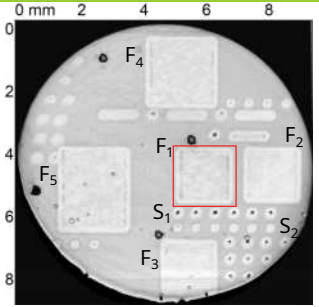
Base of the vacuum chamber

Various sample holders so that solid samples and foils can be polished



Sample holders for foils

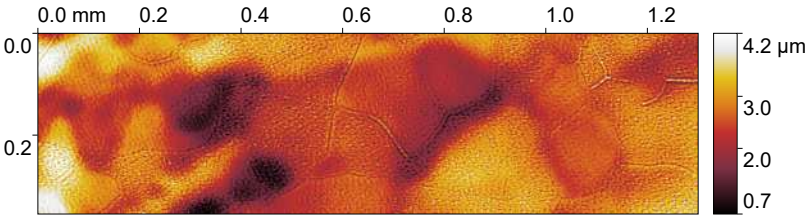
Field emission of fine grain sample

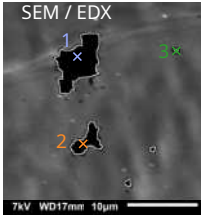


- **Last meeting:** The result of laser polishing differs between different grains (coarse grain, 4 large grains with different (hkl))

⇒ Laser polishing of a **fine grain** sample pre-polished by BCP

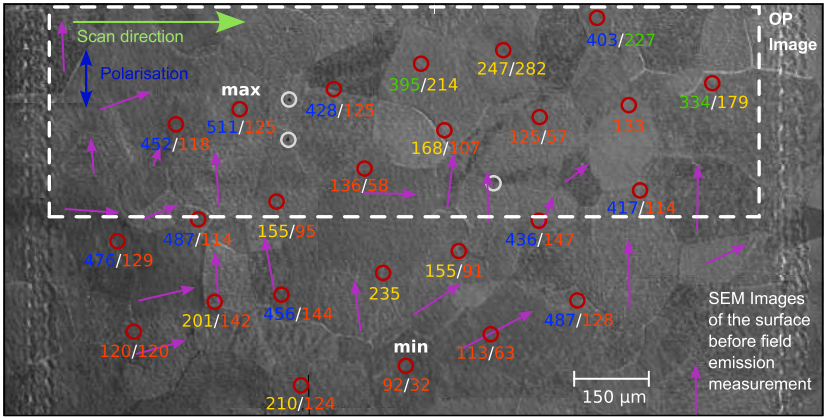
- Surfaces after BCP, $R_a \approx 24\,\text{nm}$
- Micro laser polishing:
  - $\lambda = 355\,\text{nm}$, $\tau < 8\,\text{ns}$, Overlap > 95%
  - P$= 1 \cdot 10^{-6}\,\text{mbar} \Rightarrow$ old vacuum chamber with rotary vane pump

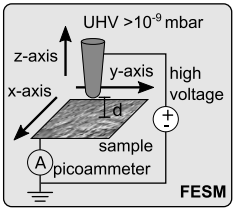




- Differences between the grains + carbon-containing particles (problem oil of the pump)

Field emission of fine grain sample





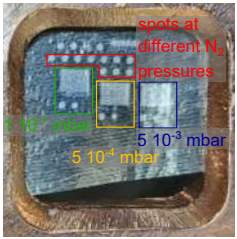
Point of field emission measurment
Carbonaceous particles through LP
Direction of wave structur

Activation field E [MV/m]
very high high medium low
first / second measurement

- 25 measurements, error of E < 10 %, 12 measurements E > 300 MV/m, $E_{max} = 511$ MV/m
- No clear correlation between emission field strength (E) and surface structures

$\Rightarrow$ Surface scans with smaller tungsten tips (current d$\approx$ 250 µm) are necessary

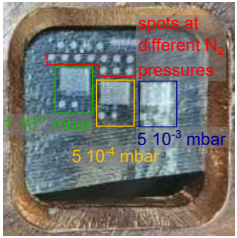
Laser polishing under a nitrogen atmosphere



