Institut für Werkstoffkunde
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Present investigation on microstructure and mechanical properties of pseudo NiTi

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Objectives of the present talk

- Pseudo NiTi High Entropy Shape Memory Alloys (HESMAs)

- Microstructure:

  - $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$ in the as-cast & heat-treated state

  ➔ difficulties with orientation determination

- Mechanical properties:

  - hardness and compression tests

  ➔ difficulties with cracks and sample size

- Summary and outlook

High entropy alloys & high entropy shape memory alloys

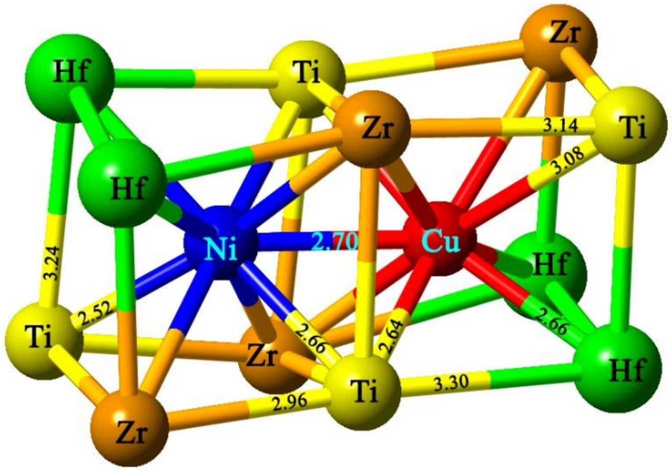
- Material development of $Ni_{50}Ti_{50}$

  (B2-structure)

- Ti-lattice-sides occupied by Ti, Hf, Zr
- Ni-lattice-sides occupied by Co, Ni, Cu

- No major „base“ element ➔ „pseudo“
- „pseudo NiTi“:
  - B2-related triclinic structure

    → lattice distortion



B2-related triclinic P1 phase of HESMA
(Firstov et al., MATEC Web of Conferences 33, 2015)

Transformation temperatures

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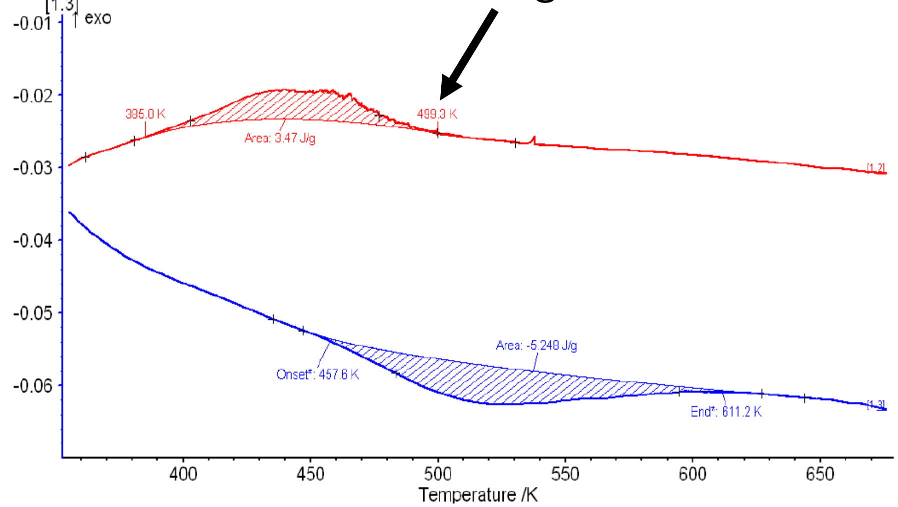
$Ni_{25}\,Cu_{25}Ti_{16.667}\,Zr_{16.667}\,Hf_{16.667}$

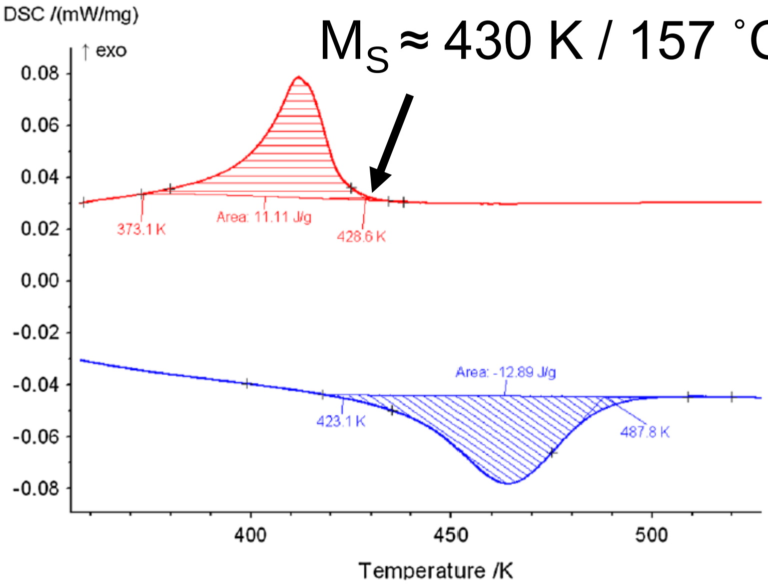
$M_S \approx 500$ K / 227 °C



(Firstov et al., Materials Today: Proceedings 2S, 2015)

$Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

$M_S \approx 430$ K / 157 °C

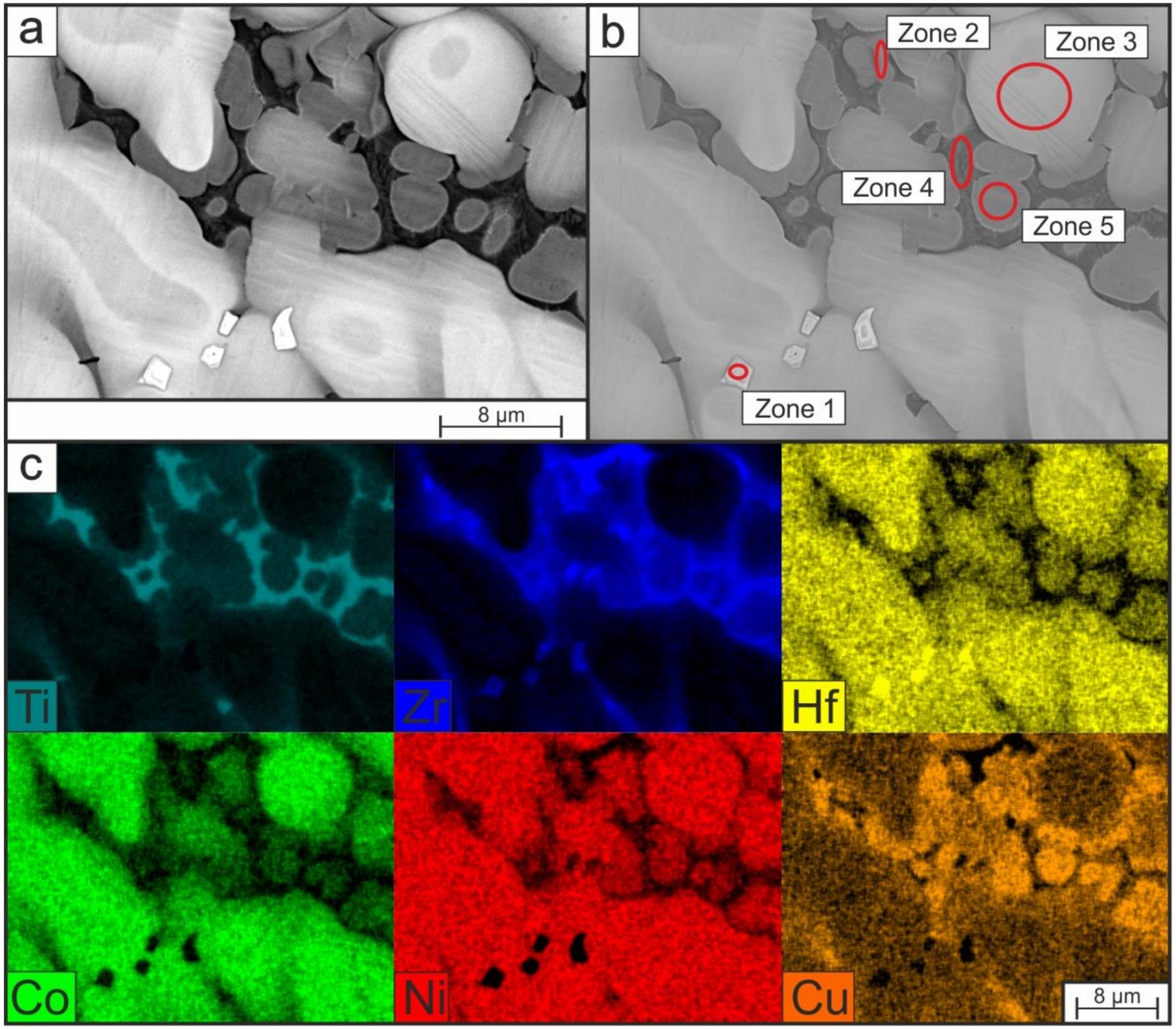


(Firstov et al., Shap. Mem. Superelasticity 1, 2015)

