TUBAF
Die Ressourcenuniversität. Seit 1765.

INSTITUT FÜR MASCHINENELEMENTE KONSTRUKTION UND FERTIGUNG
PROFESSUR ADDITIVE FERTIGUNG

# ANALYSIS AND MEASUREMENT OF PEP PARTS

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# Analysis and measurement of PEP

Plasma electrolytic polishing is a material ablating process that affects:

- Parts’ mass
- Parts’ dimensions
- Parts’ surface roughness
- Parts’ gloss

The changes due to the PEP process can be characterised by:

- Weighting parts with sensible enough scale
- Measuring parts with a micrometre
- Characterising parts with optical or tactile surface roughness measurement devices
- Characterising parts with a glossmetre

# Analysis and measurement of PEP

Analysis by human eye:

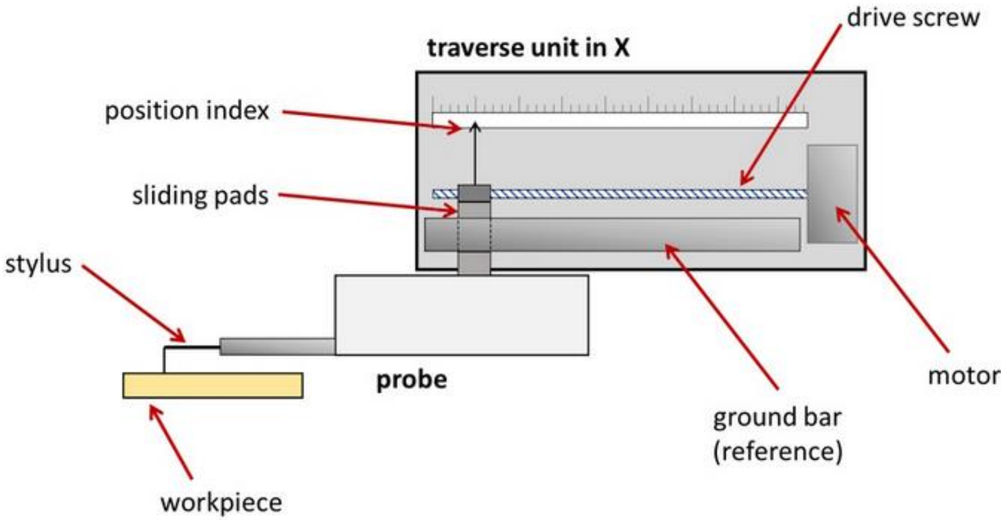
- mistakes (matte shadows, thick oxidlayers)

- with lot of experience people could see differences in set parameters (voltage, temperature, duration, orientation of the part, etc.)

# Surface roughness

Surface roughness can be evaluated using contact and non-contact methods.

Contact measurements are done with profilometers.




Working principle of a stylus profilometer

**Advantages** of contact measurement methods:

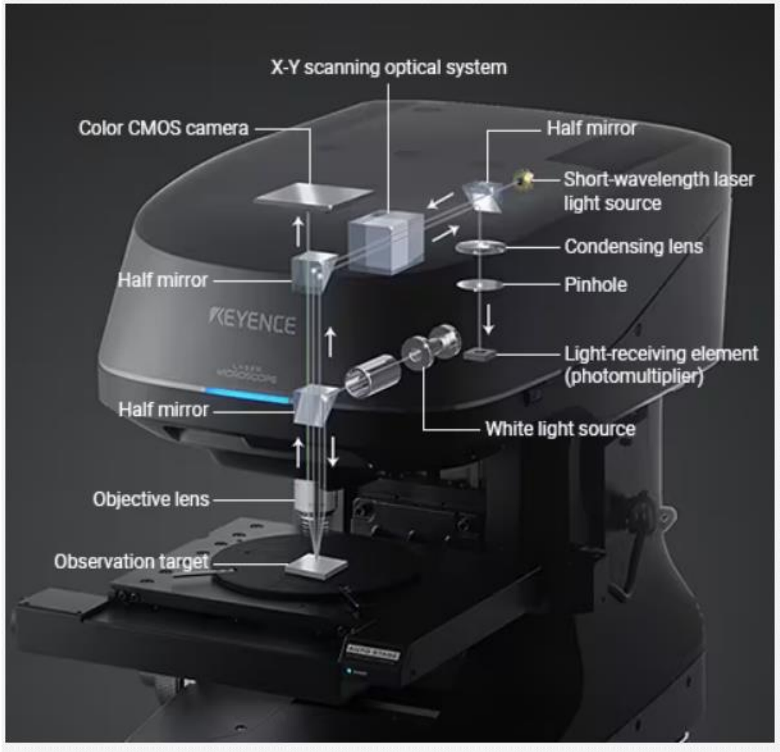
- Relatively simple to set up a measurement
- Results are not influenced by illumination parameters
- Part size is limited

**Disadvantages** of contact measurement methods:

- Parts with complex geometry are difficult to measure
- Only line roughness is available
- There is an upper limit of surface roughness that could be measured without damaging the device

# Surface roughness

Non-contact measurements are done with 3D-profilometers, i.e., laser or confocal light microscopes.




Inside system of a KEYENCE 3D-profilometer

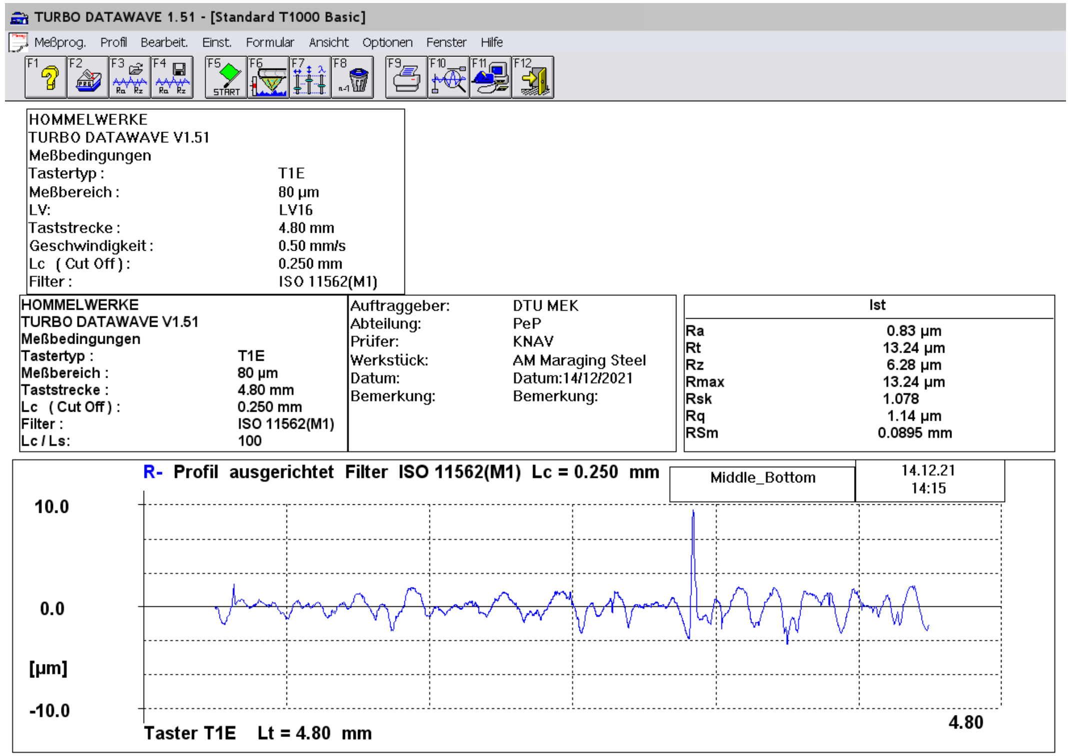
**Advantages** of non-contact measurement methods:

- Complex parts can be measured
- 3D surface evaluation is possible
- Roughness of a surface (not a line) can be evaluated

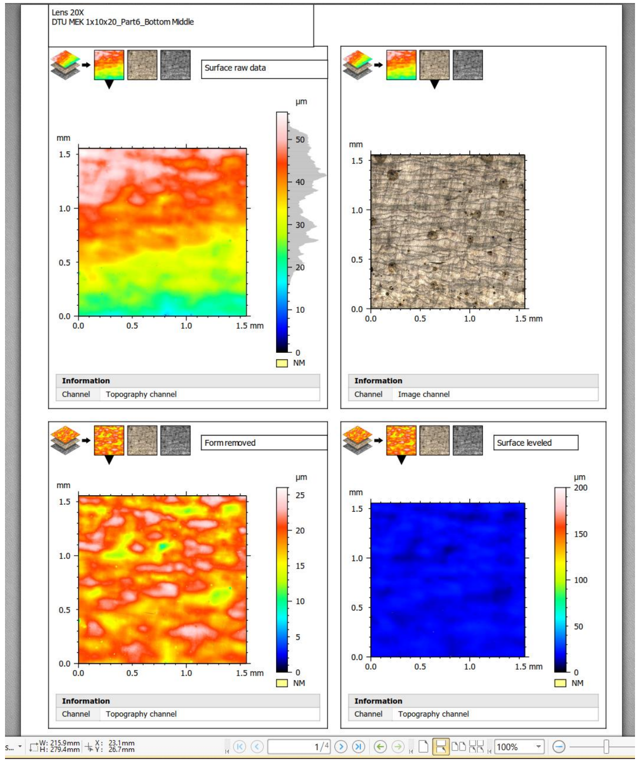
**Disadvantages** of non-contact measurement methods:

- Illumination parameters have an affect on measuring results
- Measurements of additively manufactured parts is still a challenge due to shadows of partly molten particles on a surface

# Surface roughness. Examples

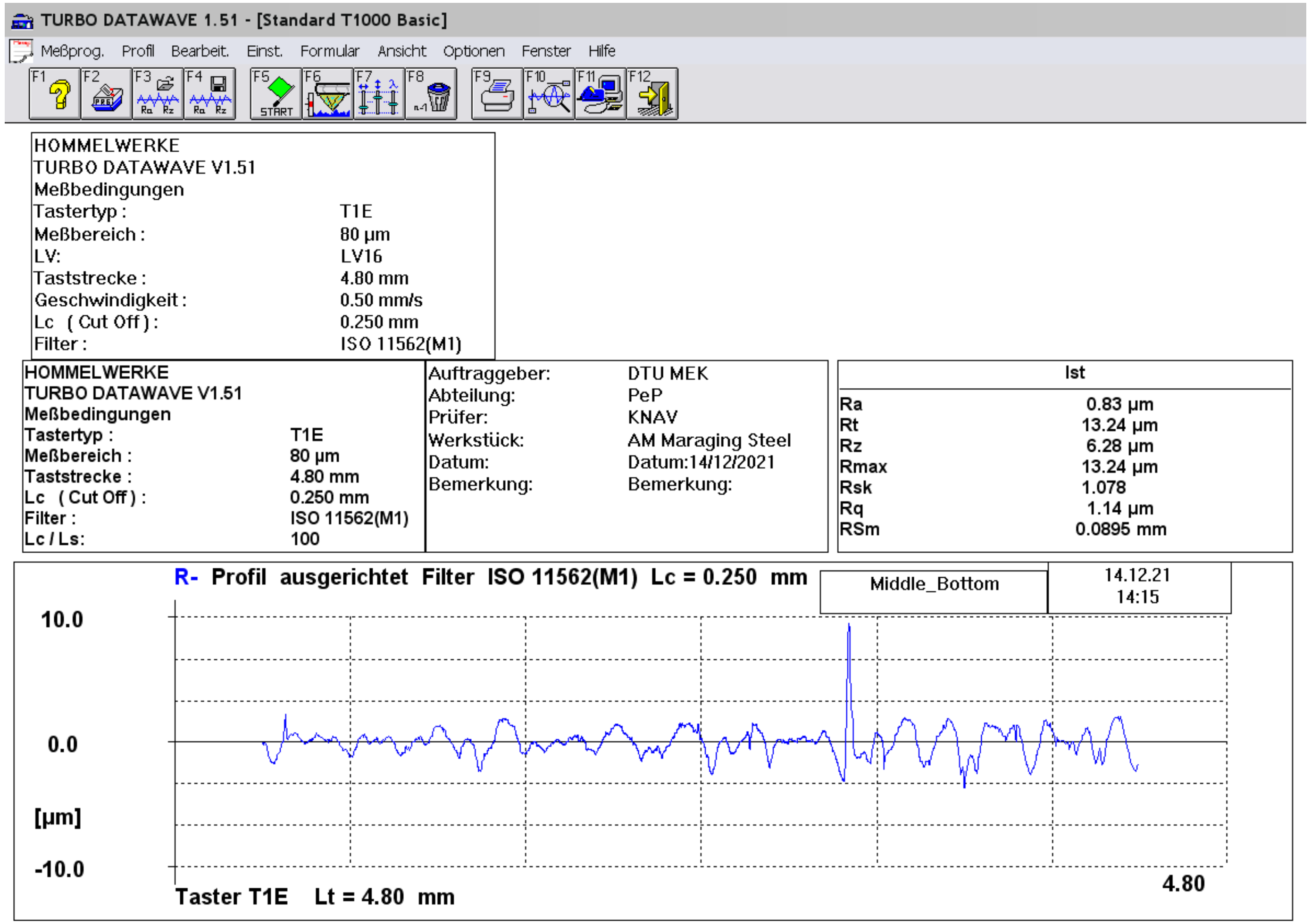



Measurement protocol of a tactile profilometer *TURB*.




A segment of a measurement protocol of a 3D-profilometer *Mahr*.