- Polishing niobium foils (thickness 25 µm and 50 µm) at different nitrogen pressures
- Polished spot at $5 \cdot 10^{-3}$ mbar $\leq P \leq 1 \cdot 10^{-6}$
- Polished surfaces from 1 mm^2 at $1 \cdot 10^{-7}$ mbar, $5 \cdot 10^{-4}$ mbar and $5 \cdot 10^{-3}$ mbar

$\Rightarrow$ EDX and SEM analyze

Laser polishing under a nitrogen atmosphere



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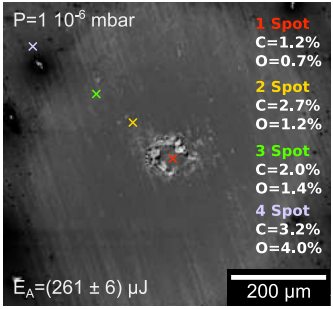
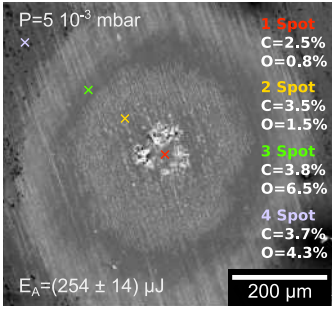
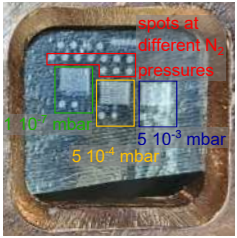




- Increased modification of the surface at higher pressures
- Low pressure $\Rightarrow$ pressure change as a result of the LP $\Delta P \approx 4 \cdot 10^{-6}$ mbar
- Change in the spatial distribution of oxygen and carbon

Laser polishing under a nitrogen atmosphere



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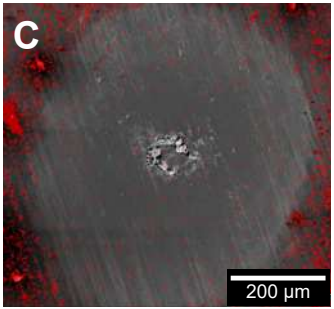

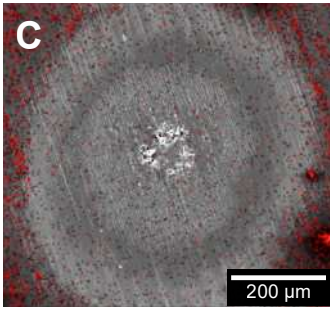

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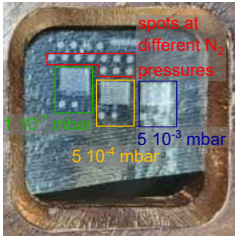

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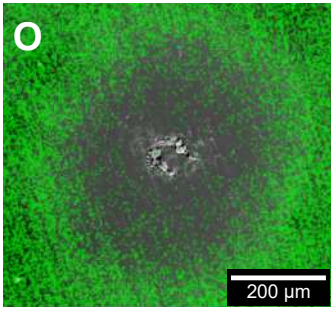