- increase of the martensite start temperature compared to NiTi

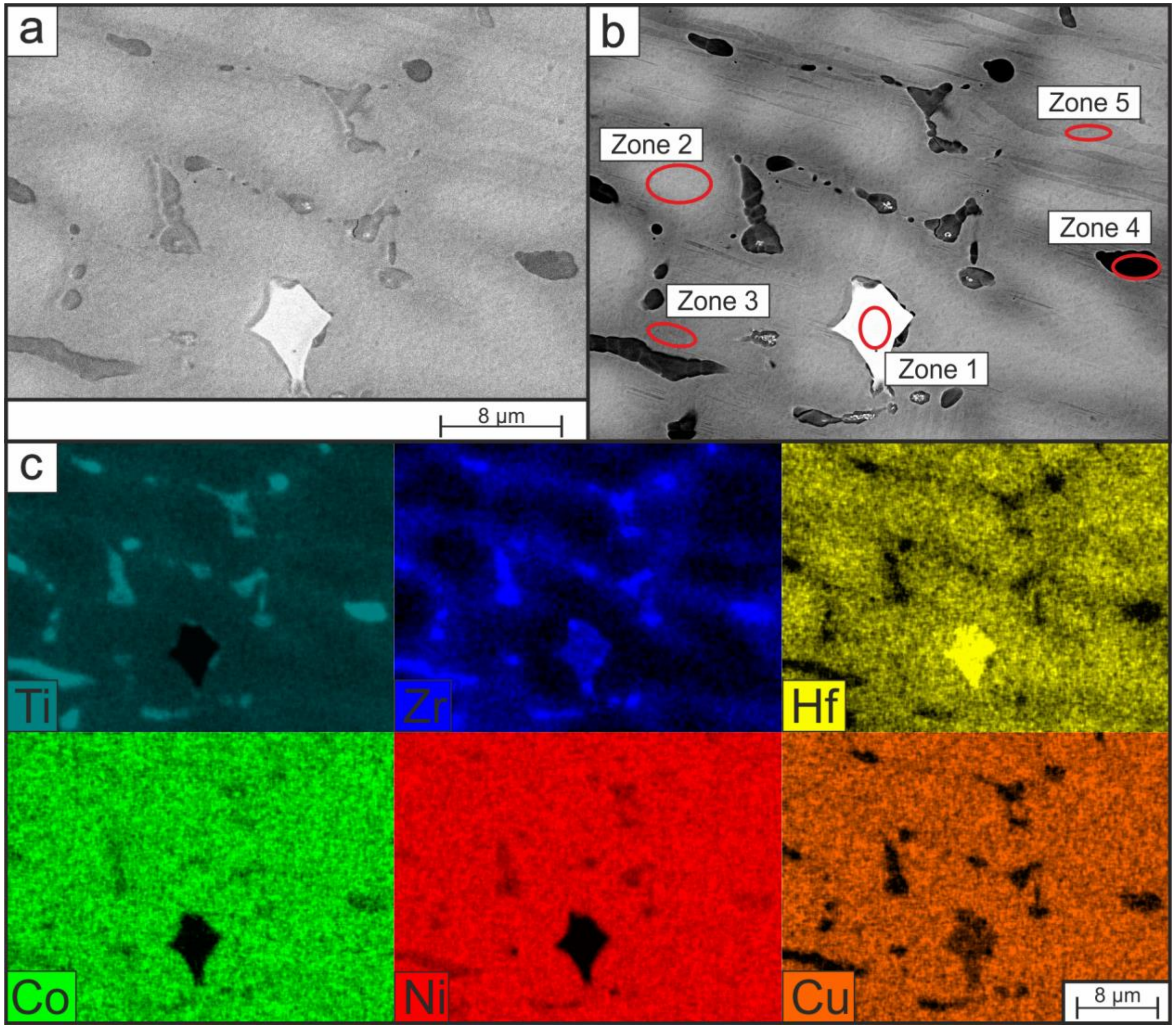
  ➔ 157-227 ºC vs. ~30 ºC

EDX: $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$ as-cast



EDX-mapping as-cast

EDX: $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$ heat-treated

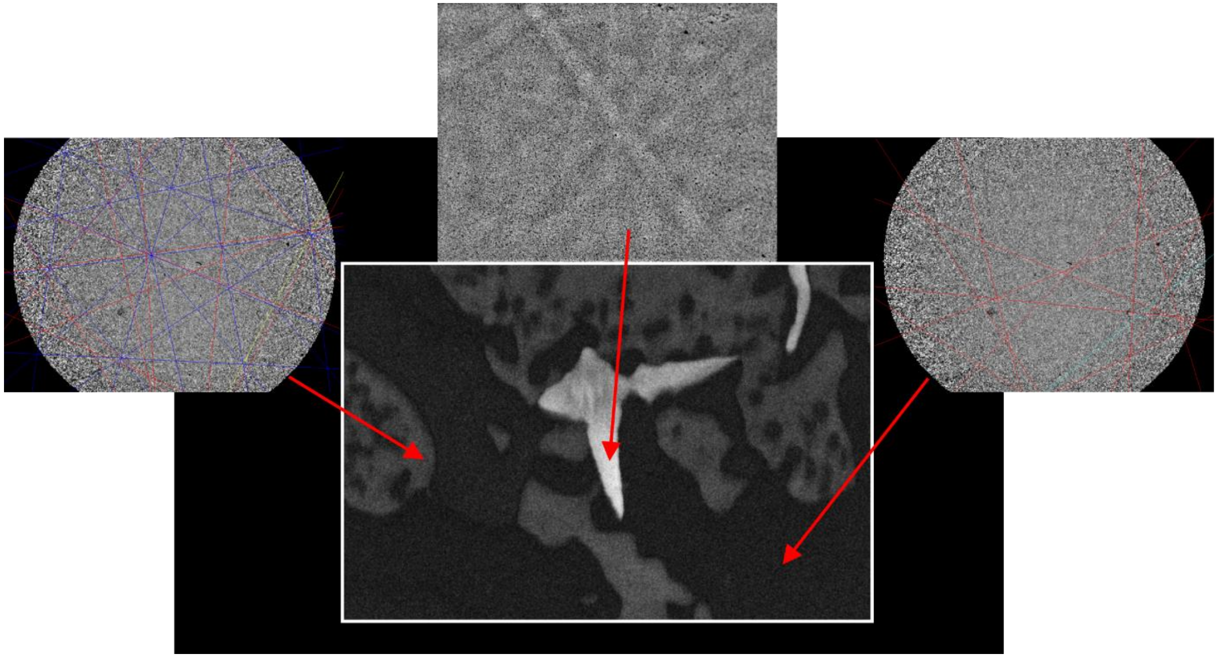


EDX-mapping heat-treated

EBSD: $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

- No pattern in interdendritic region
- Insufficient pattern-quality within dendrits
- Orientation only determinable in the Zr/Hf-precipitations

➔ EBSD-analysis not possible



quality-map and pattern of $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

possible reasons	solution	verified
insufficient preparation	different preparations	yes
surface amorphization	preparation with Focused Ion Beam (FIB)	yes
overlaid structures	TEM-investigation	yes / outlook
lattice distortion	TEM-investigation	yes / outlook

EBSD: $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

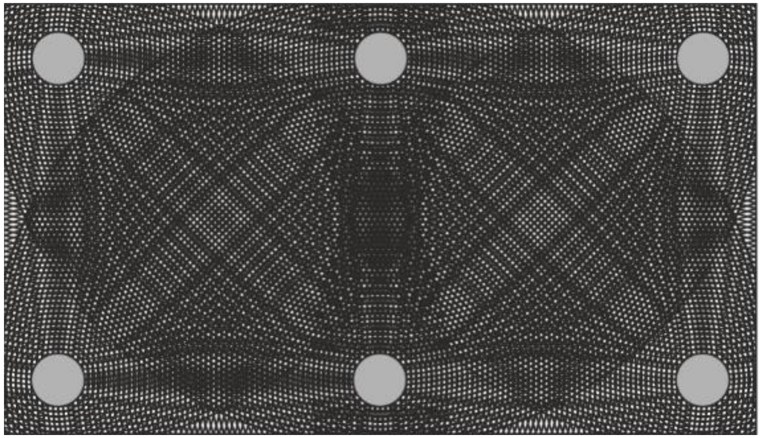
- Electron diffraction ≙ wave diffraction
- Atoms as new wave centers

