

# Phase stability, precipitation kinetics, nanoscale elemental distributions and their effect on tensile properties in refractory TiZrNbHfTa BCC high-entropy alloys

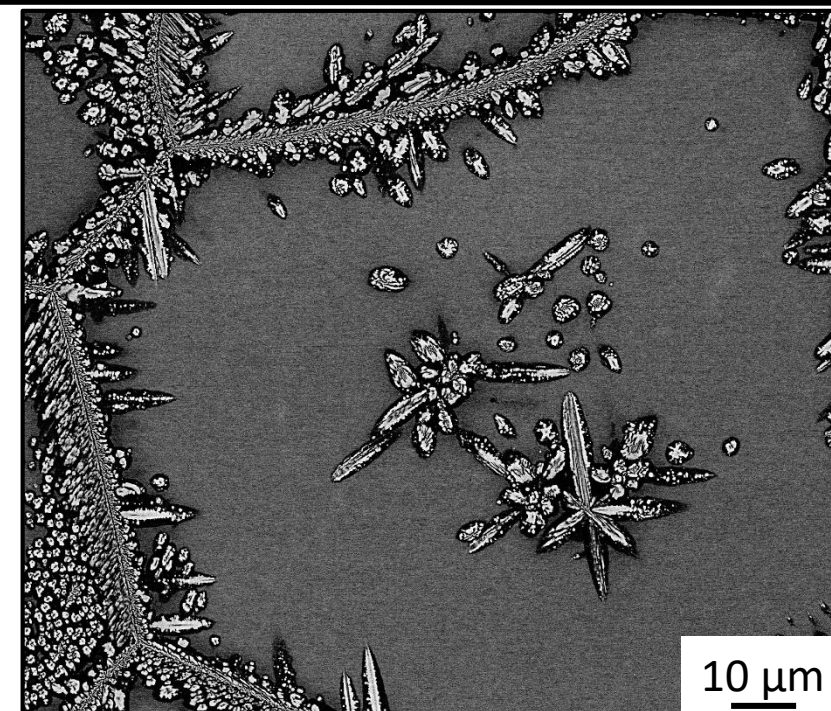
## Part I



P. Gemagami, Y. Zhao, T. Li, G. Laplanche

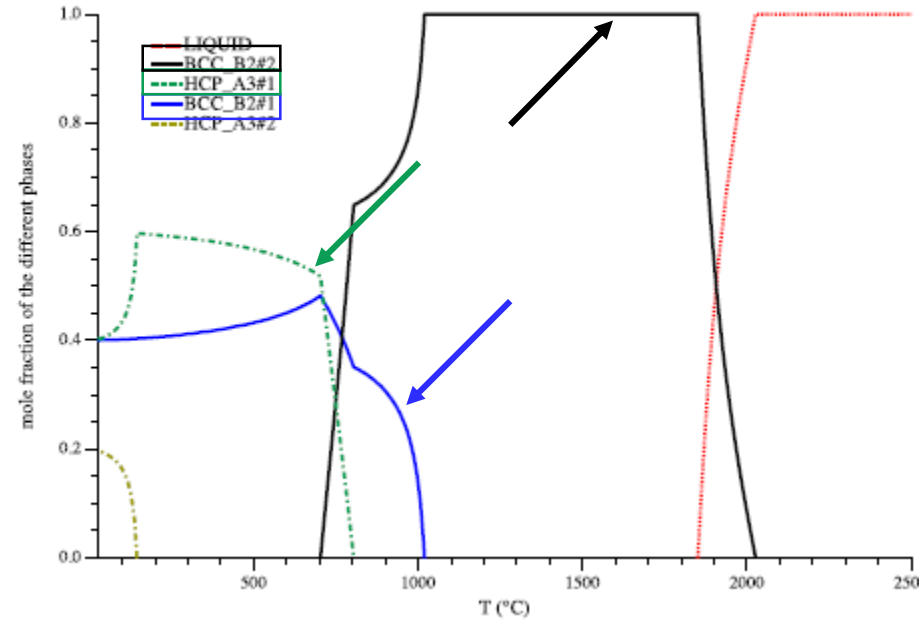


Institut für Werkstoffe, Ruhr-Universität Bochum, 44801 Bochum, Germany





## Phase stability of TiZrNbHfTa



CALPHAD calculations of the TiZrNbHfTa alloy with the TCHEA1 database, evolution of the mole fraction of the stable phases

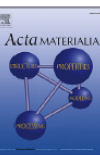
Acta Materialia 142 (2018) 201–212



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Acta Materialia

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Full length article

Thermodynamic instability of a nanocrystalline, single-phase TiZrNbHfTa alloy and its impact on the mechanical properties

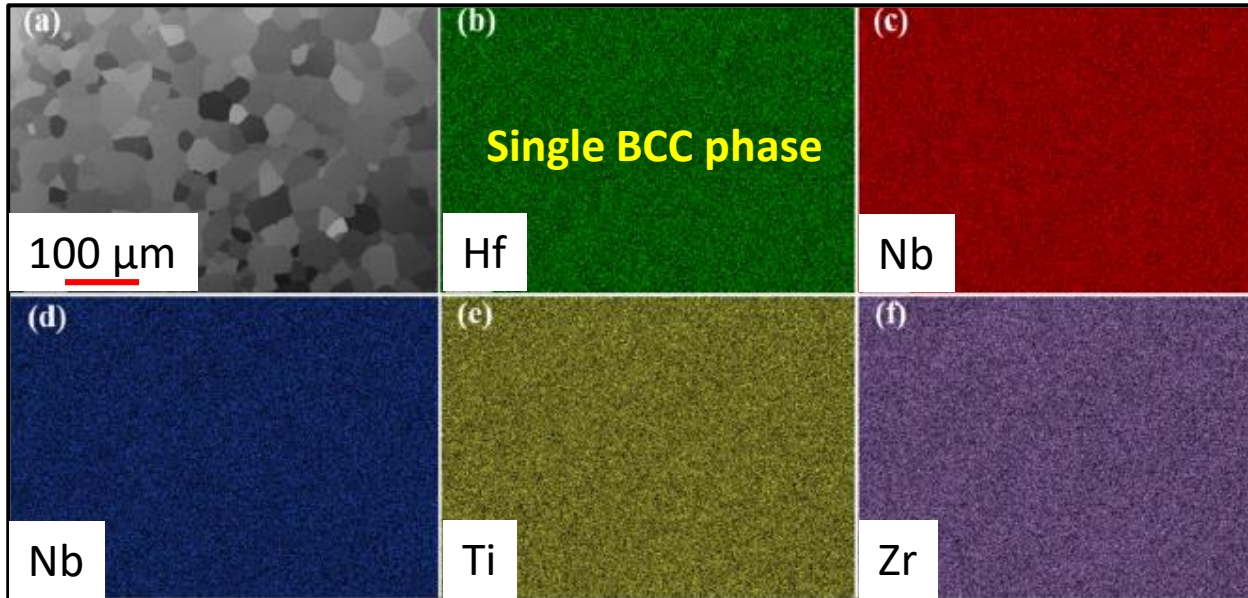
B. Schuh<sup>a,\*</sup>, B. Völker<sup>a</sup>, J. Todt<sup>b</sup>, N. Schell<sup>c</sup>, L. Perrière<sup>d</sup>, J. Li<sup>e</sup>, J.P. Couzinié<sup>d</sup>, A. Hohenwarter<sup>a</sup>





# Introduction

Cold rolled (70% reduction) → homogenized at 1200°C for 5 min and water quenched



Scripta Materialia 158 (2019) 50–56



Contents lists available at ScienceDirect

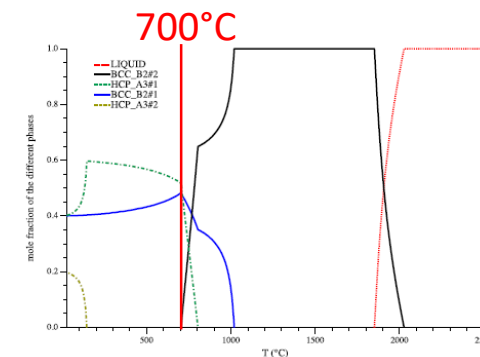
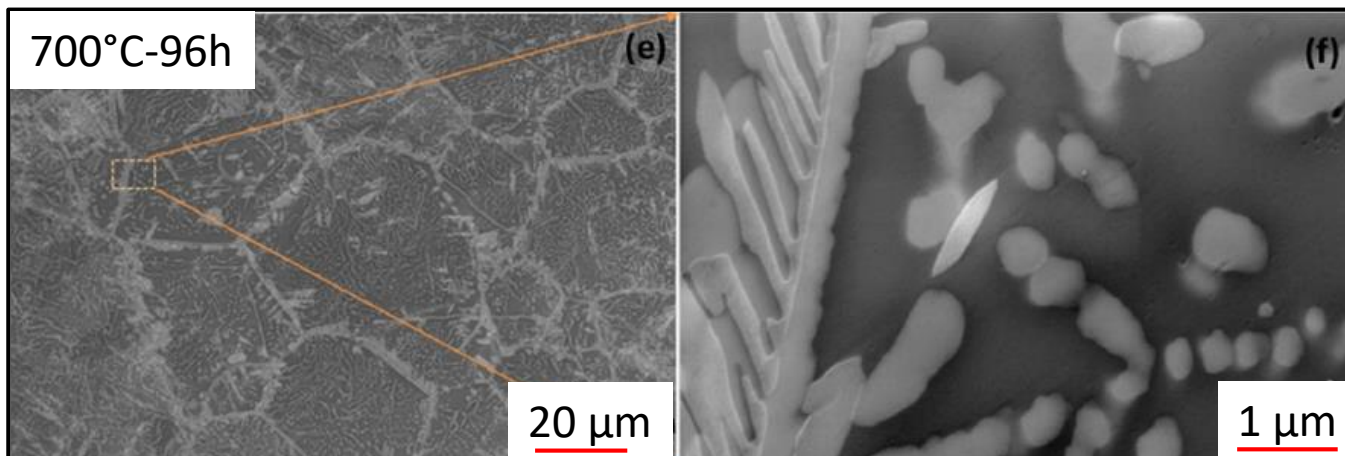
Scripta Materialia

journal homepage: [www.elsevier.com/locate/scriptamat](http://www.elsevier.com/locate/scriptamat)



Phase transformations of HfNbTaTiZr high-entropy alloy at intermediate temperatures☆

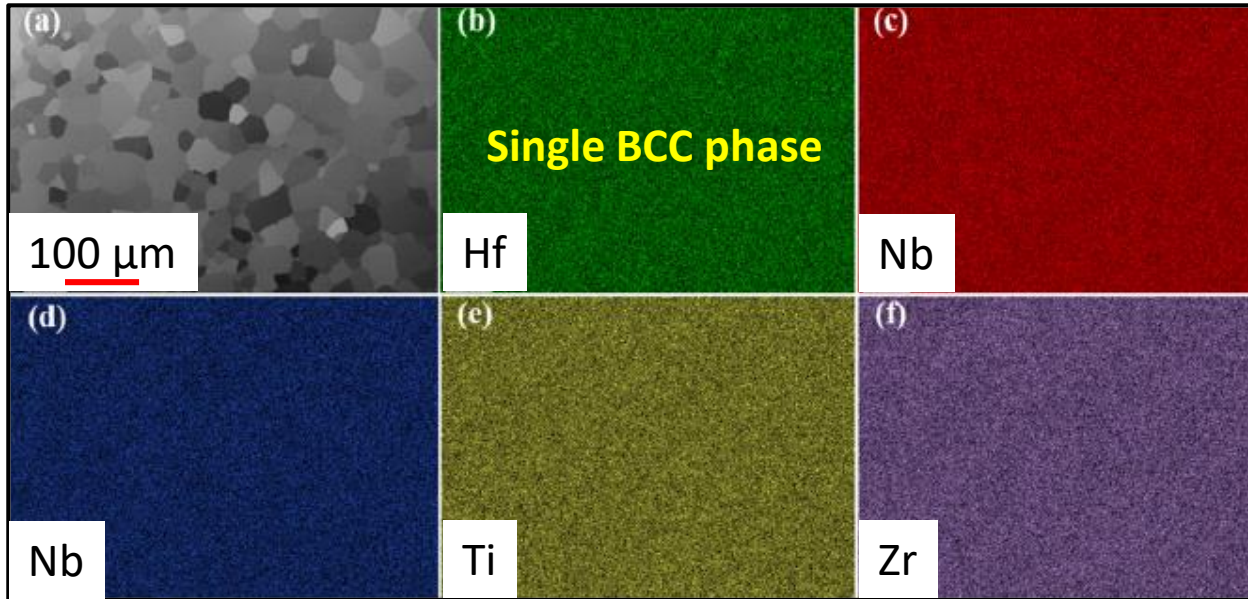
S.Y. Chen<sup>a</sup>, Y. Tong<sup>a</sup>, K.-K. Tseng<sup>b</sup>, J.-W. Yeh<sup>b</sup>, J.D. Poplawsky<sup>c</sup>, J.G. Wen<sup>d</sup>, M.C. Gao<sup>e,f</sup>, G. Kim<sup>g</sup>, W. Chen<sup>g</sup>, Y. Ren<sup>h</sup>, R. Feng<sup>a</sup>, W.D. Li<sup>a</sup>, P.K. Liaw<sup>a,\*</sup>





# Introduction

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Contents lists available at ScienceDirect