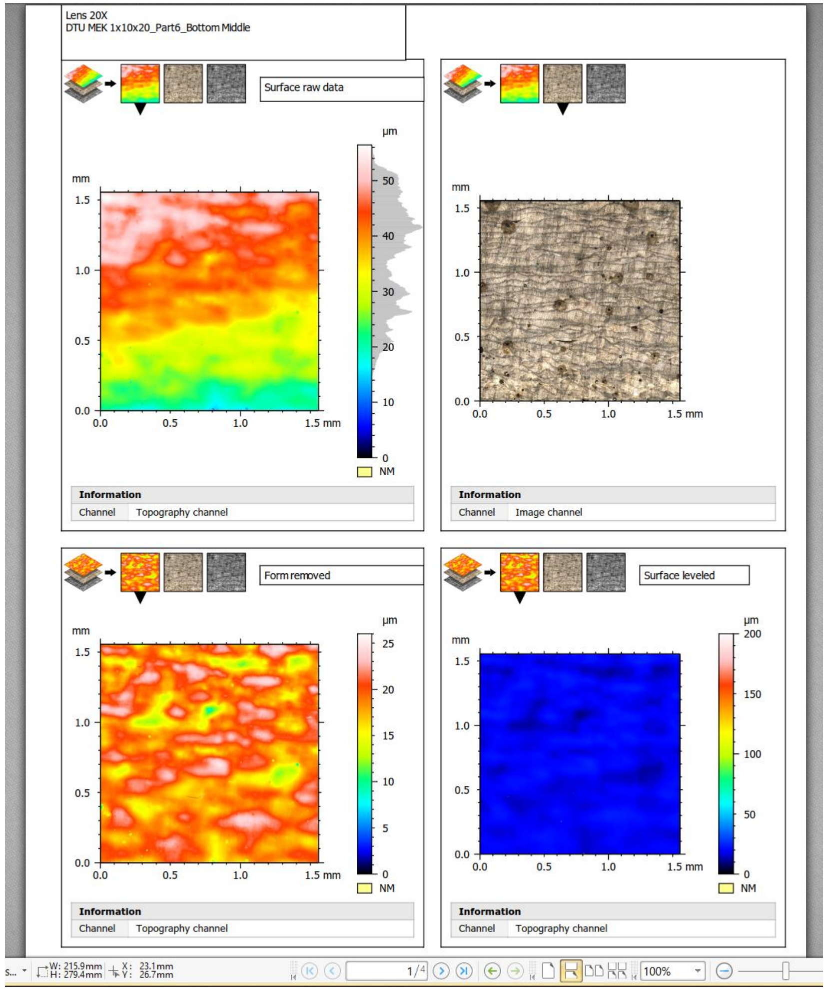
# Surface roughness. Examples in detail




To do **before** starting:

- Asses the surface roughness (gut feeling) to properly choose the measuring head
- Guess the necessary measuring distance, based on probable roughness
- Based on provided table set the measuring parameters like, filter, cut off, measuring distance

# Surface roughness. Examples in detail




To do **before** starting:

- Asses the surface roughness (gut feeling) to properly choose the necessary measuring distance
- Set the required number of images to be stitched so that the right measuring distance would be available

To do **after** measuring:

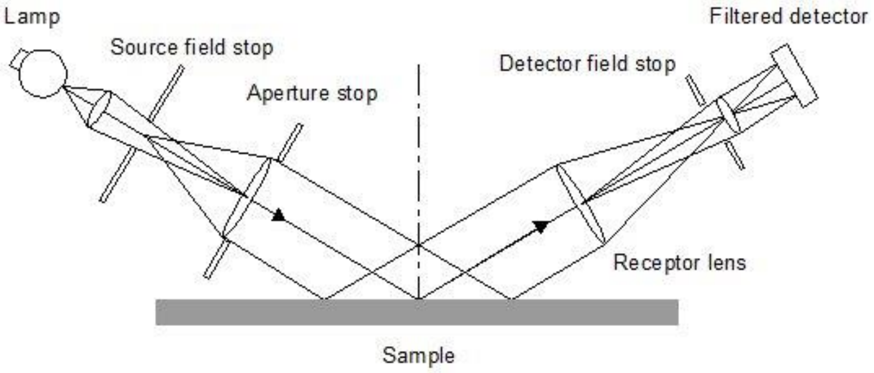
- Set up the measuring protocol with selected filters for removing the part’s form, levelling its’ surface
- Profiles can be extracted for evaluating the line roughness
- Select the right standard for evaluating the surface roughness

# Gloss measurements

Gloss measurements can be done only on a fairly large absolutely flat surface.

Surface must be clean before the measurement is done.