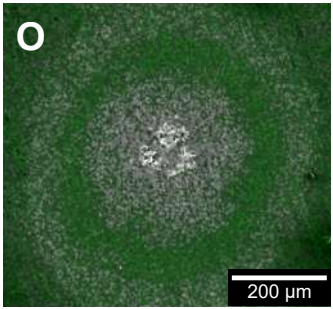

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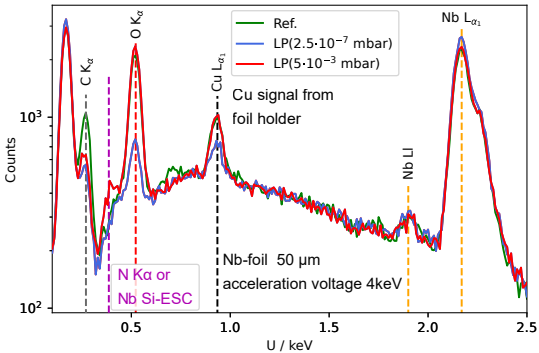
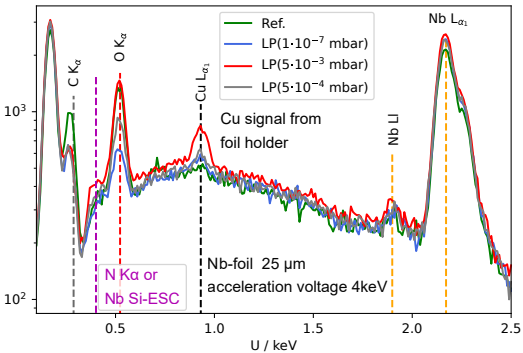
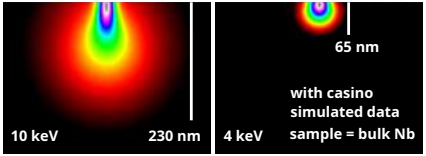
Laser polishing under a nitrogen atmosphere

Areal laser polishing ((1x1) mm^2) of 50 µm and 25 µm thick films at various pressures







Overlap of the lines of N K_α and Nb Si-ESC

$\Rightarrow$ 4 keV: small signal only at $P(N_2) = 5 \cdot 10^{-3}$ mbar

$\Rightarrow$ 10 keV (not shown): all spectra show a pronounced peak

$\Rightarrow$ at 4 keV signal is generated by nitrogen

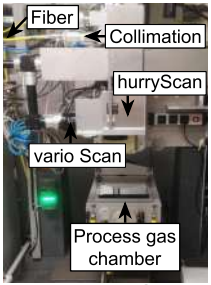
Measurements in cooperation with the Fraunhofer Institute (ILT)

- Limits of our experimental setup
  - Nanosecond laser only enables laser polishing of chemically pre-polished samples
  - Very low polishing speed due to the pulse repetition rate of 10 Hz

? Is the concept of laser polishing scalable for the production of resonators ($\approx m^2$)?

? Can chemical pre-polishing be dispensed with?

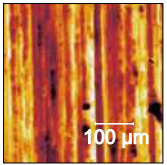
$\Rightarrow$ Polishing of unpolished Nb sheets at the Frauenhofer Institute for Laser Technology



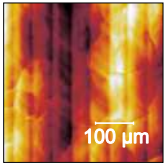
Laser polishing @ILT

- Polish under argon gas with O_2 <100 ppm
- 3-axis cnc + galvanometer scanner switch between cw and pulse laser
- Macropolishing with cw-laser
  - $P_L = (80 - 450)$ W, $d_L = (250,\ 375,\ 500)\,\mu$m, $d_y = (50,\ 75,\ 100)\,\mu$m, $v_s = (25,\ 50,\ 100)$ mm/s, $\lambda = 1080$ nm, n=1,2, 4
- Macropolishing + Micropolishing with pulsed-laser
  - $P_L = (40 - 100)$ W, step 10 W, $d_L = 280\,\mu$m, $d_y = 30\,\mu$m, $v_s = 1000$ mm/s, $\lambda = 1064$ nm, $\tau = (120, 400)$ ns, f=20 kHz

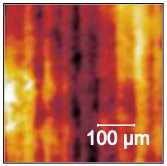
Measurements in cooperation with the Fraunhofer Institute (ILT)



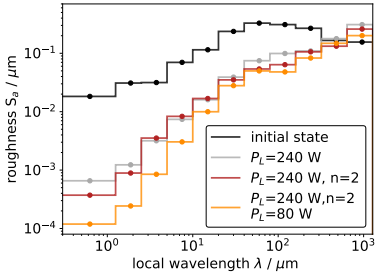
Initial state



P_L=240 W, n=2



P_L=240W, n=2 + P_L=80W



Initial condition of the Nb sheet

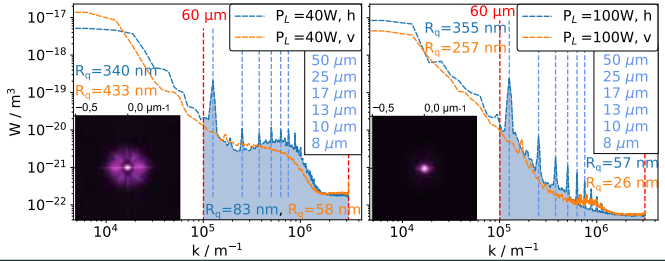
- Fine grain: (85.25 - 86.8) grain per mm^2
- R_a=(0.97-1.43) µm