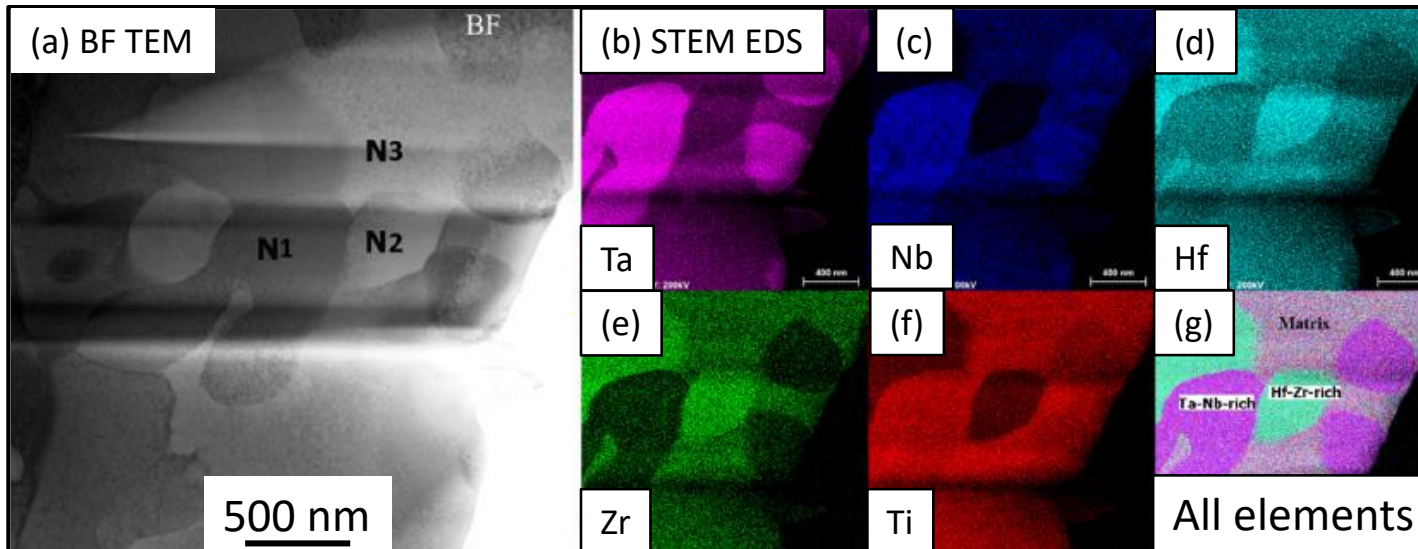
Scripta Materialia

journal homepage: [www.elsevier.com/locate/scriptamat](http://www.elsevier.com/locate/scriptamat)



Phase transformations of HfNbTaTiZr high-entropy alloy at intermediate temperatures☆

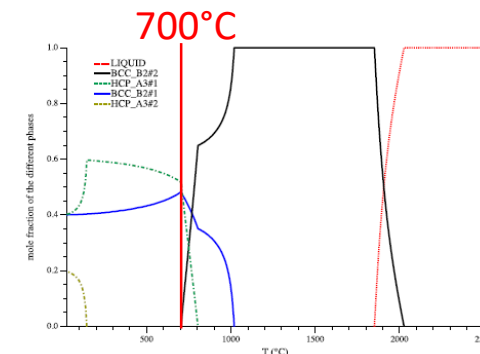
S.Y. Chen<sup>a</sup>, Y. Tong<sup>a</sup>, K.-K. Tseng<sup>b</sup>, J.-W. Yeh<sup>b</sup>, J.D. Poplawsky<sup>c</sup>, J.G. Wen<sup>d</sup>, M.C. Gao<sup>e,f</sup>, G. Kim<sup>g</sup>, W. Chen<sup>g</sup>, Y. Ren<sup>h</sup>, R. Feng<sup>a</sup>, W.D. Li<sup>a</sup>, P.K. Liaw<sup>a,\*</sup>



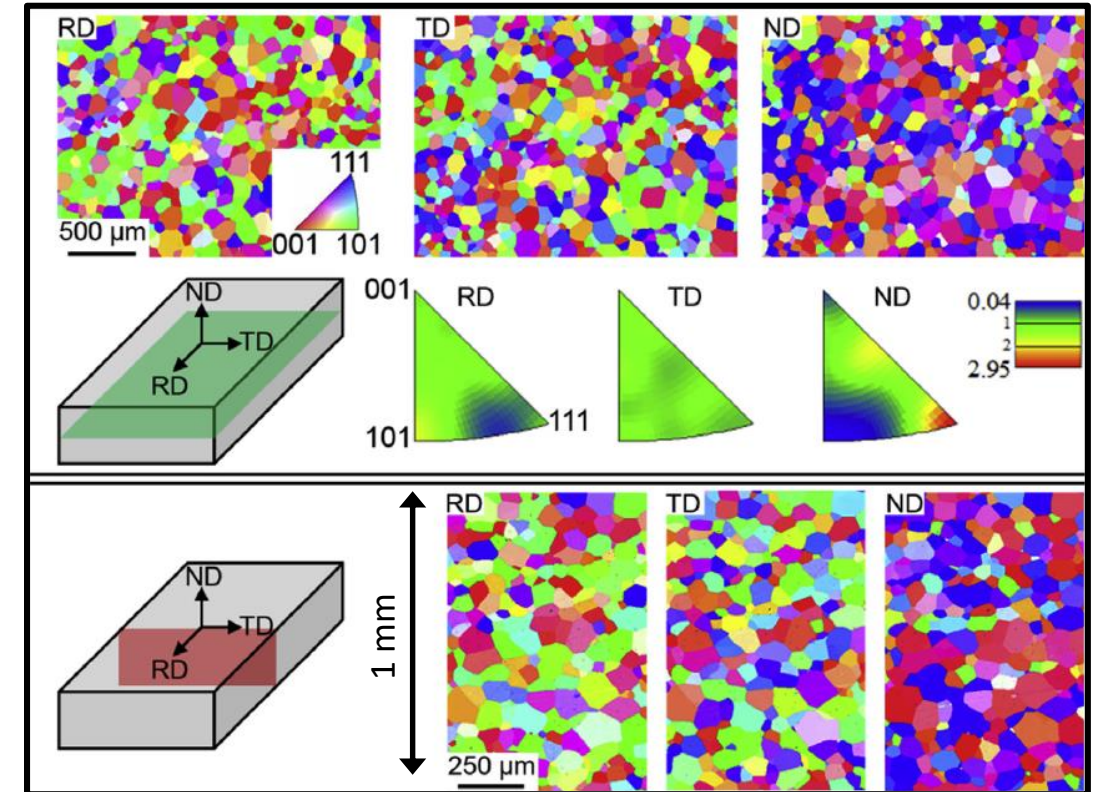
N1: **Ta-Nb** rich phase, **BCC**

N2: **Hf-Zr** rich phase, **HCP**

N3: **matrix**, **BCC**



cold rolling (~80%) + annealing at 1100°C for 5h



Laplanche et al., *Journal of alloys and compounds* 799 (2019) 538-545

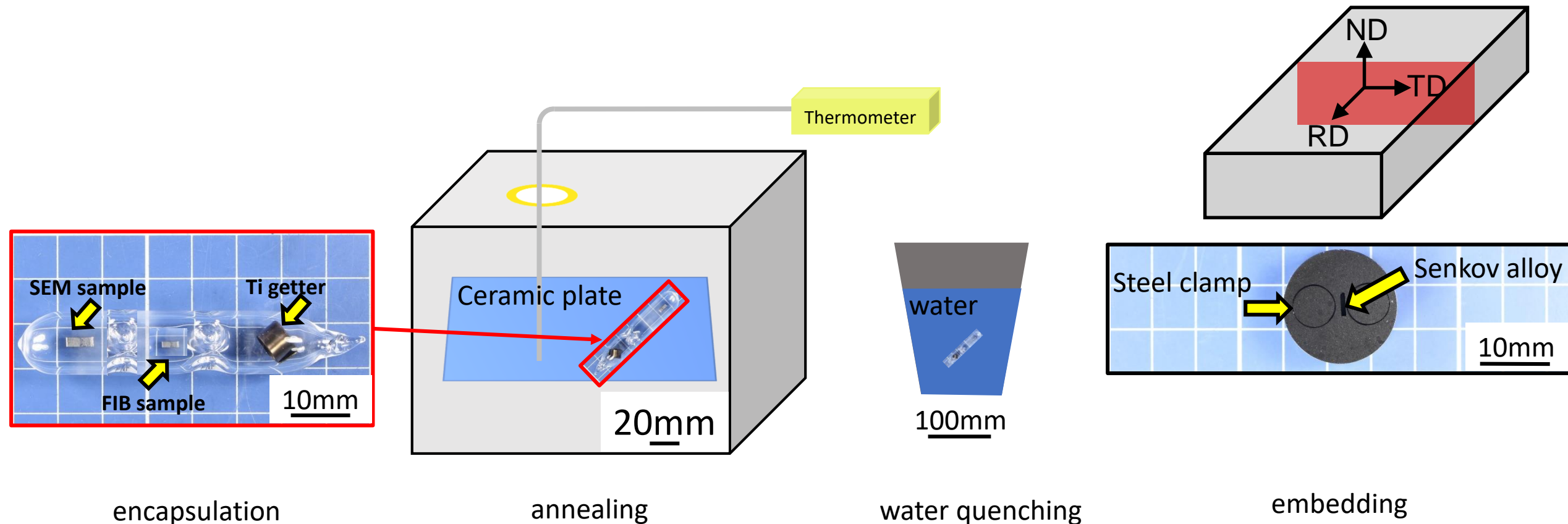


J.-P. Couzinié  
Institut de Chimie et  
des Matériaux Paris-Est

- Nearly equiaxed grains
- Nearly no texture along RD and TD
- Slight <111>- and <001>-fiber textures along ND
- average grain size **~80 μm**

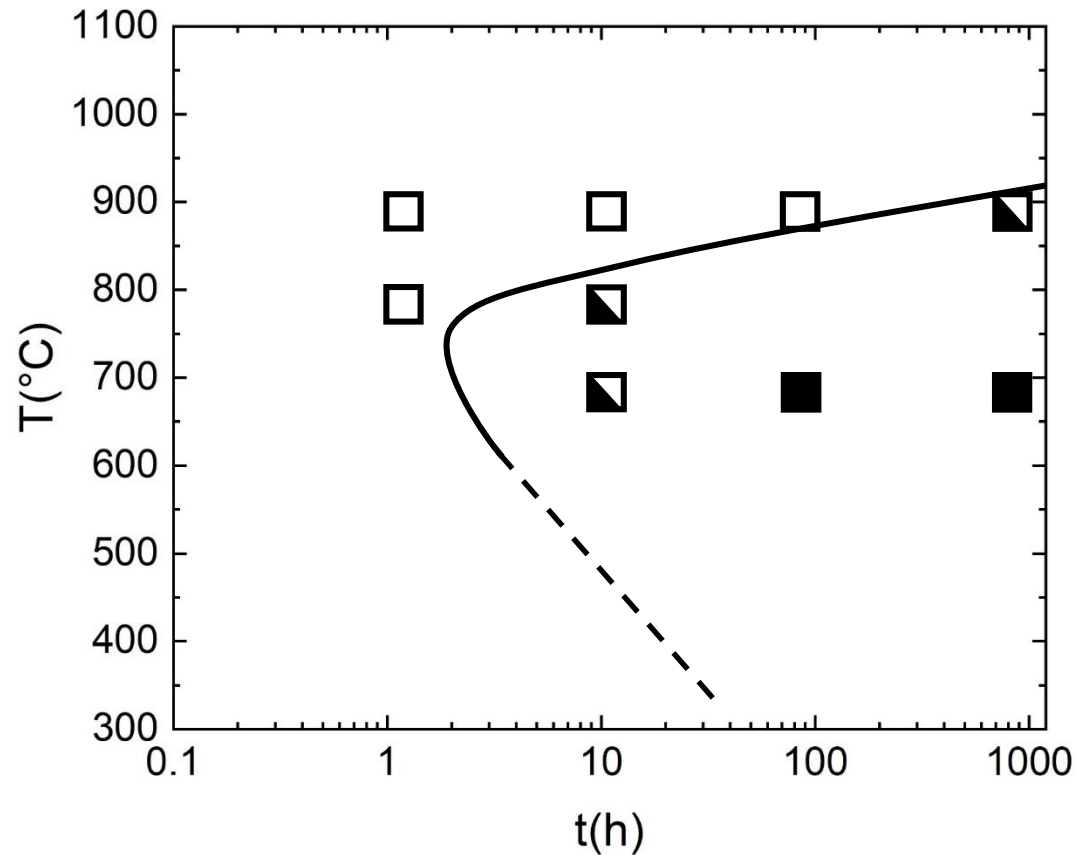


- Sheets (thickness  $\sim 1\text{mm}$ ) recrystallized at  $1100^\circ\text{C}$  for 5 h
- Specimens ( $4\text{mm} \times 2\text{mm} \times 1\text{mm}$ ) encapsulated in evacuated-quartz tubes with a Ti getter (thin coiled sheet)
- Long-term anneal + water quenching.
- **The annealed specimens were then embedded in epoxy with a steel clamp to observe the microstructure along a direction parallel to RD.**