A gloss meter ZGM 1120

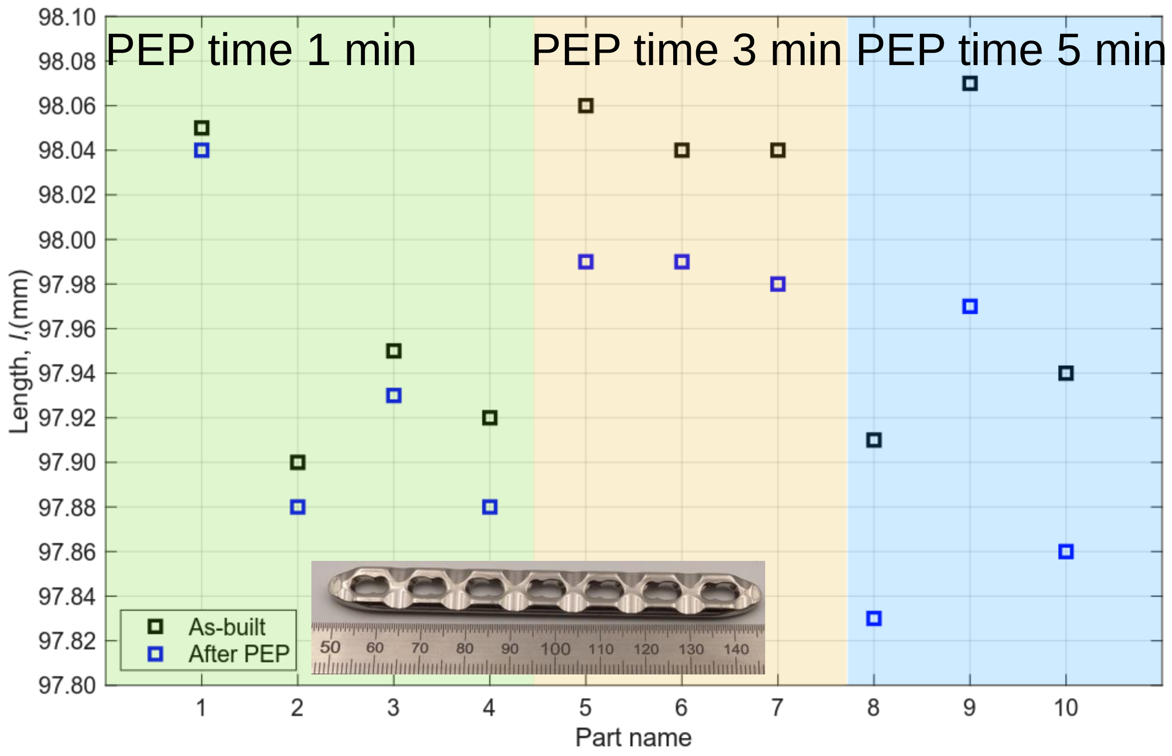



A working principle of a gloss meter

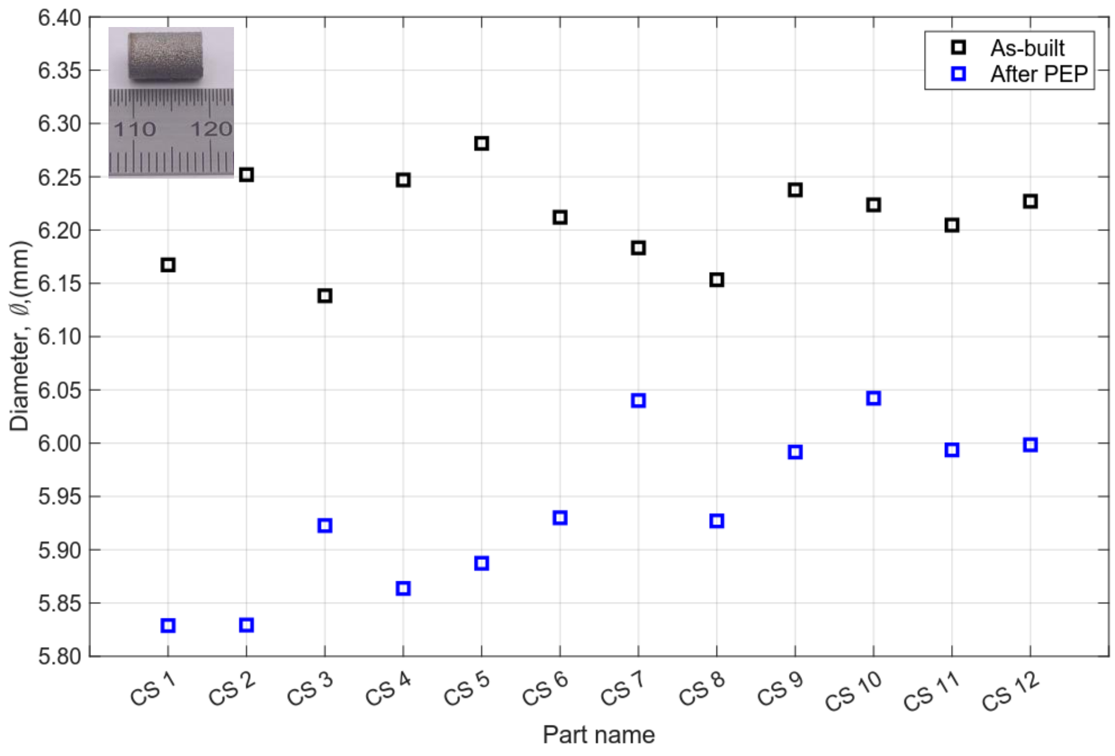
| N° | Date | Time | 20° Value | 60° Value | Group | 20° Upper limit | 20° Reference Value | 20° Lower Limit | 60° Upper Limit | 60° Reference Value | 60° Lower Limit | Sample name | Remark |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 11 | 07.10.2022 | 14:16:19 | 628.2 | 493.8 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 10 | 07.10.2022 | 14:15:49 | 653.3 | 523.2 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 9 | 07.10.2022 | 14:15:21 | 779.5 | 547.3 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 8 | 07.10.2022 | 14:14:55 | 271.9 | 494.5 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 6 | 07.10.2022 | 14:14:14 | 698.8 | 532.1 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 5 | 07.10.2022 | 14:13:48 | 508.8 | 511.2 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 4 | 07.10.2022 | 14:13:19 | 381.6 | 450.5 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 2 | 07.10.2022 | 14:12:33 | 446.9 | 478.7 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |
| 1 | 07.10.2022 | 14:12:04 | 498.4 | 513.5 | <default> | 0 | 0 | 0 | 0 | 0 | 0 | | |

A measurement protocol form a gloss meter ZGM 1120

# Dimensional accuracy, edge rounding, MRR on different positions of the part (CMM)




A length of titanium parts before and after the PEP process.

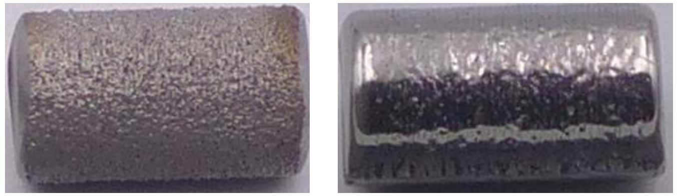



A diameter of austenitic steel parts before and after the PEP process. PEP duration was 30 min.

# Dimensional accuracy, edge rounding, MRR on different positions of the part (CMM)

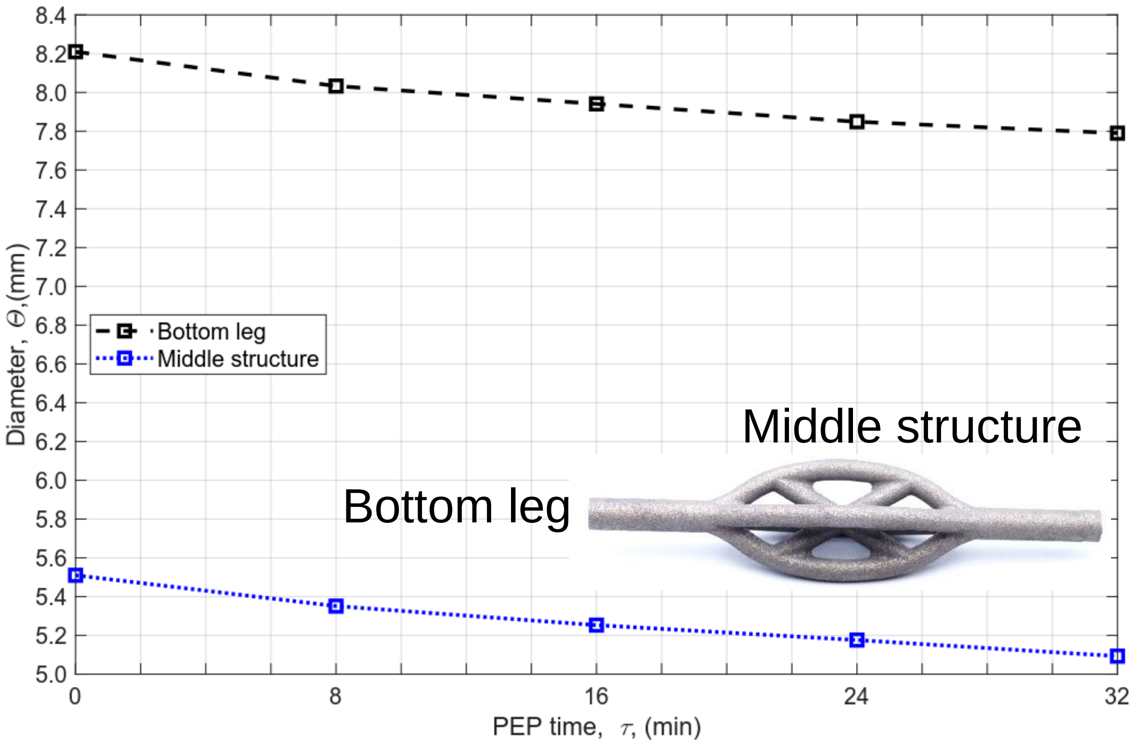
PEP is self-orienting and self-regulating process, targeting the highest peaks on the surface, or sharp edges of a part. Overexposure to the PEP process leads to edge rounding and / or dissolution of fine features.