- **Crystalline (cubic):**
- Ordered structure
- Periodic eliptical interference-bands
- Superposition of the interferences

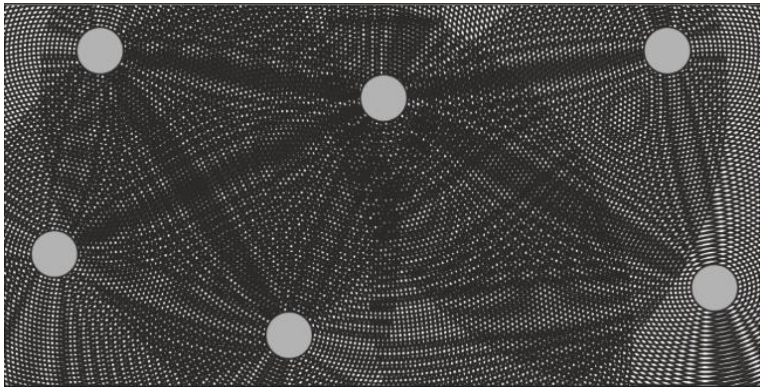
➔ **Kikuchi-pattern**

- **Irregular, distorted:**
- No ordered structure
- No congruent interference-bands
- Superposition doesn´t increase intensity

➔ **no Kikuchi-pattern**



atoms as local point source; cubic



atoms as local point source; distorted

TEM-investigation: $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

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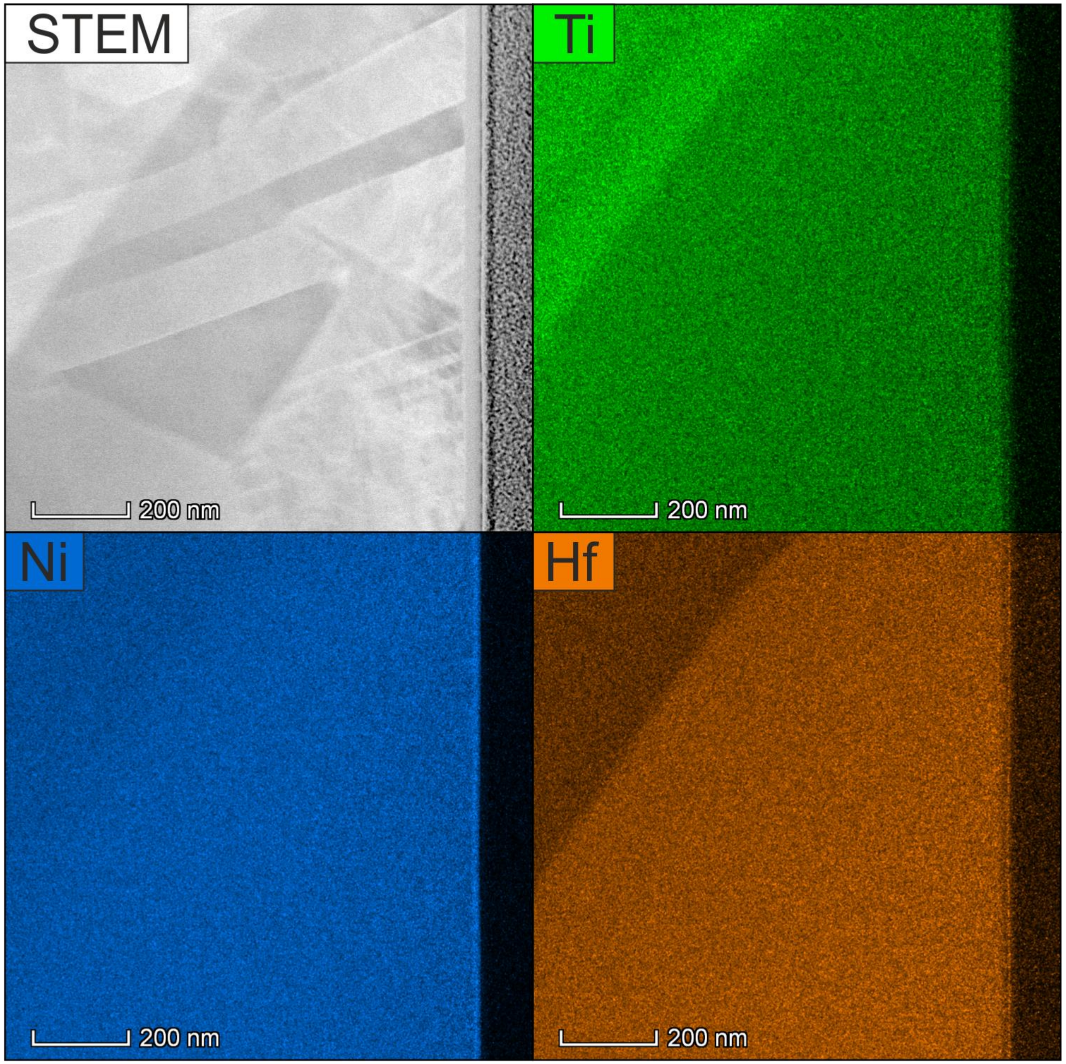
- Overlaid and parallel structures
- Low differences in element concentration
- Clear diffraction-pattern of overlaid structures