# Preliminary TTT diagram



## Temperature

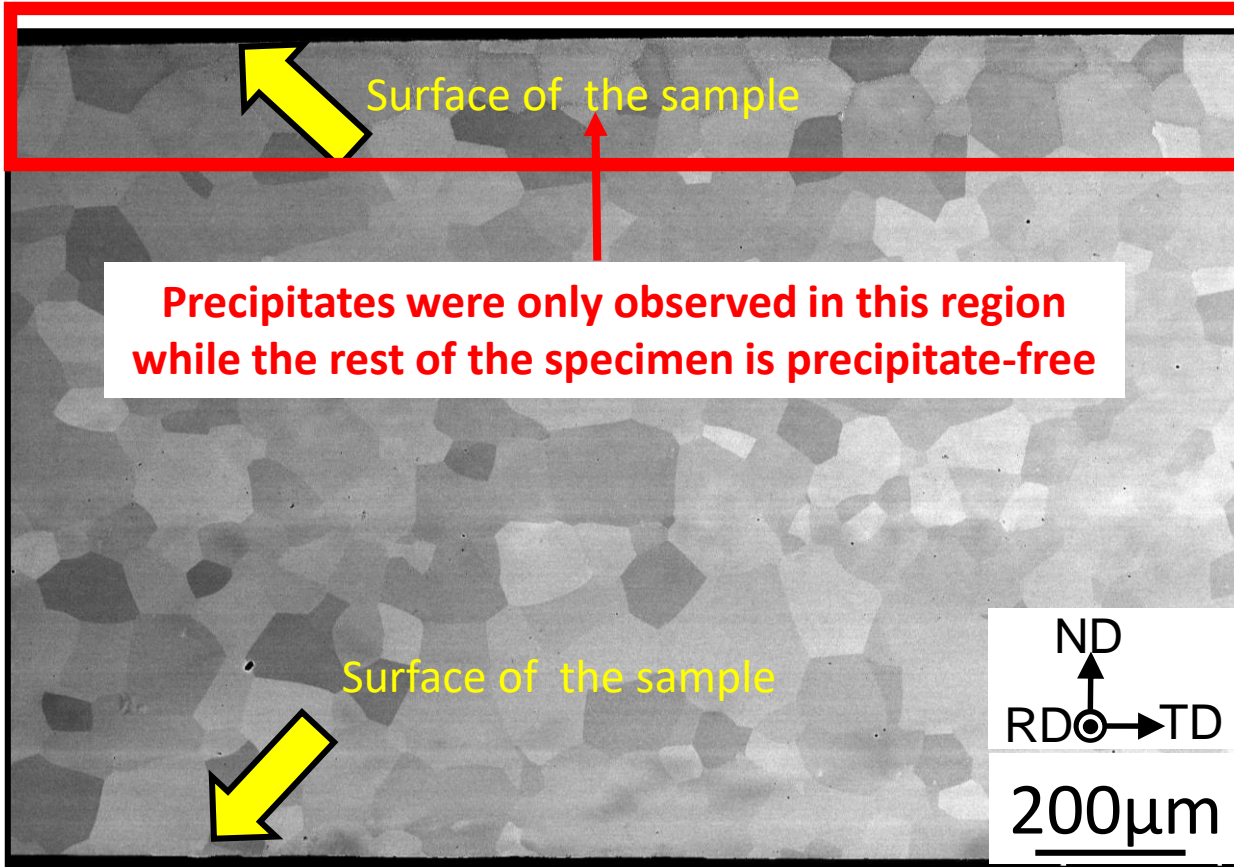
700°C	1h	10h	100h	1000h
800°C	1h	10h	100h	1000h
900°C		10h	100h	1000h



done

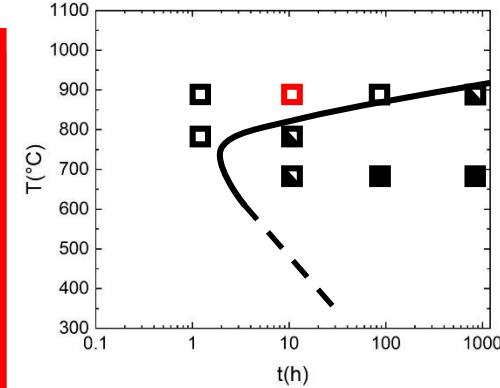
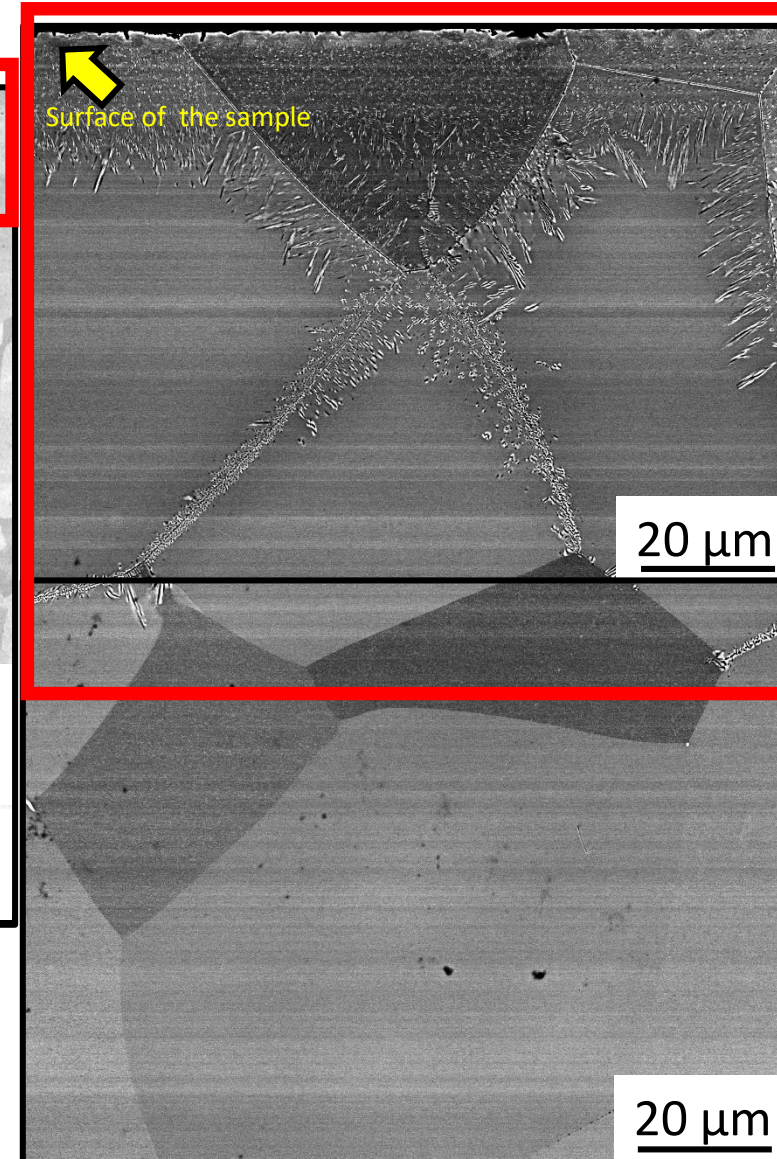


in the plan



Magnification 70x

O<sub>2</sub> and N<sub>2</sub> contamination at the surface  
Do they induce a change in phase stability?





# HT-900°C-10h

Collaboration with KIT

Effect of O<sub>2</sub> and N<sub>2</sub> on phase stability

Two cast and homogenized ingots will be produced

- TiZrNbHfTa
- TiZrNbHfTa +2 at.%O<sub>2</sub>
- TiZrNbHfTa +2 at.%N<sub>2</sub>



Leibniz Institute  
for Solid State and  
Materials Research  
Dresden



Martin Heilmaier



Alexander Kauffmann



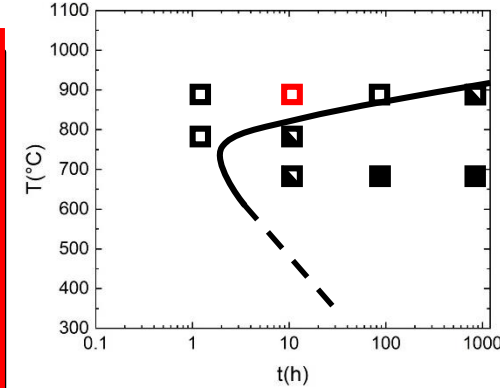
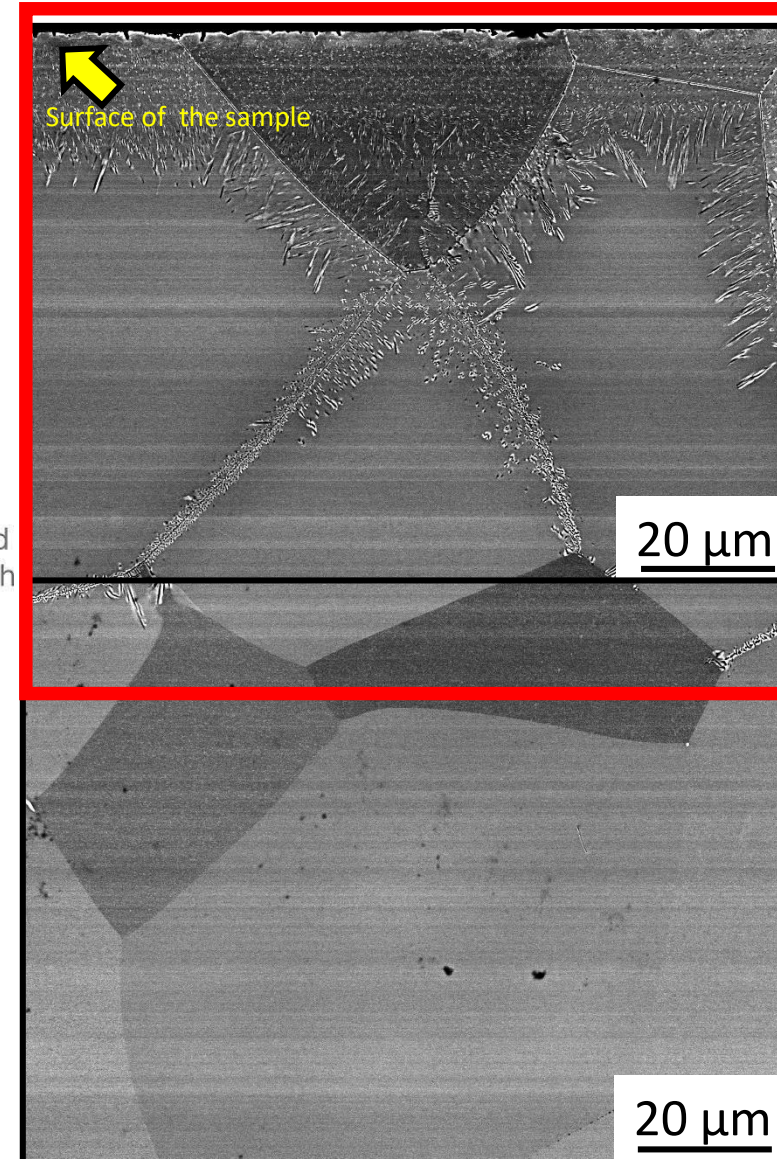
Hans Chen