Result:

Combination of macro and micropolishing significantly reduces the roughness of the samples

BUT:

Closer look: OP + PSDF + 2d-FFT



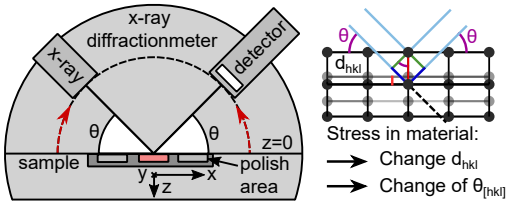
Micropolishing with different P_L

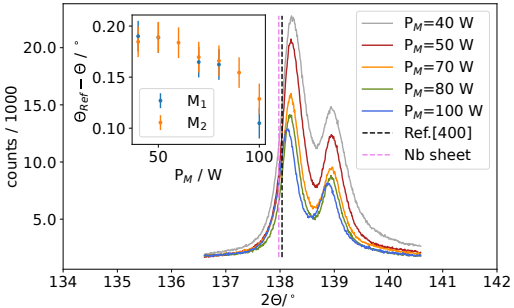
higher P_L, lower R_q

BUT:

More pronounced periodic structures as a result of laser polishing

XRD measurment on laser polished sample

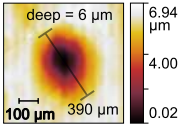


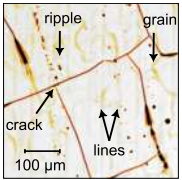


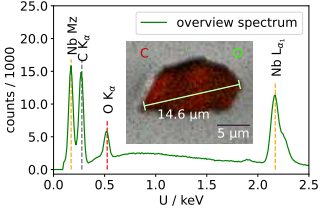
- Surfaces examined with the XRD: first macropolished, then micropolished with different laser powers (P_L)
- XRD measurements to investigate the microscopic stresses
  - ⇒ [400] Peak shifts at maximum as a result of the stresses
- **Macro LP**: Generation of thermal stress even macroscopic deformations of the sample are visible
- **Micro LP**: The higher the P_L, the smaller the shift of the [400] peak

⇒ Laser micropolishing reduces the induced stresses

Problems







- Initial state: Deep holes in surface ⇒ LP cannot remove these holes
  ⇒ Artifact of original structure remains

⇒ Limit of macro laser polishing

- Laser polishing creates characteristic structures
  - Steps between the lines
    ⇒ perhaps reducible by Gaussian profile instead of flat-top
  - Ripple: Artifact of the solidification front of the molten pool
  - Grain: Steps between the grains
  - Cracks: Cracks as a result of the temperature gradient (macro LP)
    - Carbonaceous particles on the laser polished surface + no change in oxygen content (polished in an argon inert gas atmosphere)

⇒ Macro LP possible but many problems exist

⇒ Goal-oriented: Combination of pre-polishing (e.g. BCP) and subsequent micro LP under vacuum

Conclusion

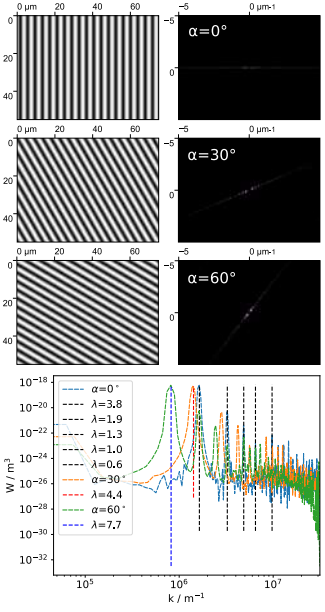
- Extensive optimization of the experimental setup (new GUI, new vacuum chamber, nitrogen supply ..)
- Fine grain sample:
  - Differences between the grains and between the grain boundaries
  - FESM: $E_{max} = 511$ MV/m, but some very low values $\Rightarrow$ No assignment between values and surface texture
  - Two-dimensional scans with a finer tip
- Polishing of niobium foils under different nitrogen pressures $\Rightarrow$ maximum pressure has an effect on the topography produced