➔ Single structures not differentiable



EDX of $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$ lamella

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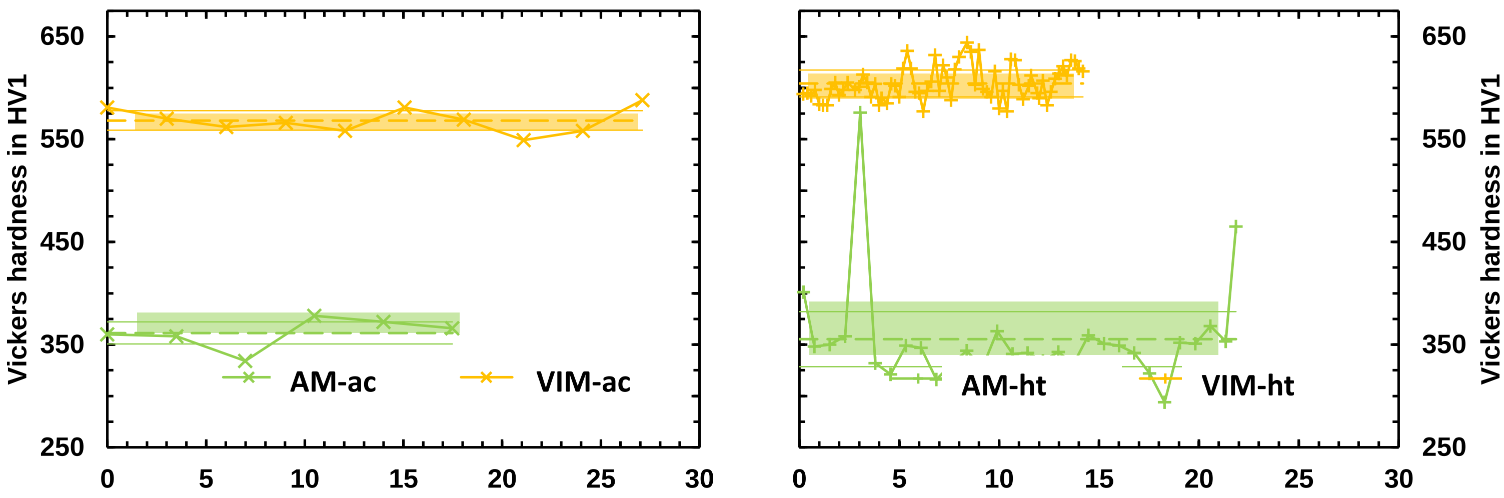
Mechanical testing: Materials & processing

- Material:
  - $Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$
  - $Ni_{25}Cu_{25}Ti_{16.667}Zr_{16.667}Hf_{16.667}$

- Different manufacturing:
  - Vacuum induction melting (VIM)
  - Arc melting (AM)

- Available tests:
  - Hardness tests
  - Compression & compression fatigue test

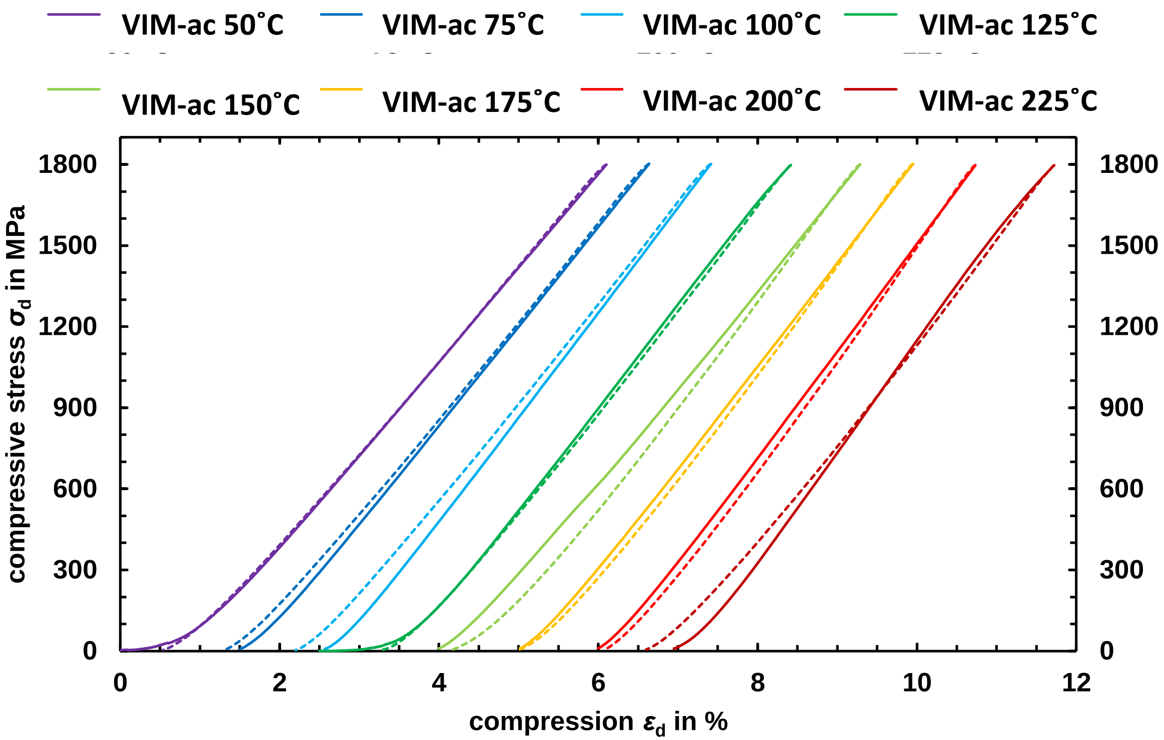
Comparison VIM – AM: hardness



- VIM shows a greater Vickers hardness

- Heat treatment doesn´t signifcantly increase the Vickers hardness of AM produced samples