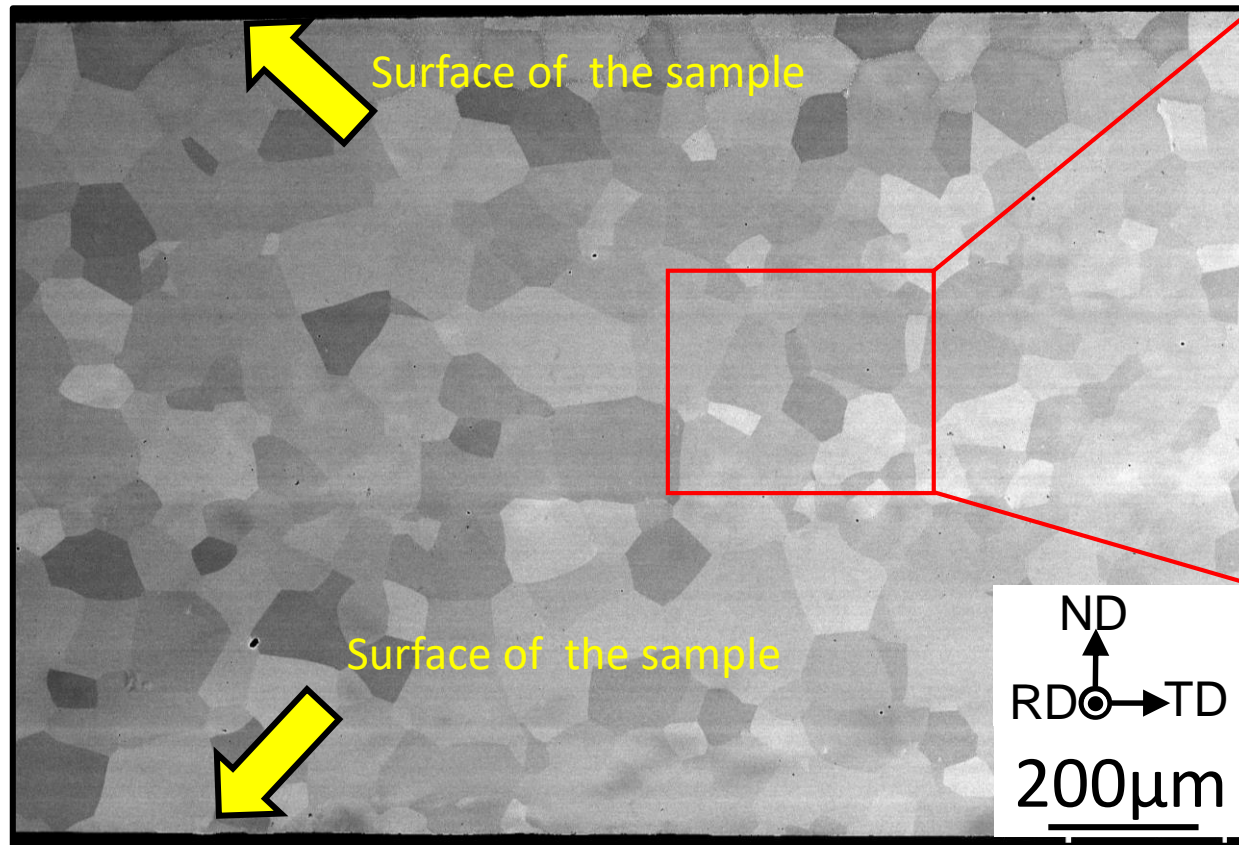
Jens Freudenberger

Casting

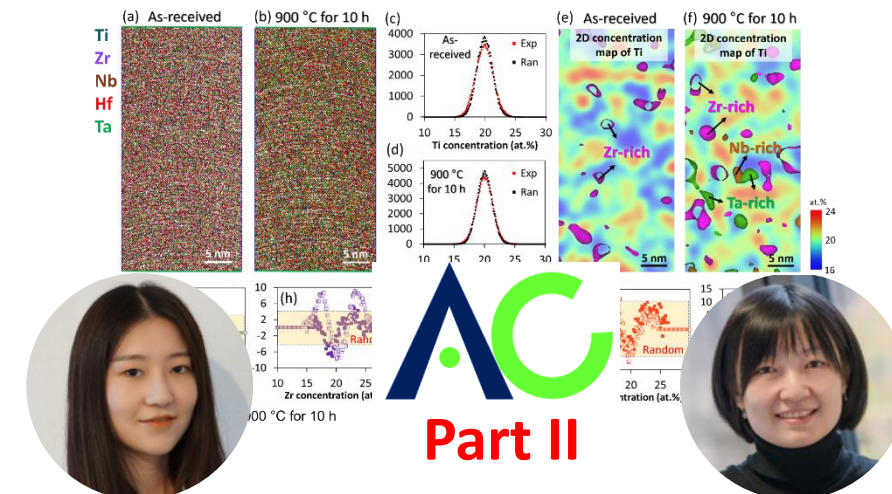
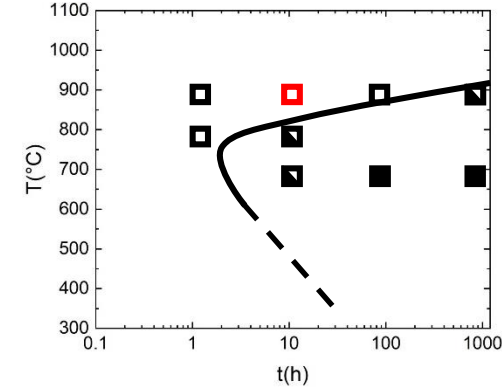
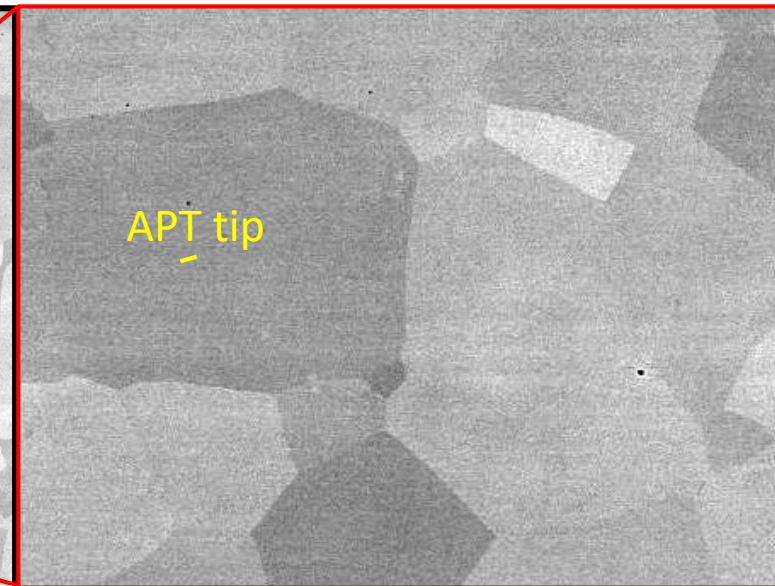
Forging



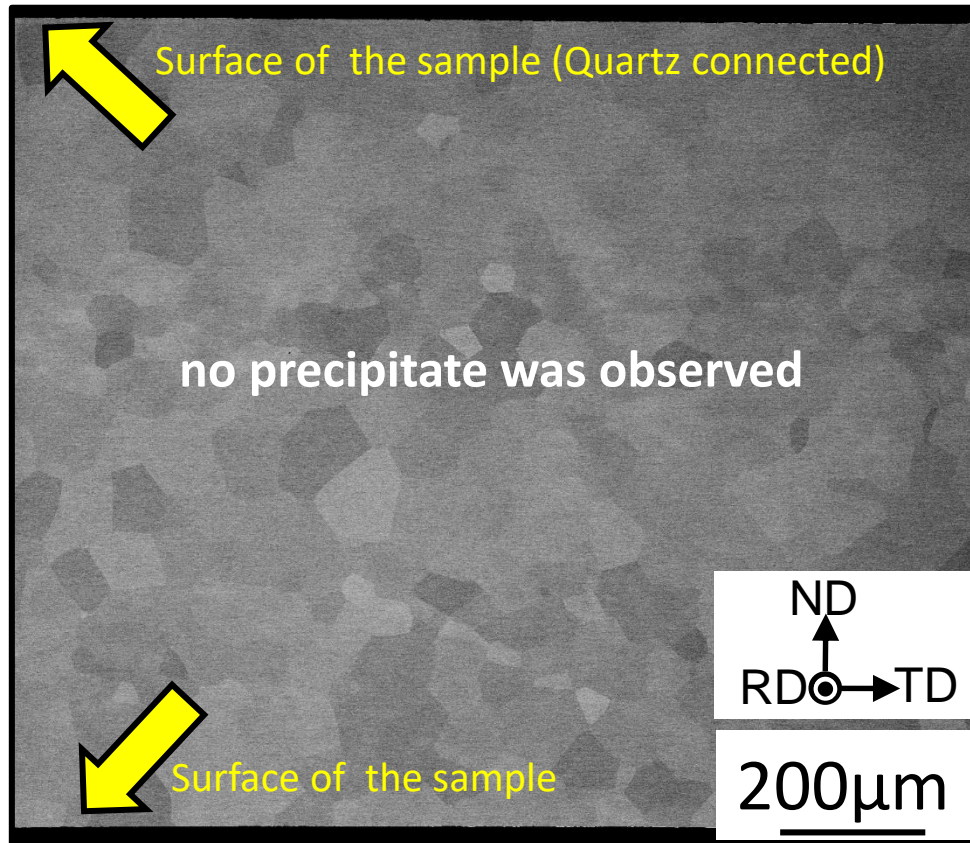
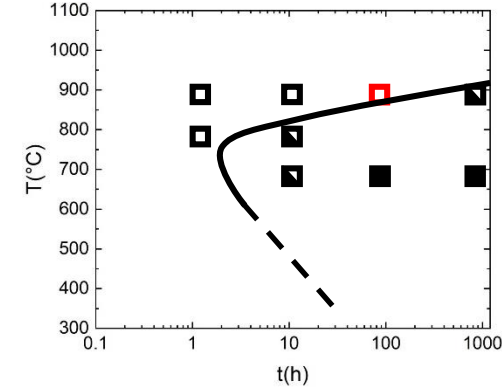




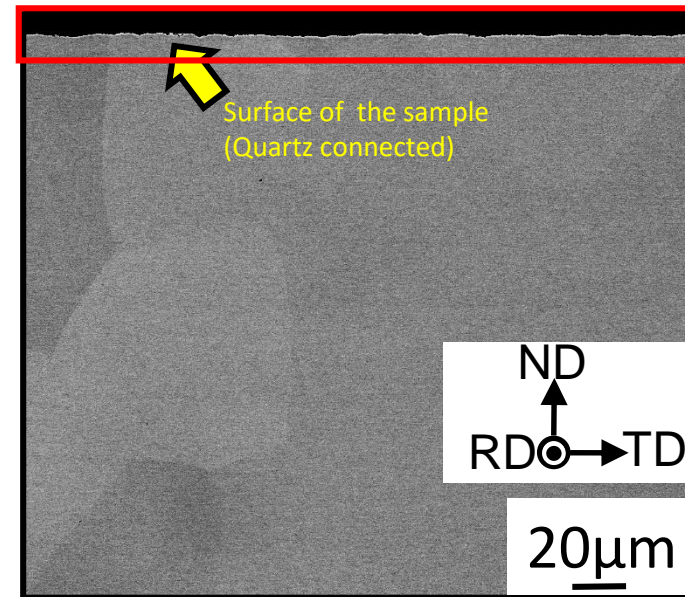
Magnification 70x



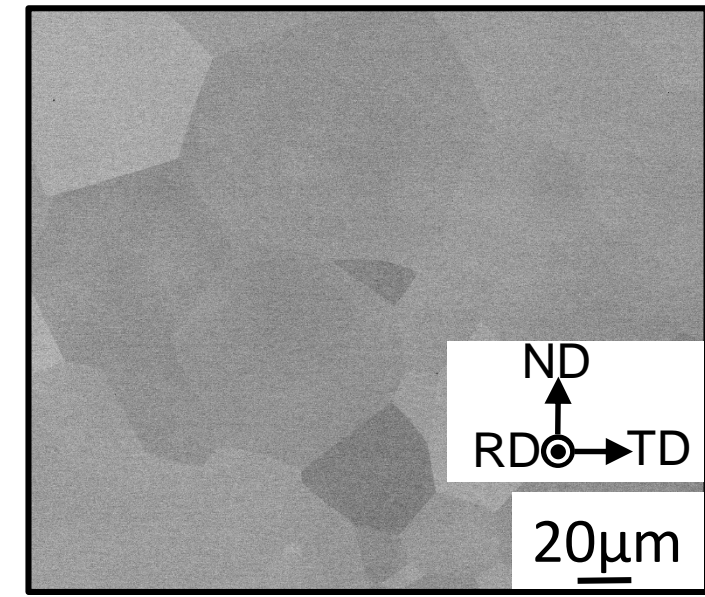




Upper part of the sheet



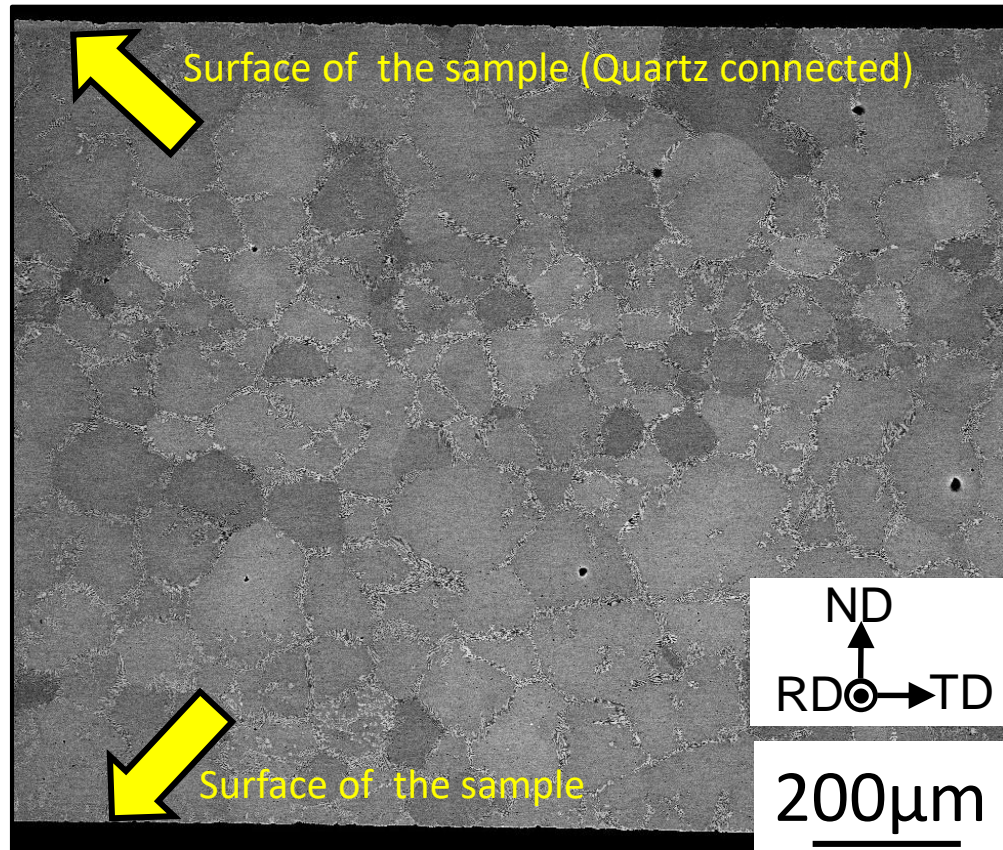
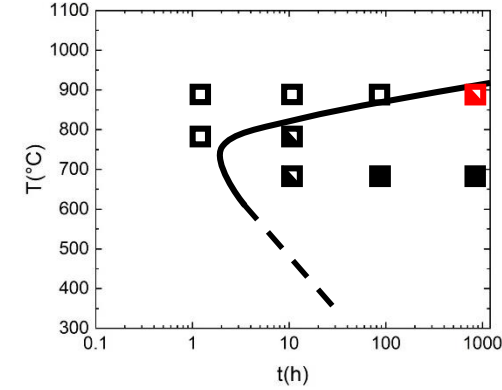
Middle part of the sheet



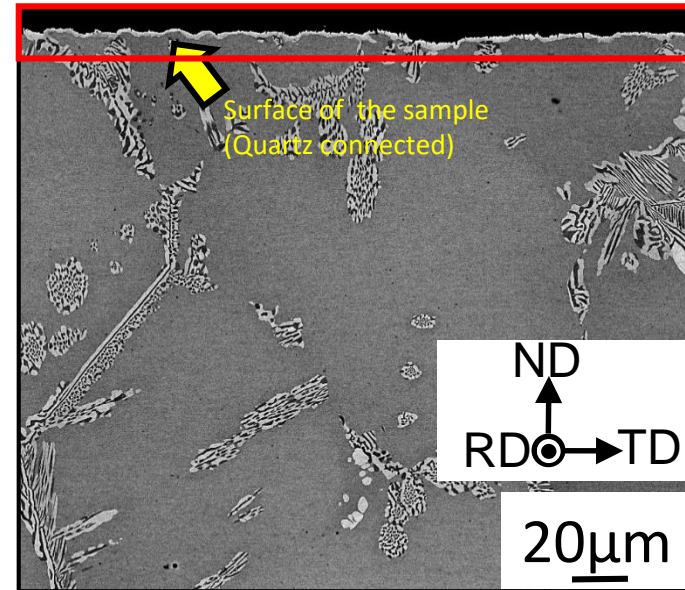
Better vacuum in quartz tube?  
The layer was dissolved?