  $\Rightarrow$ Presumably detection of nitrogen after laser polishing for $P(N_2) = 5 \cdot 10^{-3}$ mbar
  - Measurements of laser-polished foils under nitrogen atmospheres at DESY, EXAFS
- Cooperation with the Fraunhofer Institute
  - Macropolishing reduces the surface roughness of unpolished Nb sheet metal
    BUT: some problems
  - Micropolishing further reduces roughness and thermal stresses

Questions

Questions?

Power spectral density function



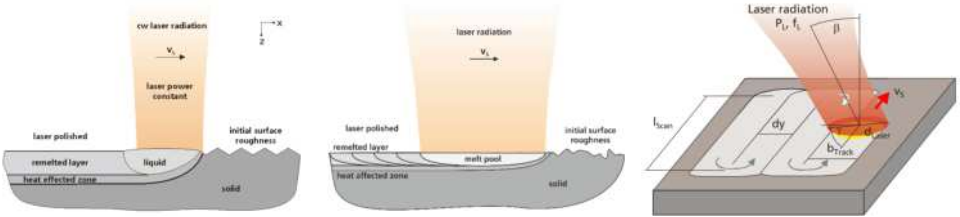
- Power spectral density function
  - $P(\omega) = P(-\omega) = \int_{-\infty}^{\infty} R(\tau_A) \exp(-i\omega\tau_A) d\tau_A.$

  $\Rightarrow$ Peaks in sequence of periodic structures

  $\Rightarrow$ Recording the direction of the PSDF in relation to the periodic structures shifts the peak position
- Frequency-dependent R_q values
  - $R_q^2 = \int_{k_{min}}^{k_{max}} PSDF dk$
- 2d-Fourier transformation
  - $F(k,l) = \sum_{n=0}^{N-1} \sum_{j=0}^{N-1} f(n,j) \exp\left[-2\pi i \left(\frac{kn+lj}{N}\right)\right]$