Additively manufactured parts out of Ti6Al4V before (left) and after (right)



Additively manufactured parts out of CrMnNi before (left) and after (right) PEP

# Dimensional accuracy, edge rounding, MRR on different positions of the part (CMM)




Diameter of a part as a function of the PEP time

- Accurately the dimensional change of a part could be assessed only by using a micrometer with a high resolution (submicron area).
- Without a forced convention, i.e. induced electrolyte stream during the PEP process, the reduction in dimension is of the same magnitude on the outer areas of the part as of the inner structures. Therefore is the material removal rate (MRR) too.
- With induced electrolyte flow a local material ablation on the part can be achieved, ergo the intensity of local MRR could be induced.

# Material removal rate and process efficiency (conductivity, electrolyte temperature and concentration influence on en. used per achieved MRR)

There are many factors that influences material removal intensity:

- *p*H value of the electrolyte. Each material can be efficiently polished only in a certain material specific *p*H range;
- Electrolyte conductivity. For each material there is an optimal electrolyte conductivity window where the PEP process can be carried out;
- Applied voltage. There is an optimal voltage range for each material for efficient polishing, and thus material ablation.

# Material removal rate and process efficiency (conductivity, electrolyte temperature and concentration influence on en. used per achieved MRR)

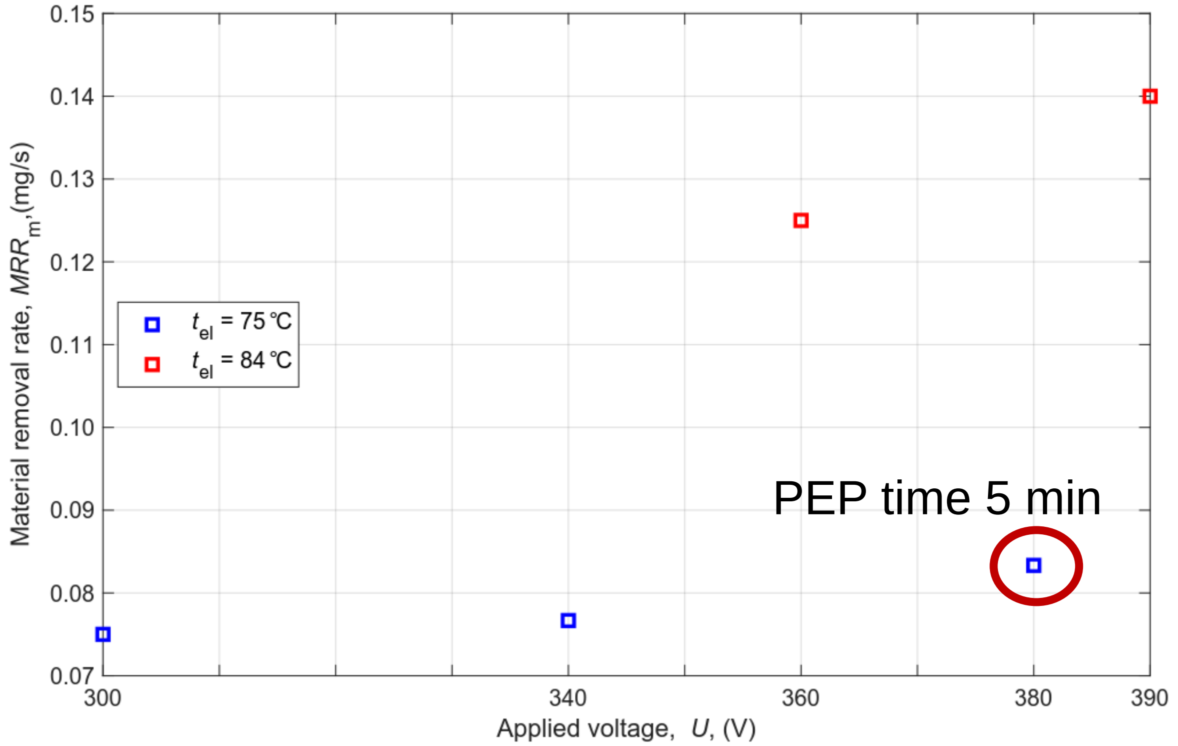
Material removal rate can be calculated based on mass difference or on volumetric change of the part's dimensions.

$$MRR_{m,i} = \frac{m_0 - m_i}{\tau}$$

$$MRR_{V,i} = \frac{\pi l \left(\frac{d_0^2 - d_i^2}{4}\right)}{\tau}$$

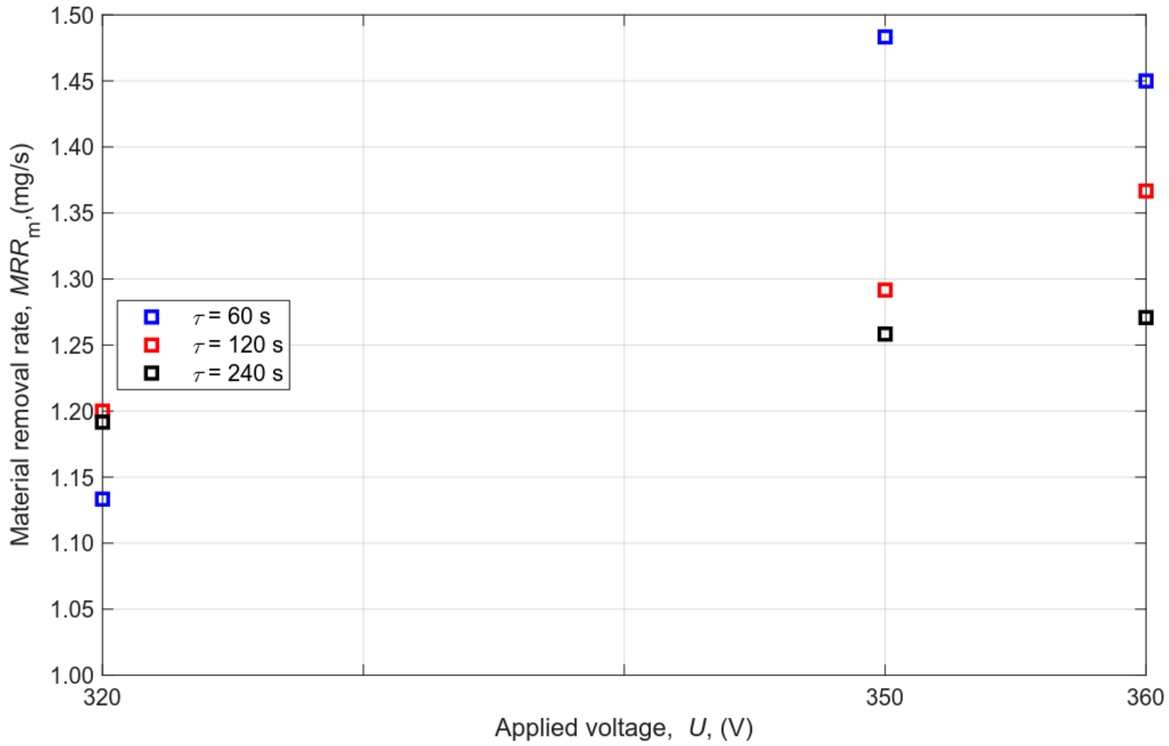
Mass-based *MMR*m. *m* is mass, *τ* is time and subscripts 0 and I refers to before and after, respectively.