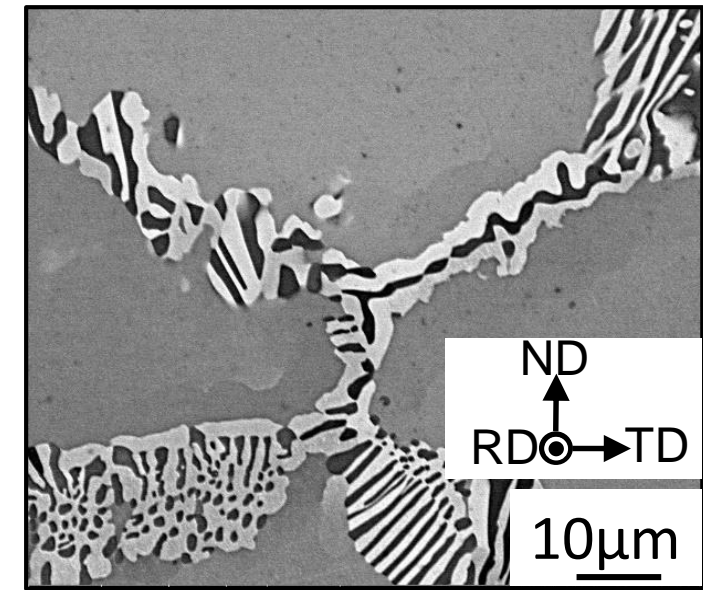
# HT-900°C-1000h



Upper part of the sheet



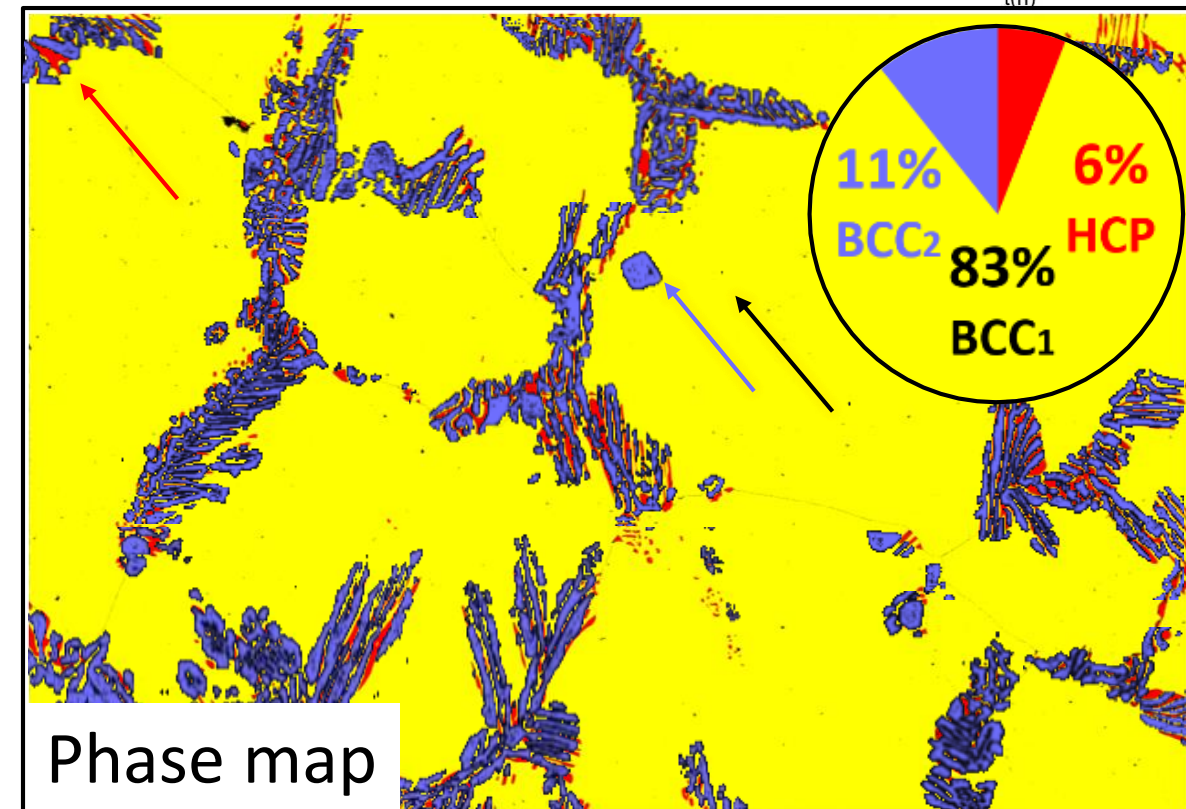
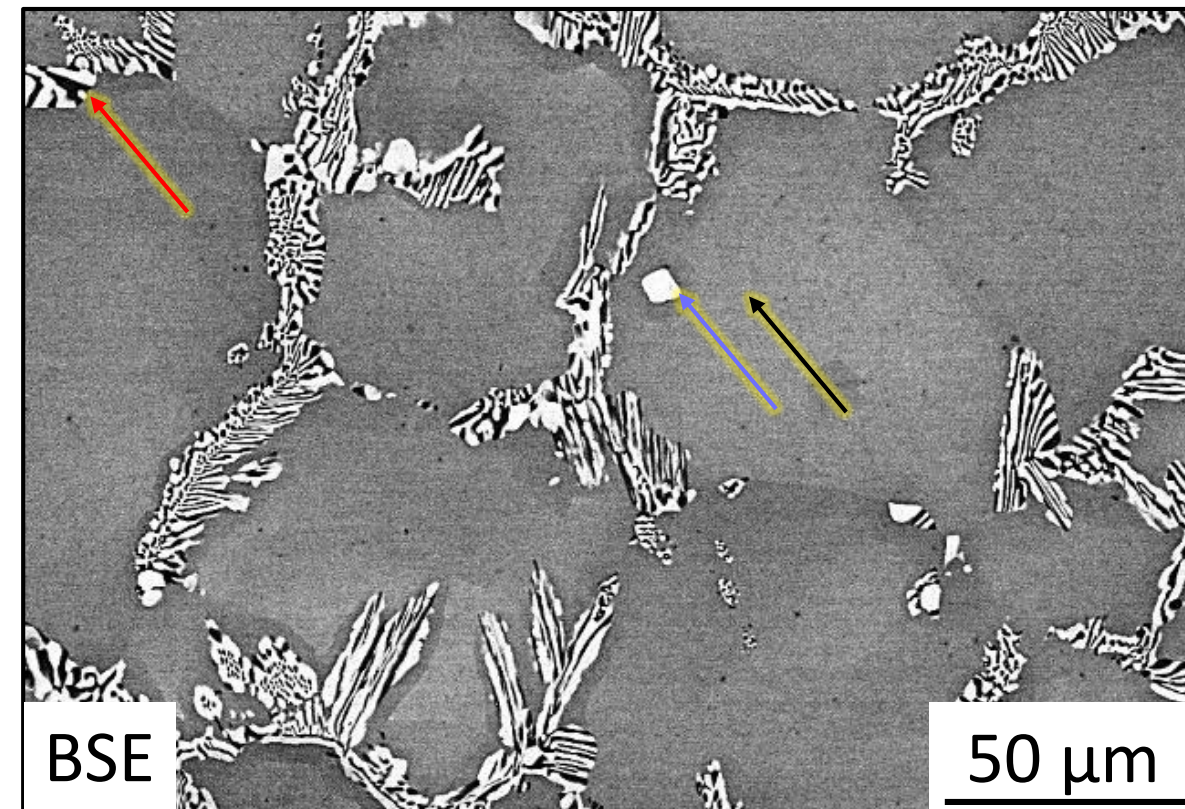
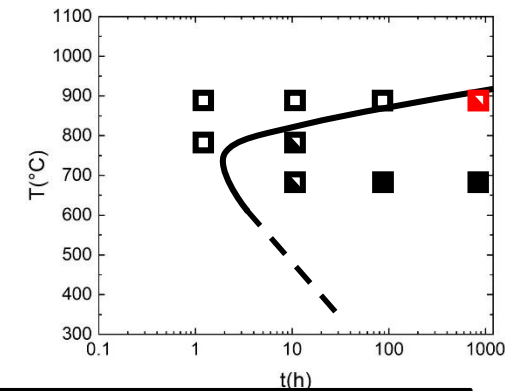
Middle part of the sheet



Precipitates were observed mostly at grain boundaries

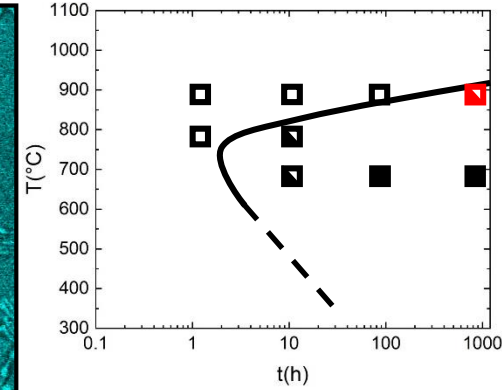
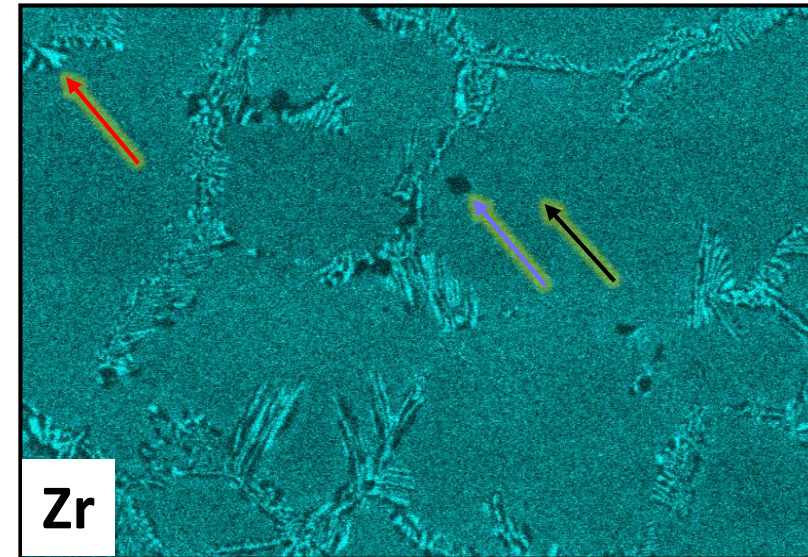
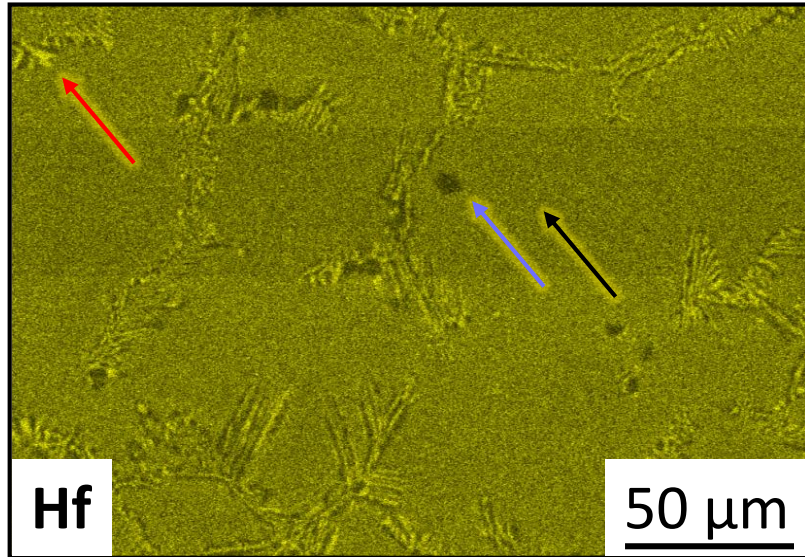