VIM : loading test

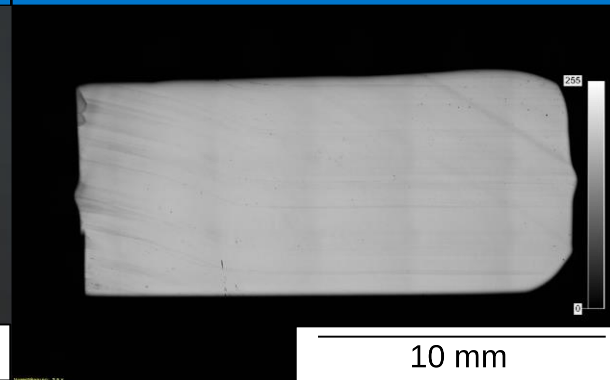


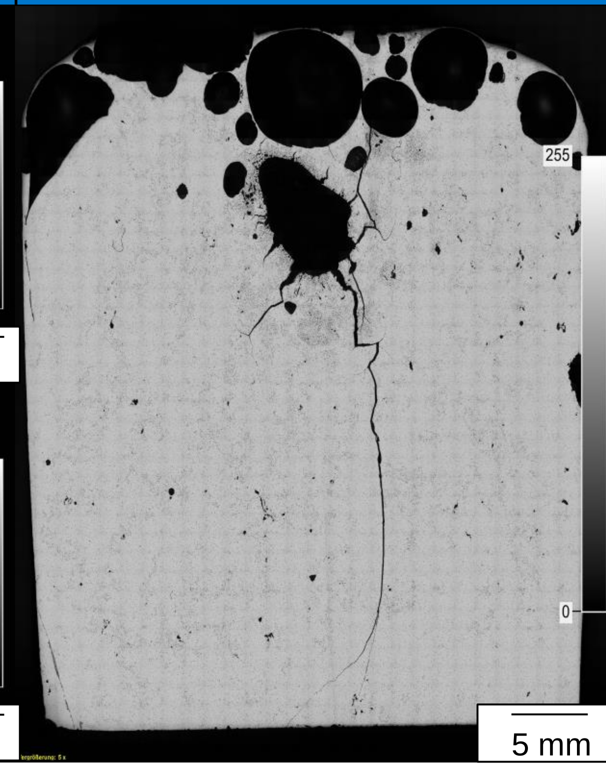
- Curves imply a superelasticity at higher temperatures

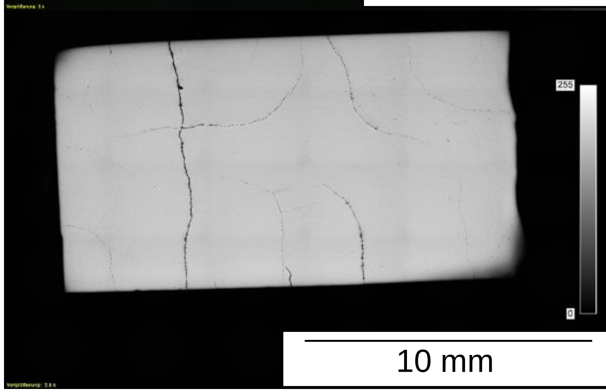
- Compressive strength: 2000 - 2500 Mpa
- Sample size: Ø 2 mm x 4 mm

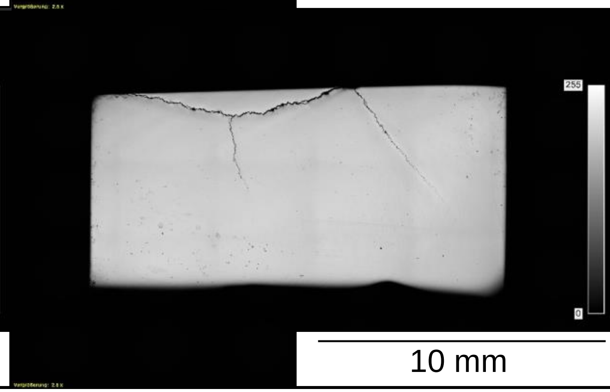
Mechanical testing materials

$Ni_{25}Cu_{25}Ti_{16.667}Zr_{16.667}Hf_{16.667}$	$Ni_{25}Cu_{15}Co_{10}Ti_{16.667}Zr_{16.667}Hf_{16.667}$	(VIM)