Volume-based *MMR*V. *d* is diameter.




*MRR*m of additively manufactured AMZ4 as a function of applied voltage. PEP time 10 minutes.

# Material removal rate and process efficiency (conductivity, electrolyte temperature and concentration influence on en. used per achieved MRR)



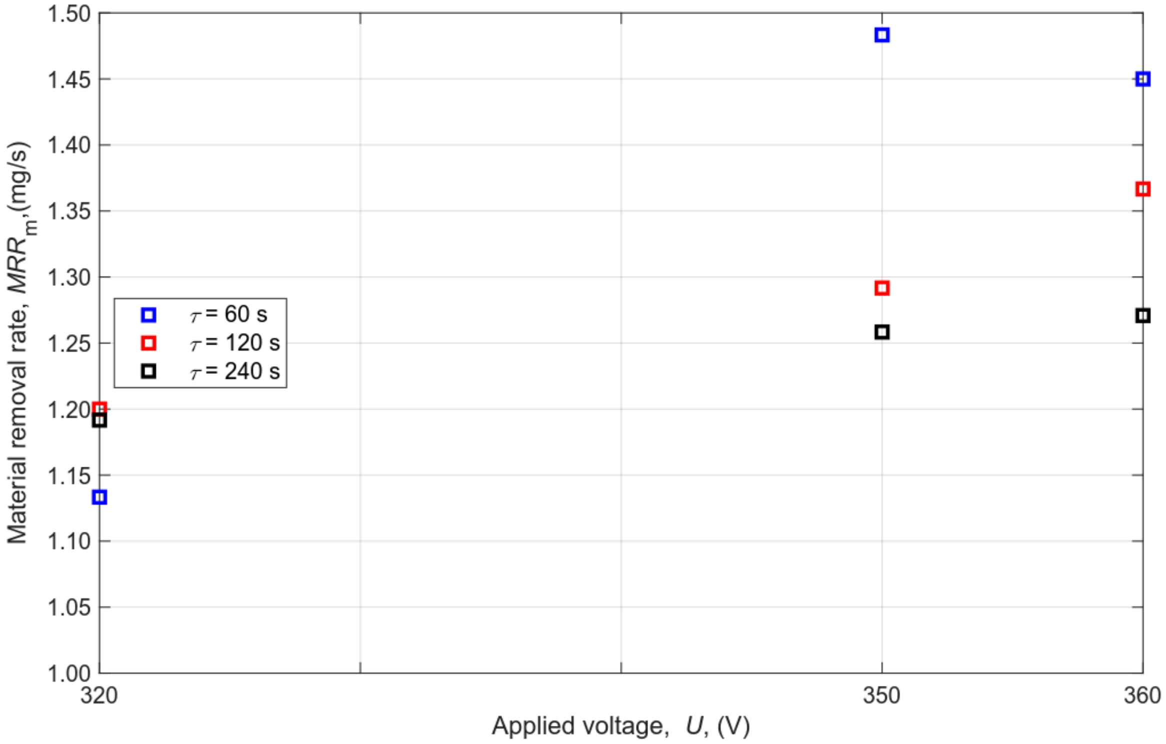

*MRR*m of Nitinol wire with transformation temperature 4.5 °C as a function of electrolyte temperature

The influence of electrolyte temperature on the *MRR* intensity is material specific:

- For AMZ4 with increasing $t_{el}$ $MRR_m$ is increasing;
- For Nitinol with increasing $t_{el}$ $MRR_m$ is decreasing.

! With increasing PEP time $MRR_m$ for Nitinol is decreasing.

# Material removal rate and process efficiency (conductivity, electrolyte temperature and concentration influence on en. used per achieved MRR)

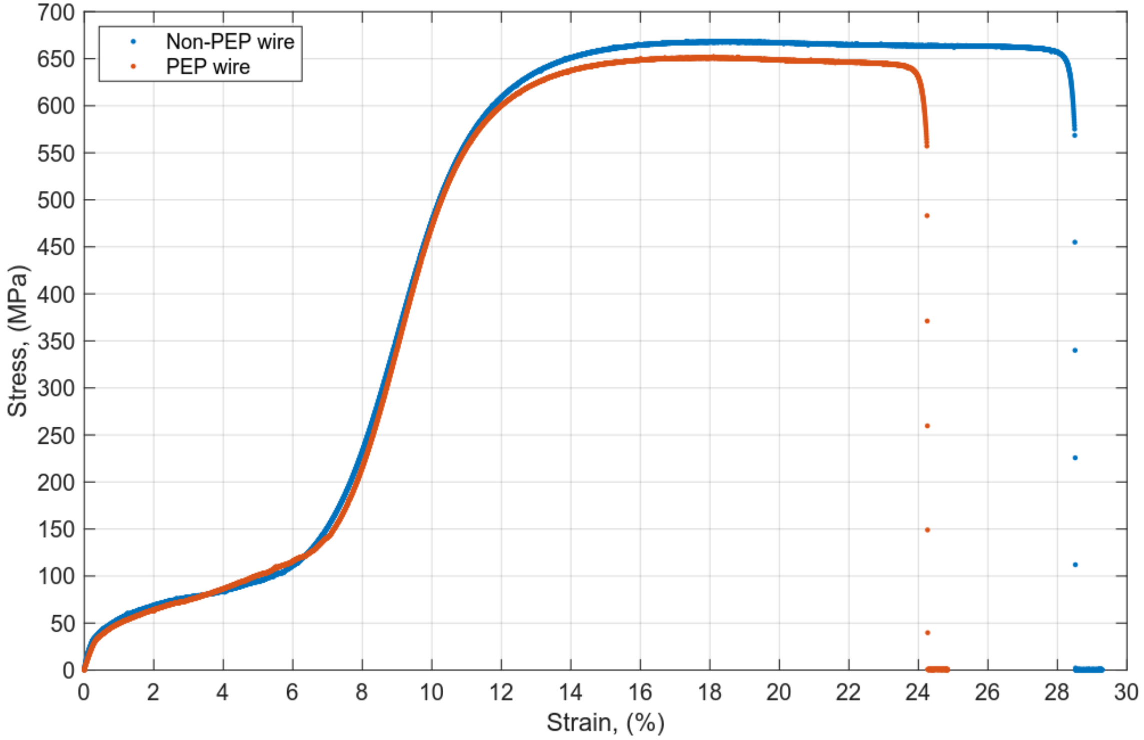



*MRR*m of brass discs as a function of applied voltage

! *MRR*m is influenced by multiple variables that depending on the materials type has varying effect:

- Applied voltage;
- Electrolyte temperature;
- Processing time

# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)

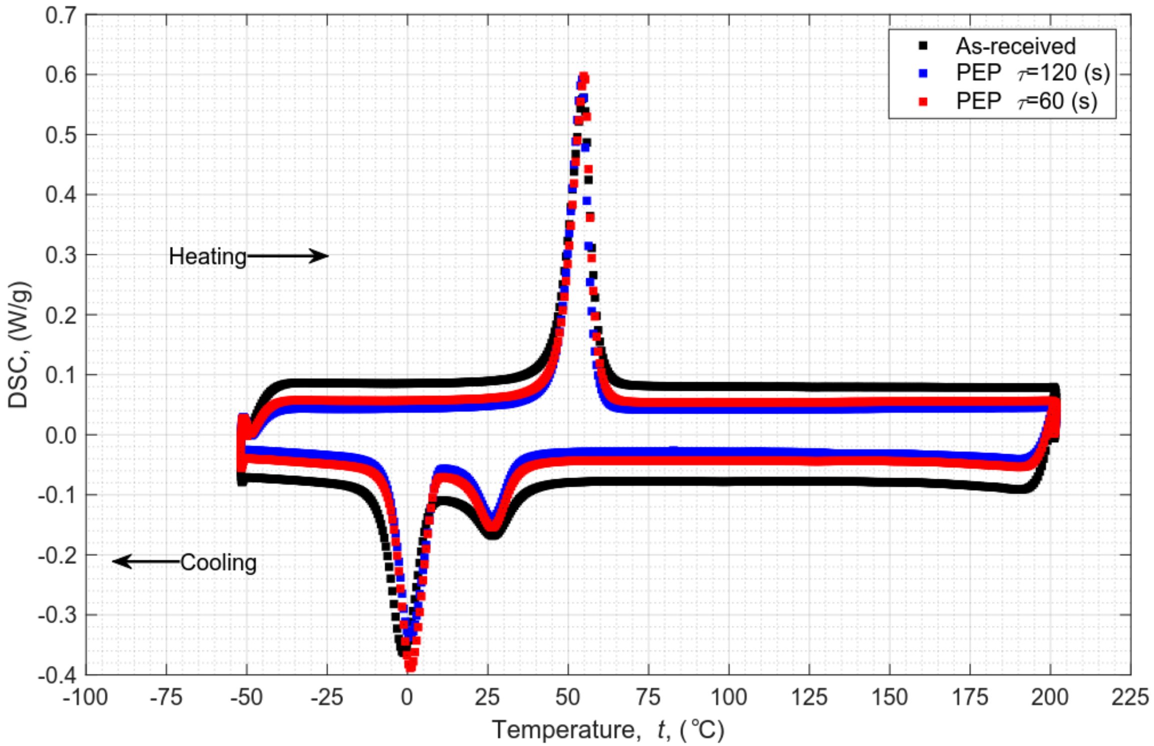



Mechanical test on a Nitinol wire before and after PEP under tensile loading

PEP is a thermal process that might have an effect on various material properties like:

- Mechanical stability;
- Transformation temperature ( relevant for functional materials like Nitinol);
- Microstructure

# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)

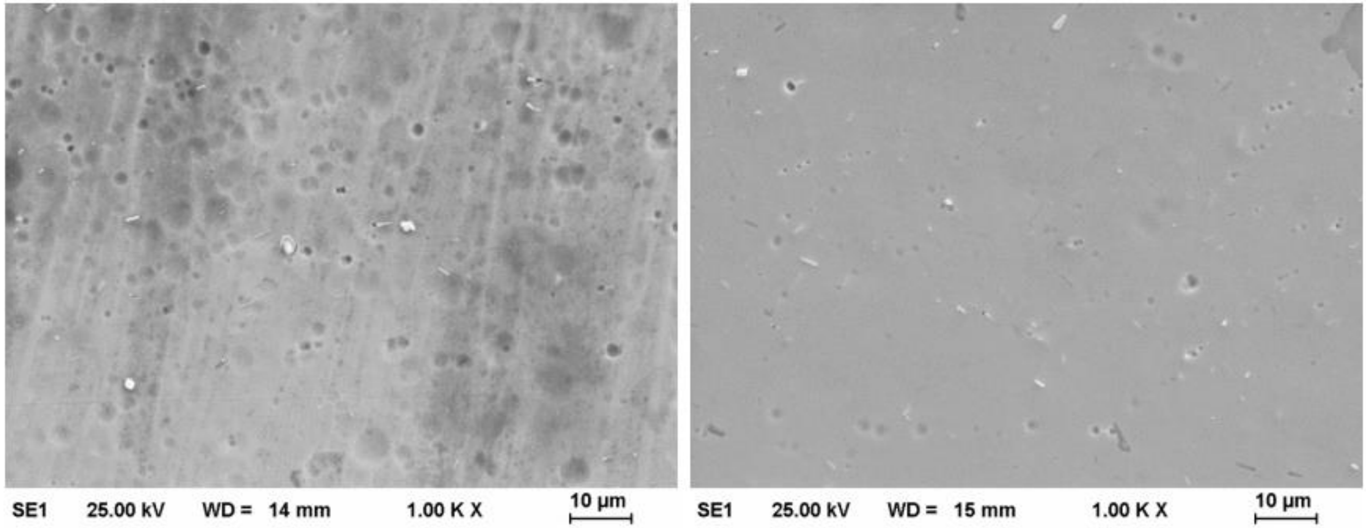



DSC test on a Nitinol wire before and after PEP

PEP shows a minor influence on DSC measurements of Nitinol wire:

- With increasing PEP time the peaks tend to shift towards higher temperatures;
- With increasing PEP time the measured enthalpy slightly decreases.

# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)




SEM pictures of Nitinol plates before (left) and after (right) PEP. The process time 3 min

PEP smoothens the surface, however cannot remove the deep cavities or non-metallic inclusions.

- *Ra* before PEP was 0.15 µm in the region where SEM measurement was taken;
- After 180 s of PEP *Ra* was reduced to 0.09 µm in the same region.