# HT-900°C-1000h





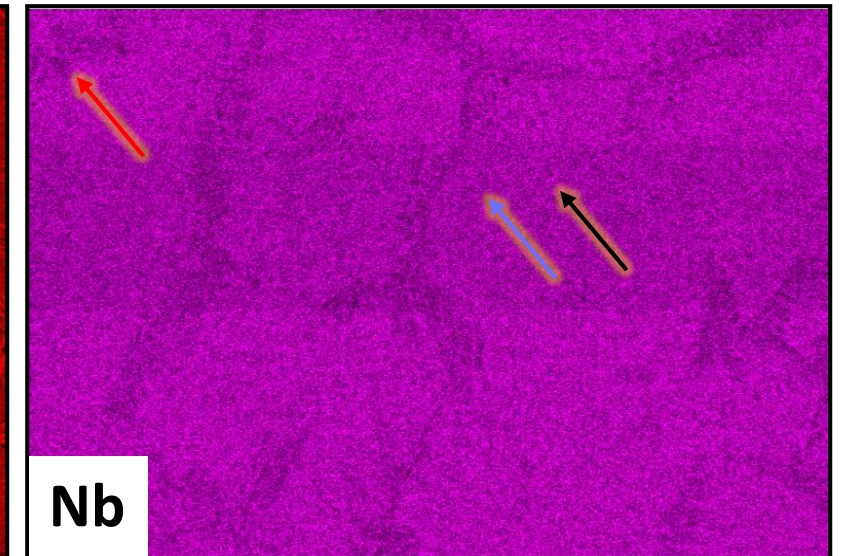
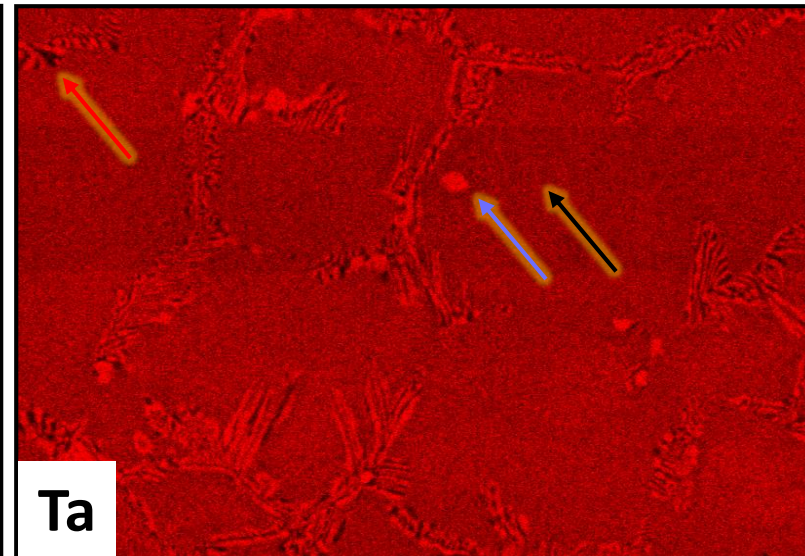
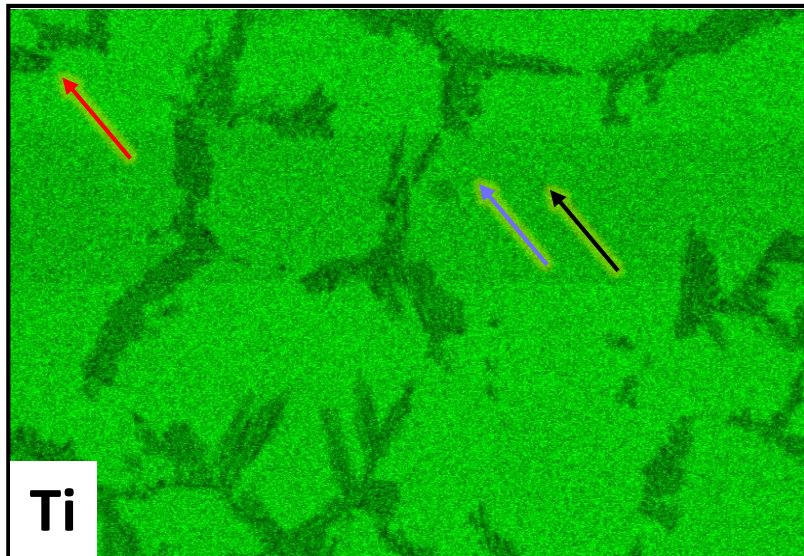
# HT-900°C-1000h



HCP-precipitates: enriched in Hf, Zr

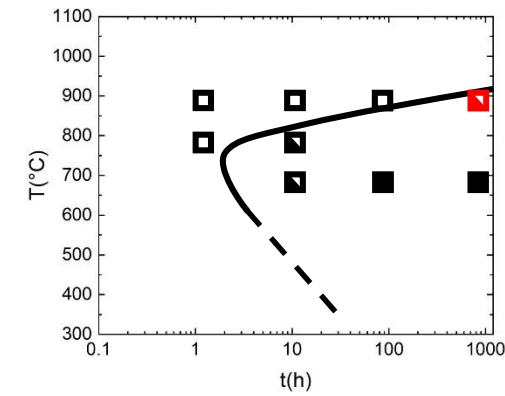
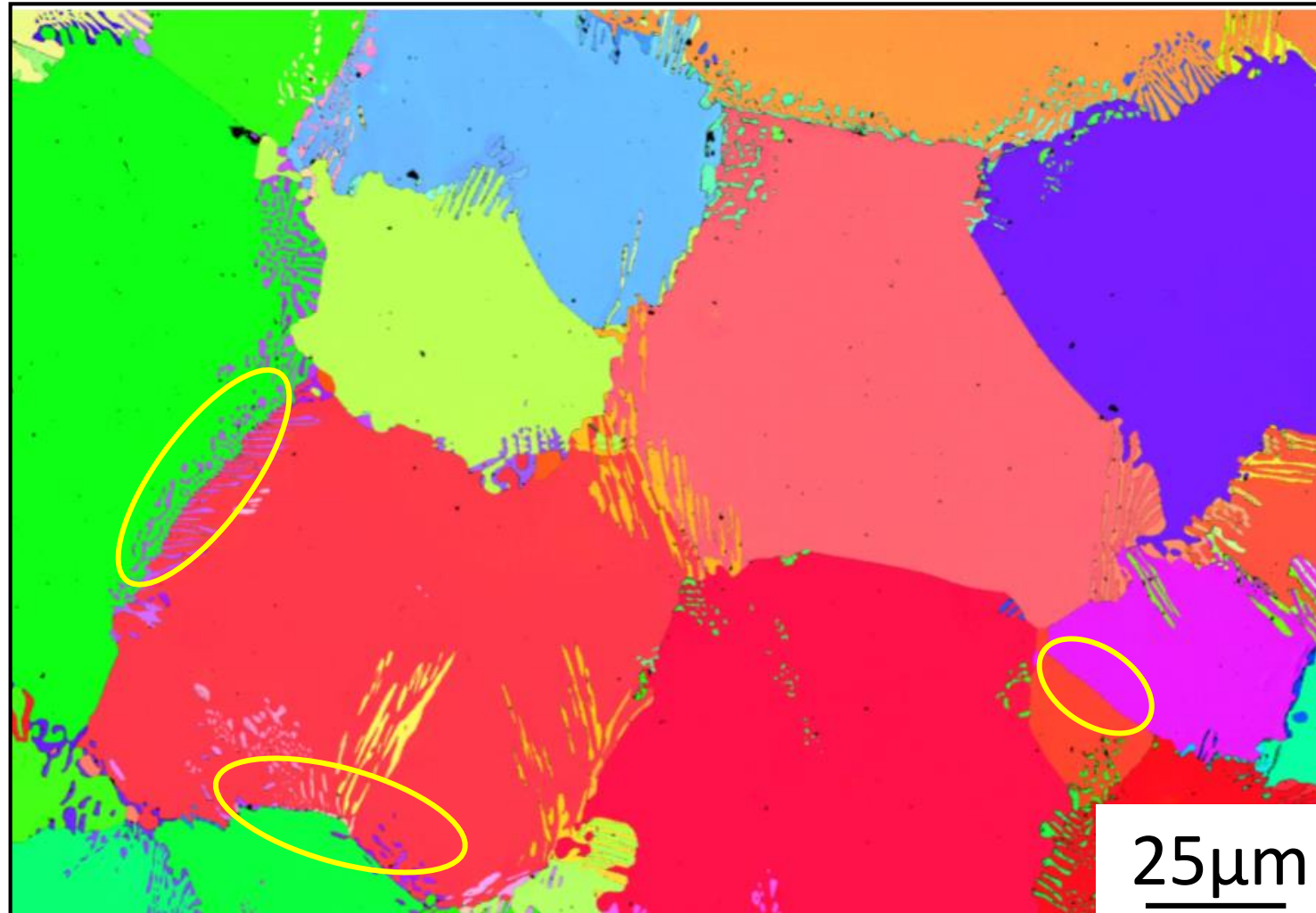
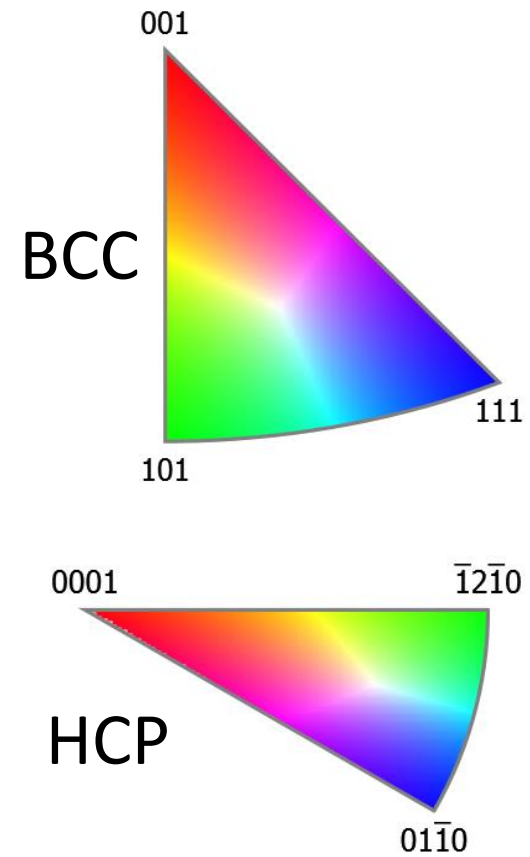
BCC precipitates: enriched in Ta

BCC-matrix: mixture, slight enrichment in Ti



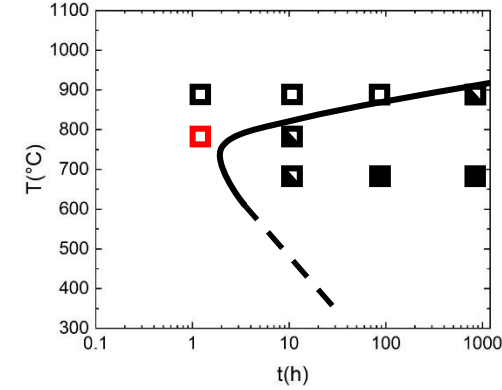
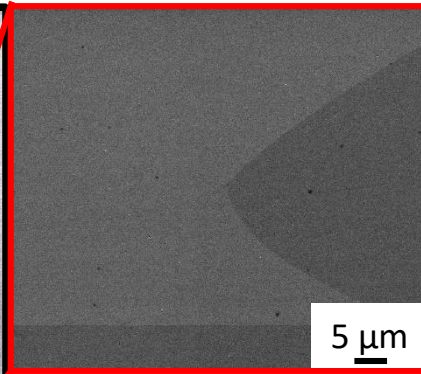
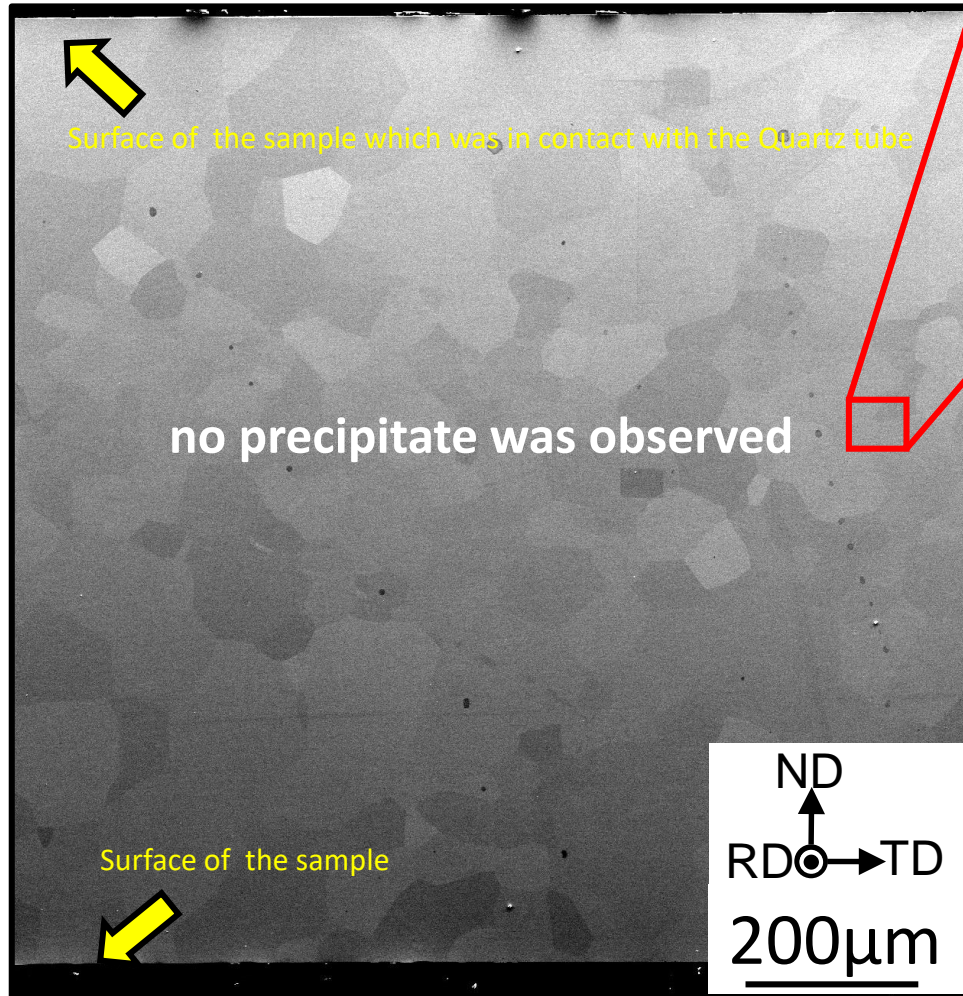