  $\Rightarrow$ Alignment of periodic structures is recognizable

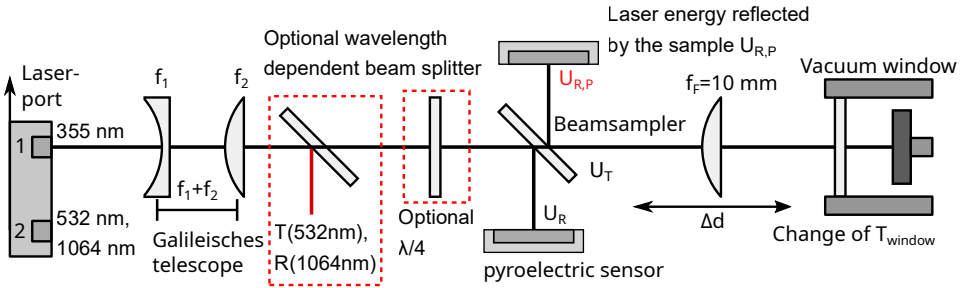
Laser polishing

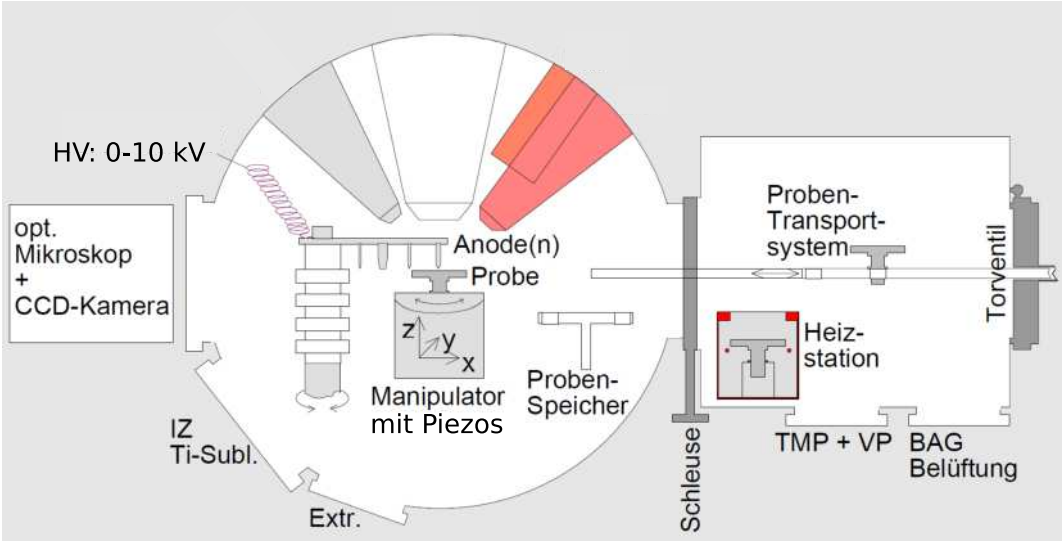


- Macro laser polishing
  - Polishing with a CW laser $\Rightarrow$ Generation of a continuous melt
- Micro laser polishing
  - Surface cools down completely between each pulse $\Rightarrow$ no coherent melt is created
- Parameter
  - Laser: λ, pulse duration (τ) , f, continuous wave (cw) or pulse laser
  - Sample: roughness, grain size and orientation
  - Process parameter: hatch distance (dy), scanning velocity (vs), pressure, beam diamter (dL) number of passes (n), process gas (Argon, vacuum)

Optimization of the experimental setup

- New laser beam path
  - Polarization filter, 532 nm / 1064 nm beam splitter, measurement of reflected laser radiation




HV: 0-10 kV
opt.
Mikroskop
+
CCD-Kamera
Anode(n)
Probe
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y
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Manipulator
mit Piezos
Proben-
Speicher
IZ
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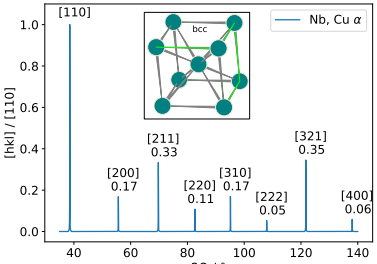
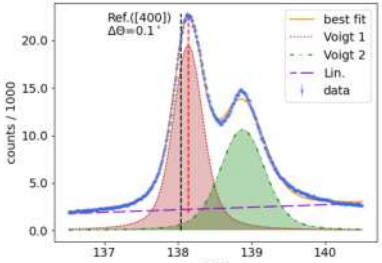
system
Torventil
Heiz-
station
Schleuse
TMP + VP
BAG
Belüftung

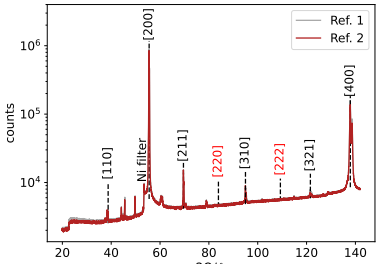
XRD-data



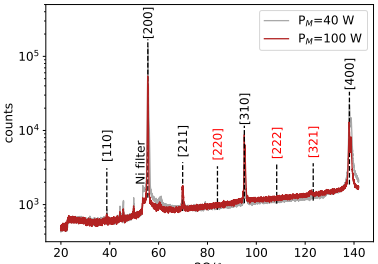
(a)



(b)



(c)



(d)

a Simulated Nb diffractrogram with the expected intensity of the peaks

b Approximation of the [400] peaks by a linear combination of: 2 Voigt functions and a straight line

c Diffractograms of two untreated Nb sheets

d Diffractogram of two laser-polished surfaces