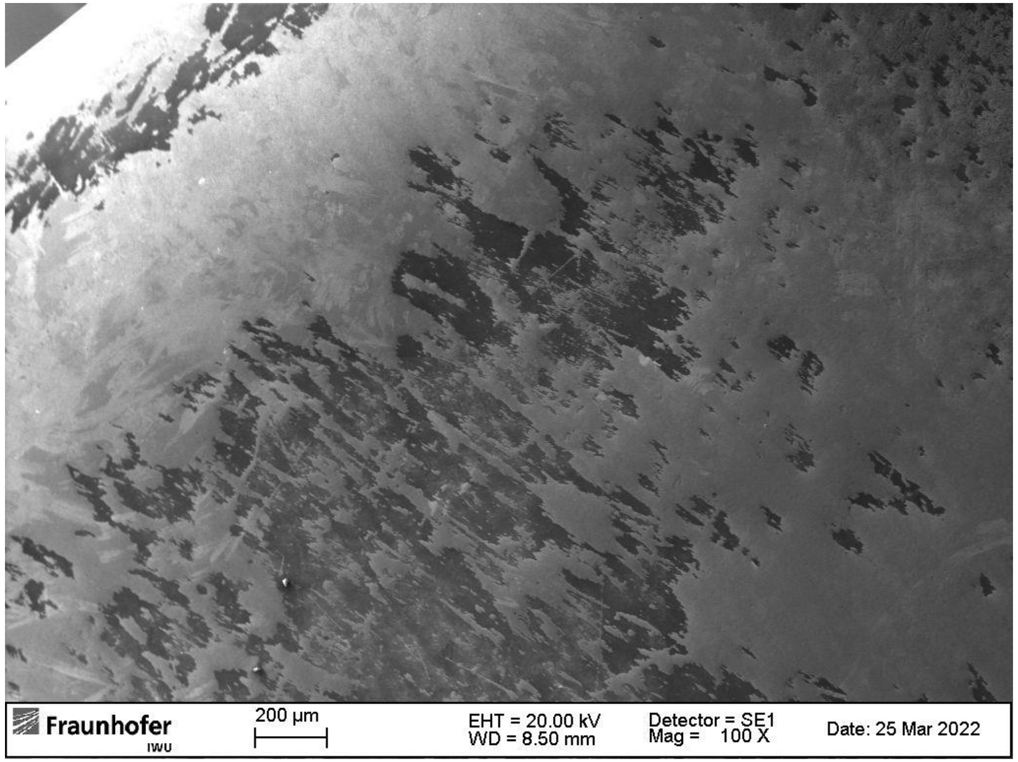
# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)

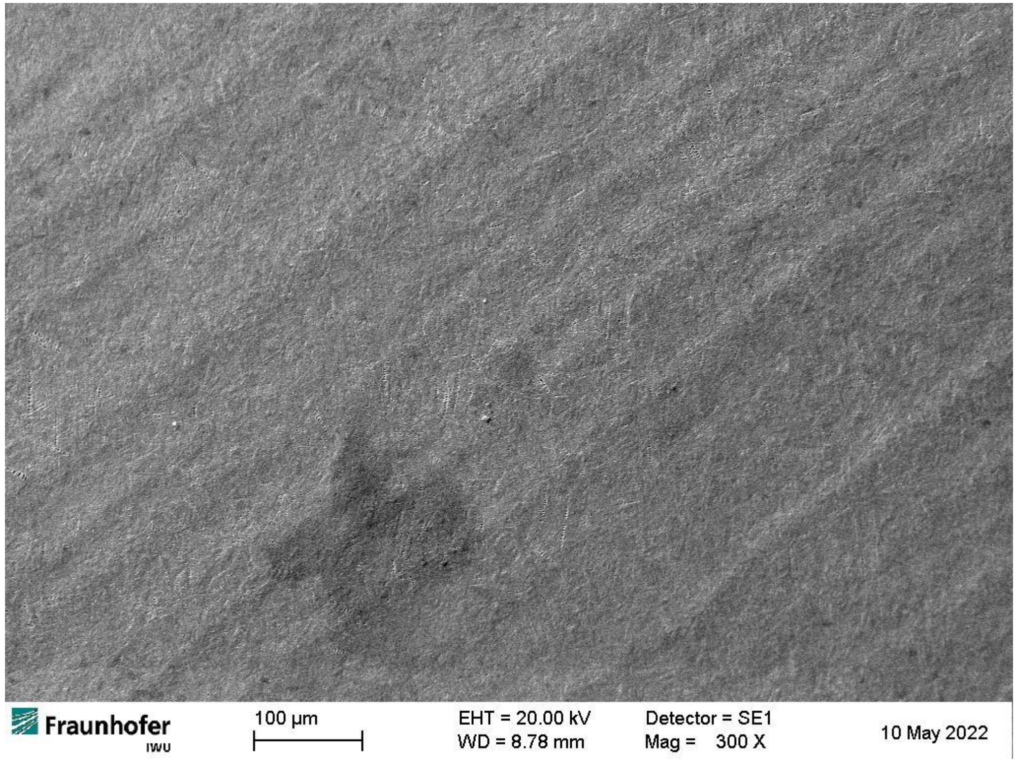
| Sample No. | PEP time, $\tau$, s | Ni in at% | | Ti in at % | |
|---|---|---|---|---|---|
| | | Before PEP | After PEP | Before PEP | After PEP |
| 1 | 10 | 51 | 51 | 49 | 49 |
| 2 | 30 | 52 | 51 | 48 | 49 |
| 3 | 60 | 52 | 51 | 48 | 49 |
| 4 | 120 | 52 | 51 | 48 | 49 |
| 5 | 180 | 52 | 51 | 48 | 49 |

Results of EDX measurements of Nitinol plates before and after various PEP duration

PEP does not affect the chemical composition of a material significantly. The obtained results are in the range of accuracy of the EDX measurements

# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)




SEM pictures of carburised steel gear segment before (left) and after (right) an additional PEP step. The additional PEP process time 6 min

# Microstructure / thermal influence (e.g. referring to SEM / EDS measurements)

| Element | Carburised steel gear segment, w % | | | | | |
|---|---|---|---|---|---|---|
| | Average | Minimum | Maximum | Average | Minimum | Maximum |
| | Before PEP | | | After PEP | | |
| C | 5.41 | 5.1 | 5.89 | 14.58 | 3.81 | 43.73 |
| O | 1.31 | 1.13 | 1.63 | 10.85 | 6.3 | 15.4 |
| Na | - | - | - | 0.18 | 0.14 | 0.22 |
| Mg | - | - | - | 0.43 | 0.11 | 0.74 |
| Al | 0.1 | 0.1 | 0.1 | 1.97 | 0.2 | 6.91 |
| Si | 0.2 | 0.19 | 0.21 | 2.06 | 0.18 | 7.26 |
| P | - | | | 0.27 | 0.27 | 0.27 |
| S | 0.1 | 0.1 | 0.1 | 0.28* | 0.28* | 0.28* |
| Cl | - | - | - | 0.09 | 0.09 | 0.09 |
| K | - | - | - | 0.68 | 0.09 | 1.26 |
| Ca | - | - | - | 0.33 | 0.33 | 0.33 |
| Ti | - | - | - | 0.13 | 0.13 | 0.13 |
| Cr | 0.91 | 0.88 | 0.97 | 0.76 | 0.45 | 0.97 |
| Mn | 0.42 | 0.38 | 0.47 | 0.42 | 0.24 | 0.55 |
| Fe | 88.49 | 87.87 | 89.13 | 71.65 | 45.69 | 90.4 |
| Ni | 2.8 | 2.68 | 2.96 | 2.19 | 1.23 | 3.05 |
| Mo | - | - | - | 0.27 | 0.25 | 0.28 |
| Cu | 0.28 | 0.28 | 0.28 | - | - | - |

EDX results on an additionally polished carburised steel gear segment.

- Previously the segment was polished for 30 min using a plastic fixator.
- The segment was not passivated after PEP, thus corroded.
- After additional 6 min of PEP the corroded spots were removed. The segment was passivated.
- EDX measurements were done on multiple spots.