# HT-900°C-1000h

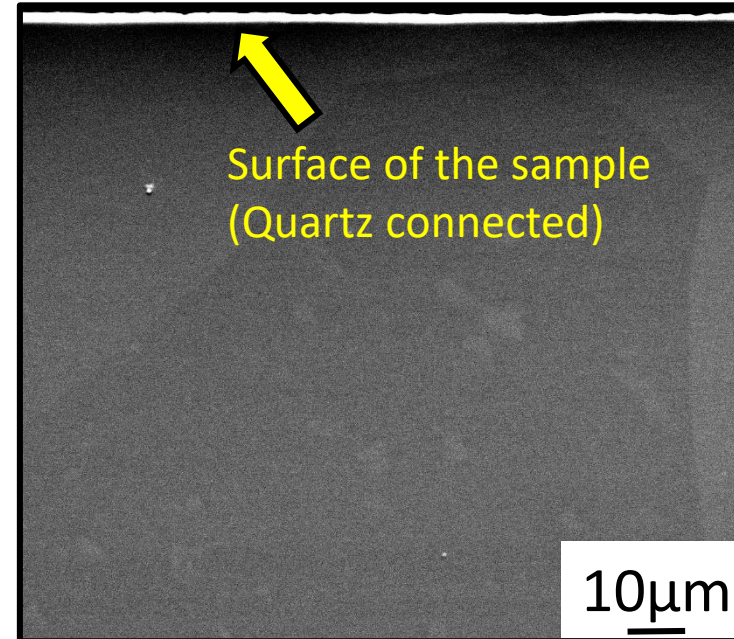




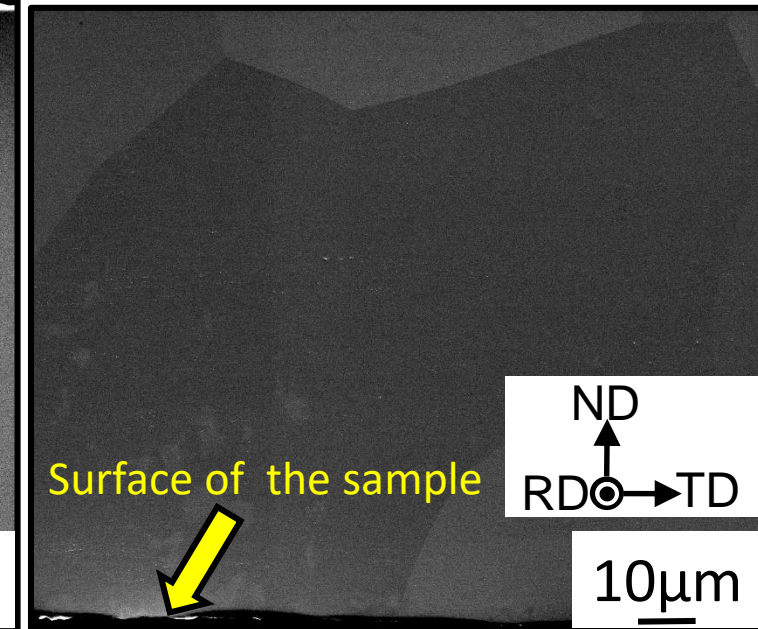
Magnification 70x



Magnification 1000x



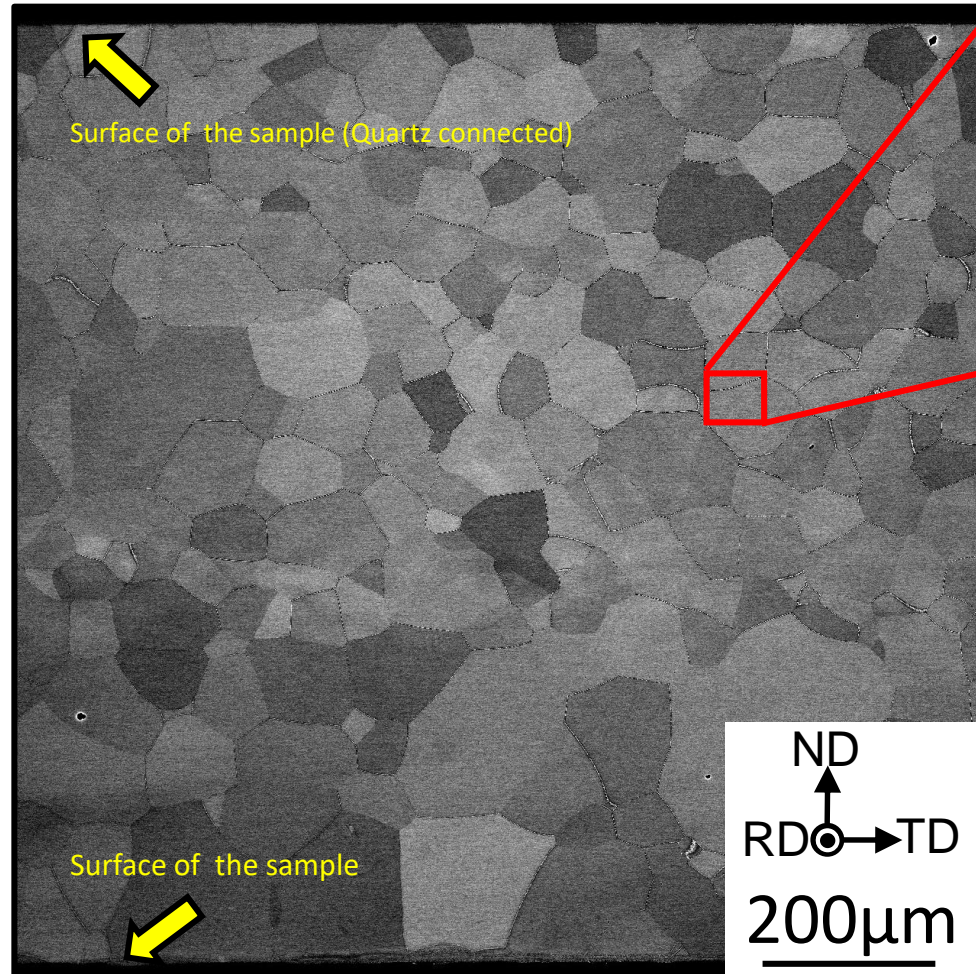
Upper part of the sheet



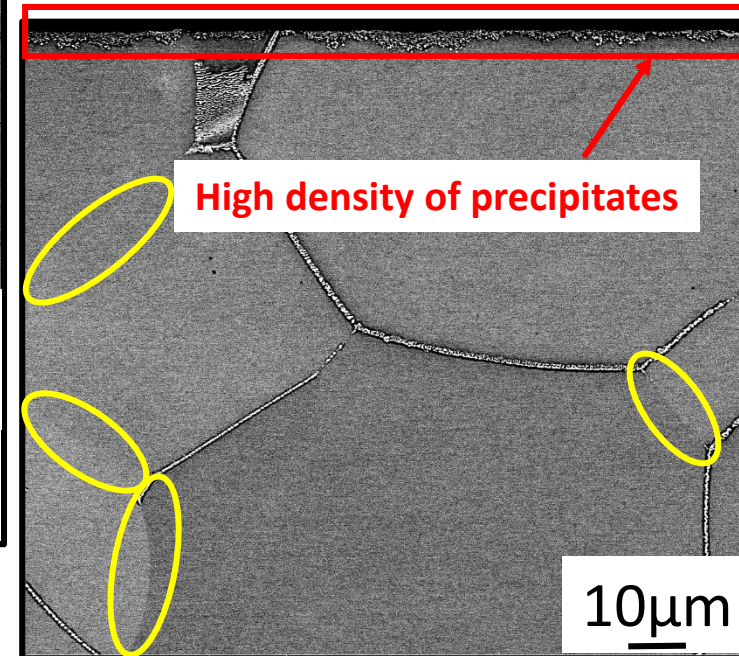
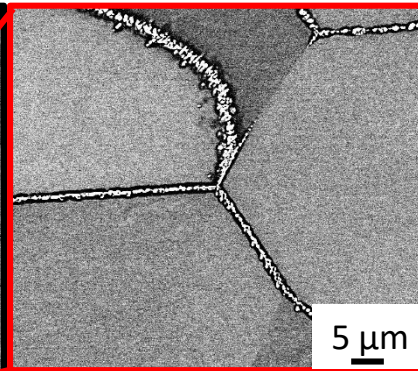
Bottom part of the sheet



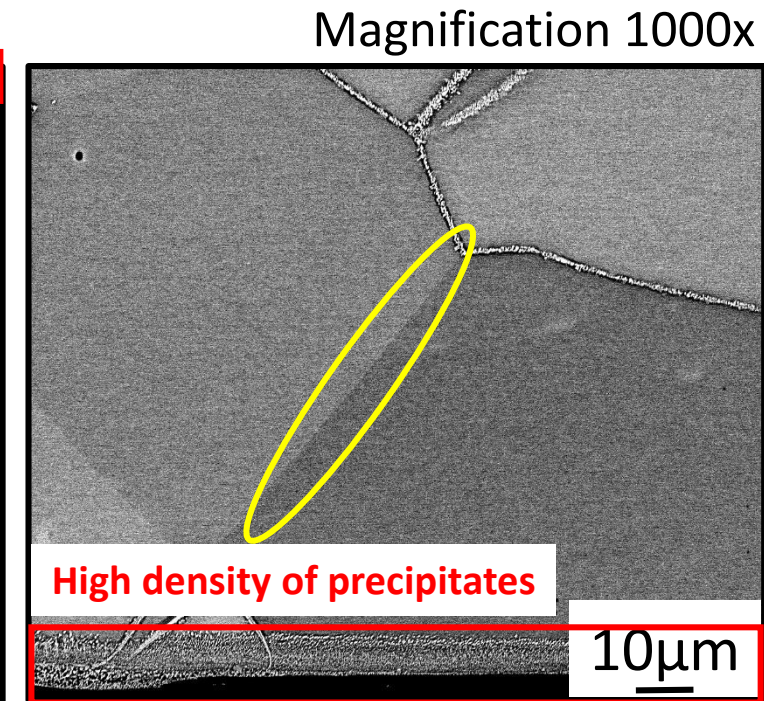
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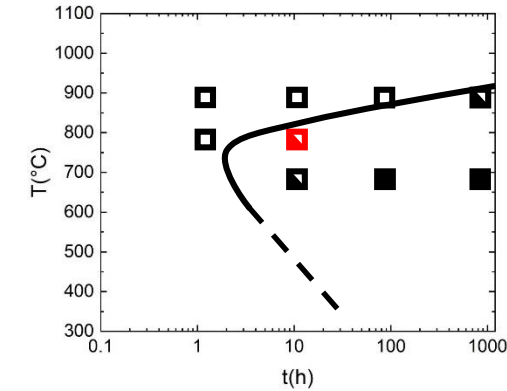
Precipitates were observed almost along some of the grain boundaries