Heattreatment of $Ti_{16.667}Zr_{16.667}Hf_{16.667}Ni_{25}Cu_{15}Co_{10}$

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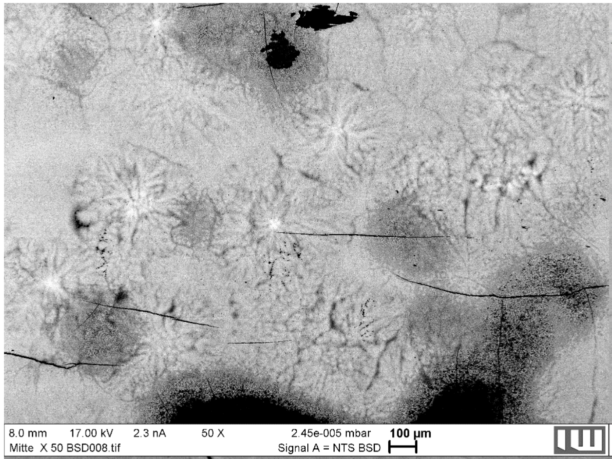
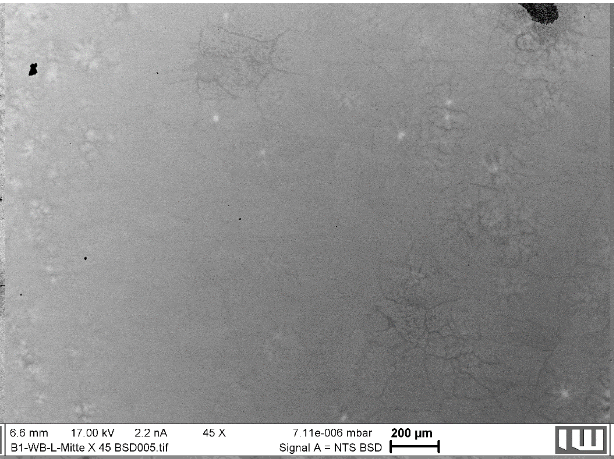
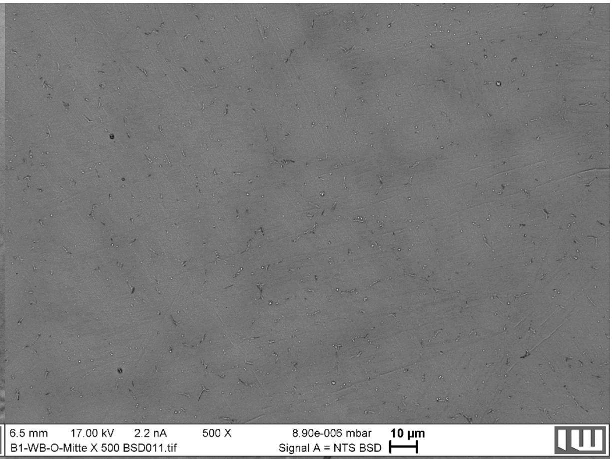
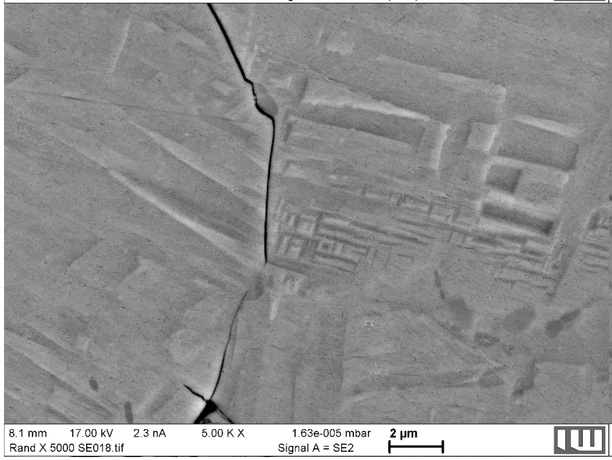
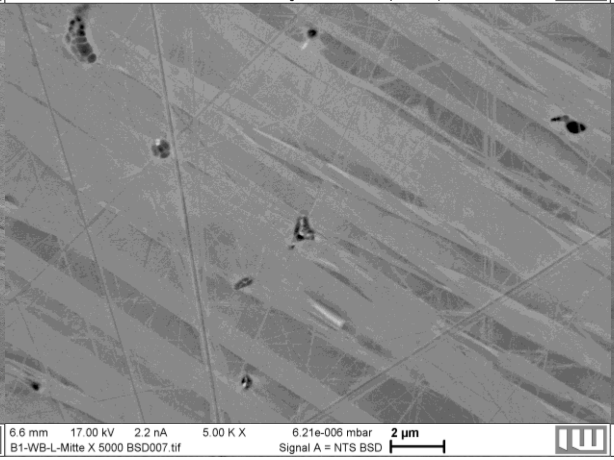
quenched	air (vacuum)	furnace (vacuum)

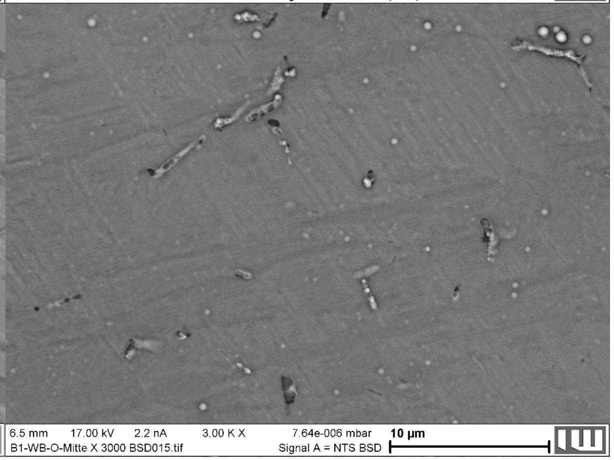






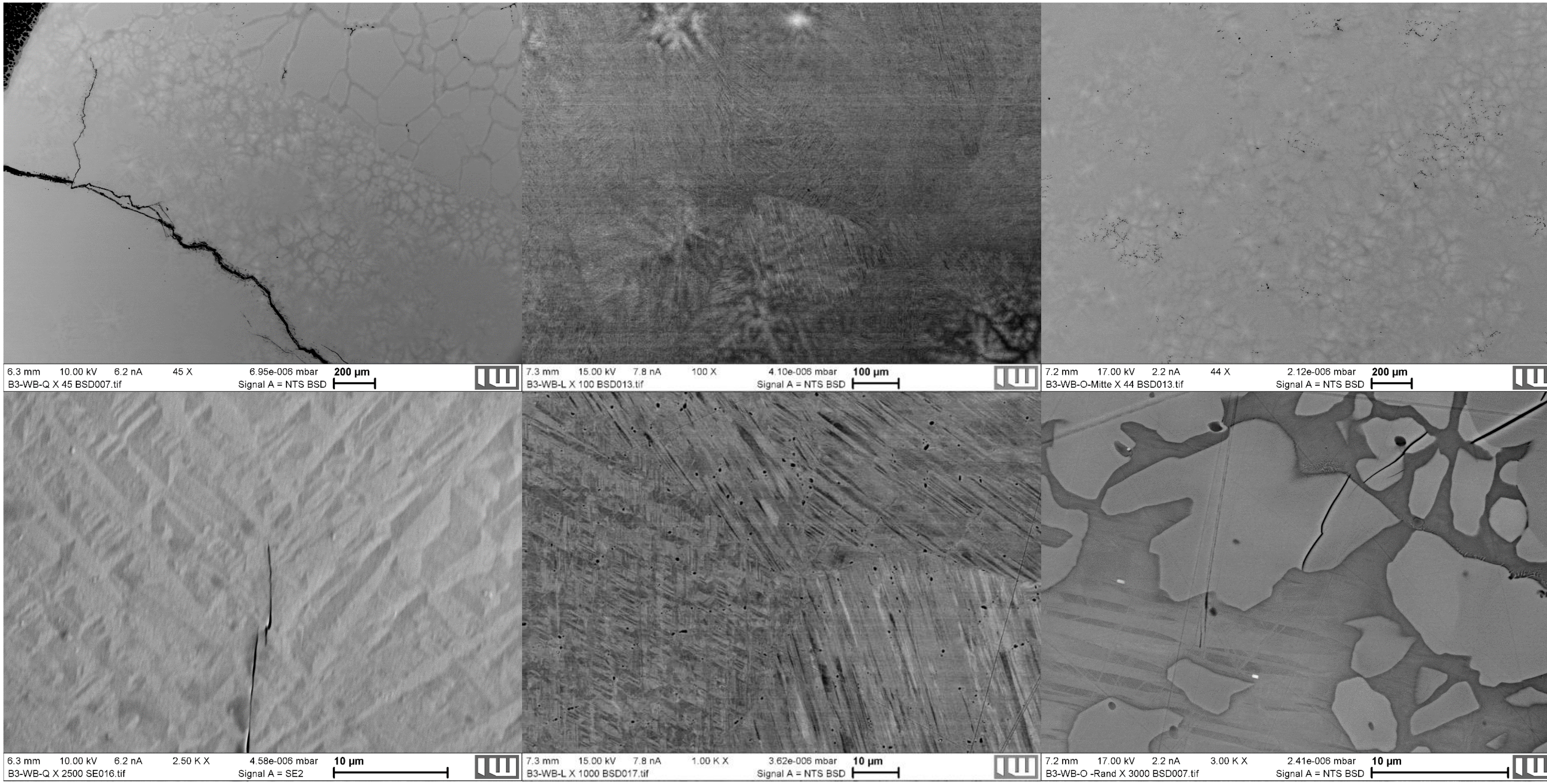






Heattreatment of $Ti_{16.667}Zr_{16.667}Hf_{16.667}Ni_{25}Cu_{25}$

quenched	air (vacuum)	furnace (vacuum)



Summary and outlook

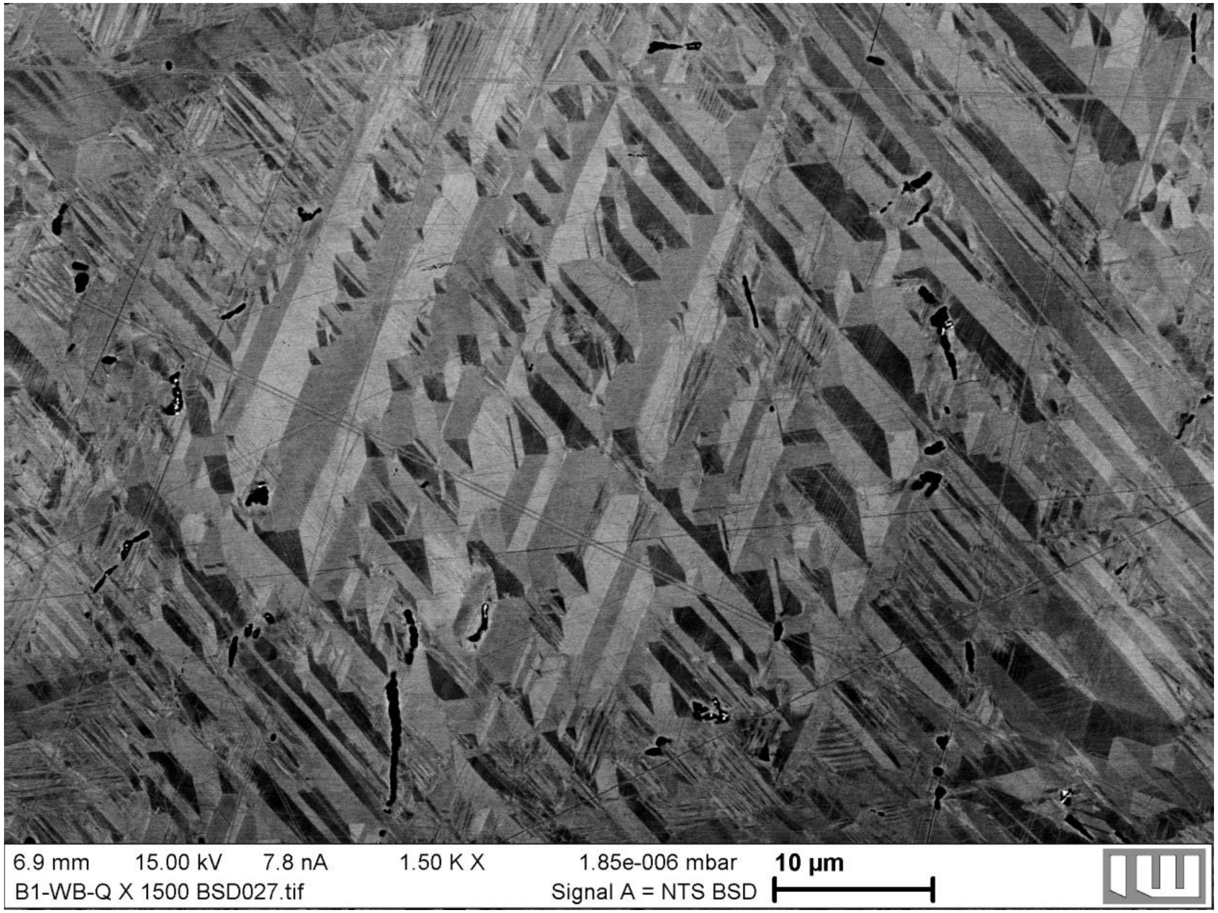
- Heat-treatment isn´t able to remove differences caused by the processing

- Mechanical properties depend on the manufacturing method
- the superelastic effect can be suspected based on the compression tests

- The cooling rate is important in order to receive samples sufficient in size for mechanical testing

- **outlook & further investigation:**
  - Loading experiments on $Ni_{25}Cu_{25}Ti_{16.667}Zr_{16.667}Hf_{16.667}$ & further new materials provided by our research partner at Bochum university
  - Loading experiments and tensile tests on homogenized samples
  - Increase of the loading temperature in order to investigate superelasticity
  - Mechanical & functional fatigue testing influenced by temperature

Best results after controlled cooling



- ✓ Martensite present
- ✓ Crack free
- ✓ Samples with sufficient size