Upper part of the sheet



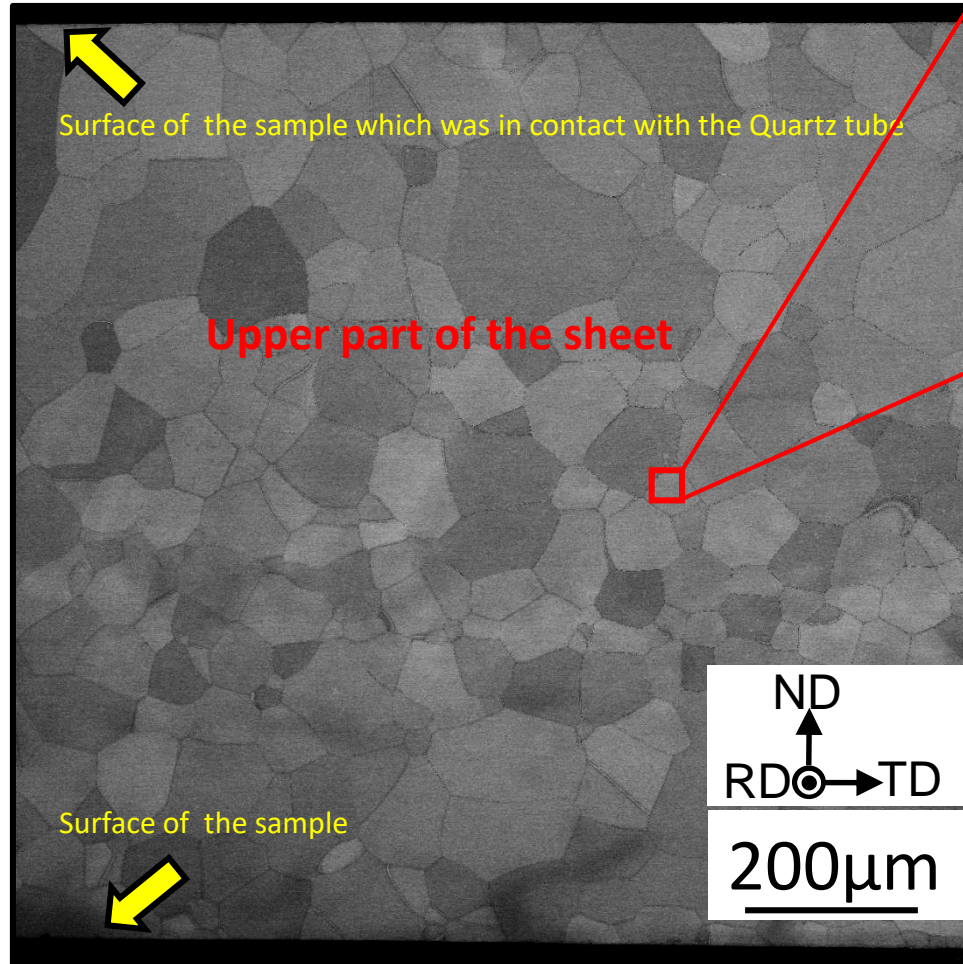
Bottom part of the sheet



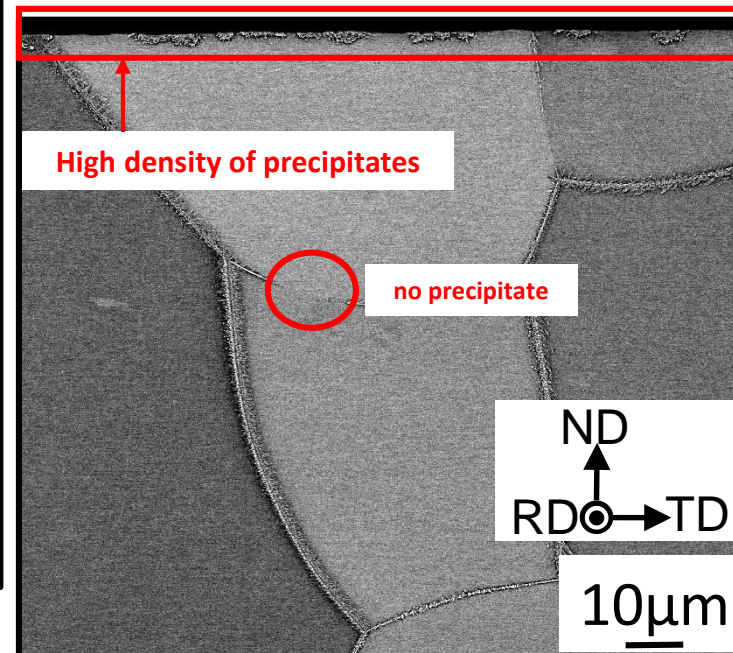
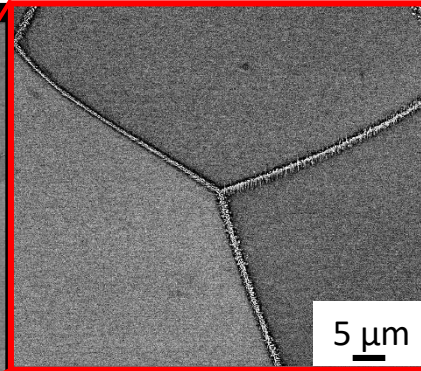


# HT-700°C-10h

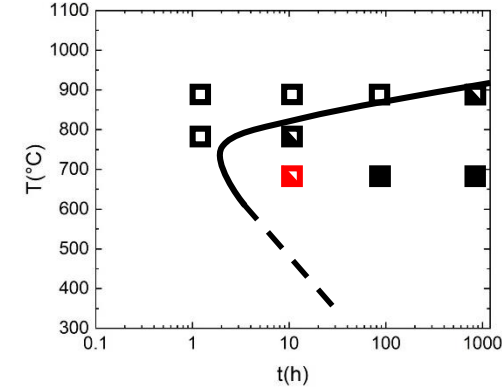
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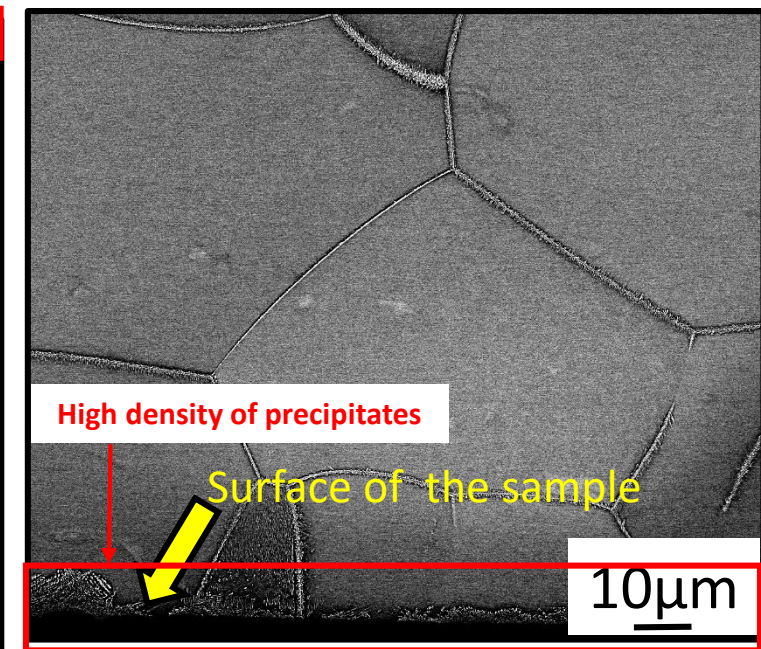
Precipitates were observed almost along all the grain boundaries



Upper part of the sheet



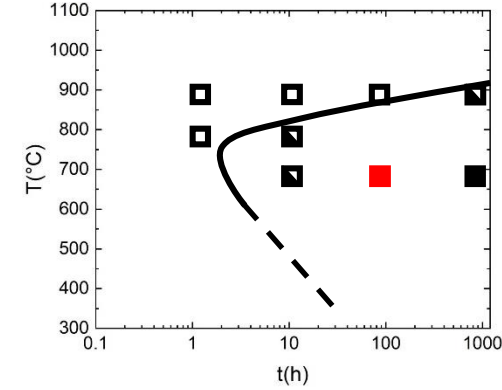
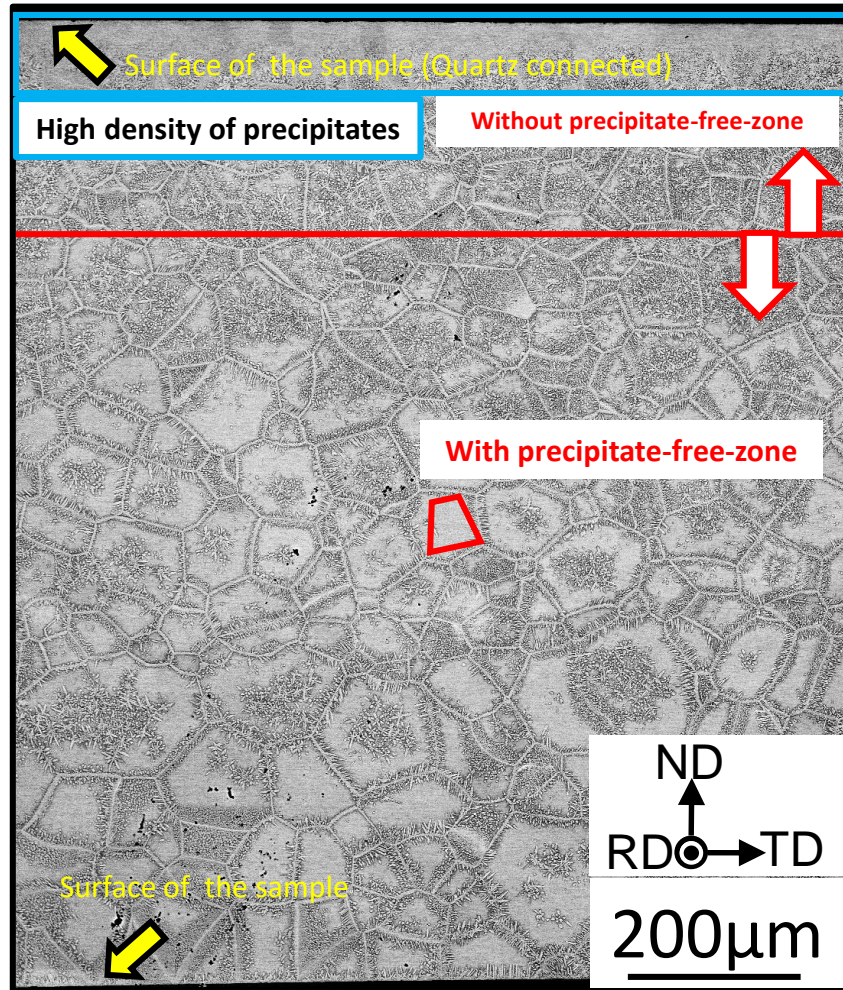
Magnification 1000x



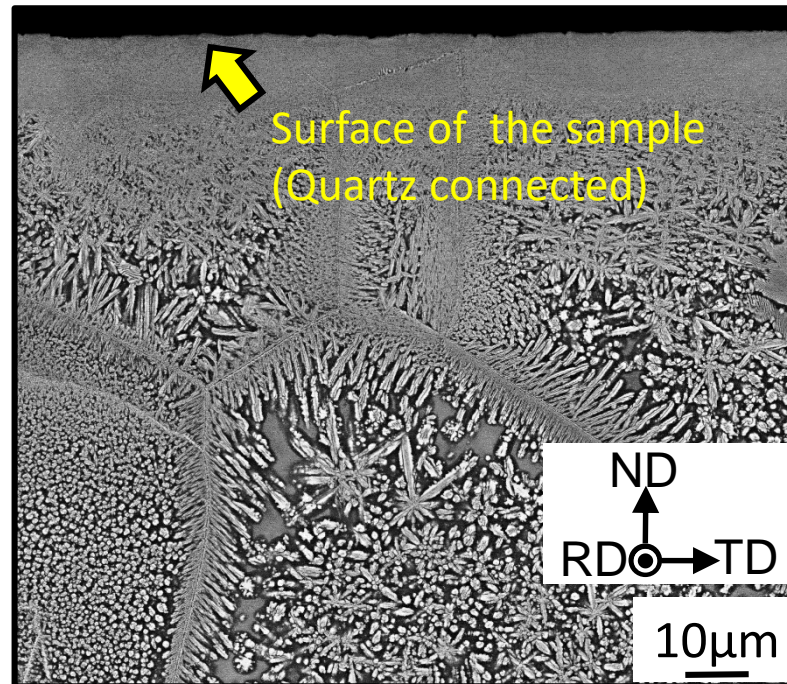
Bottom part of the sheet



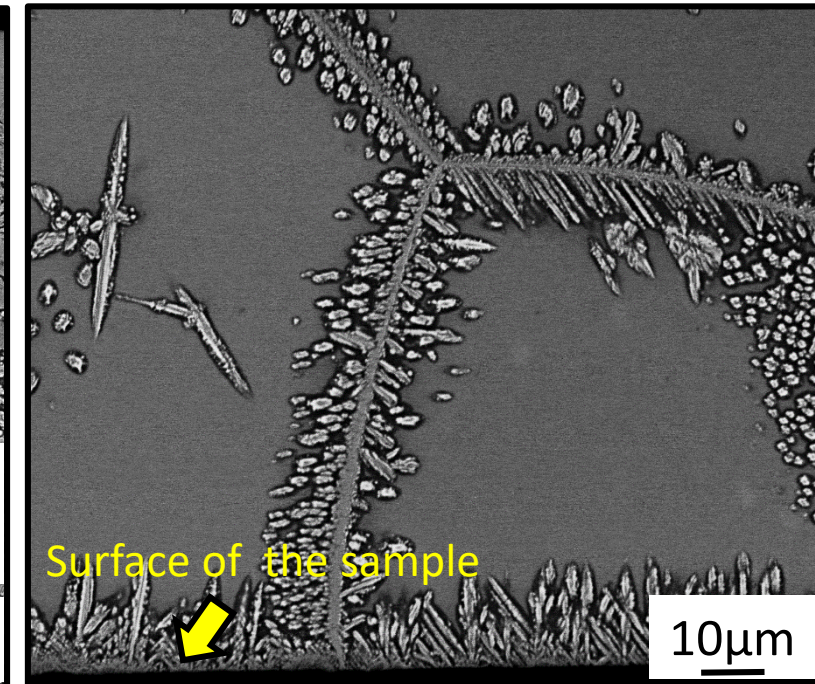
Magnification 70x



Upper part of the sheet



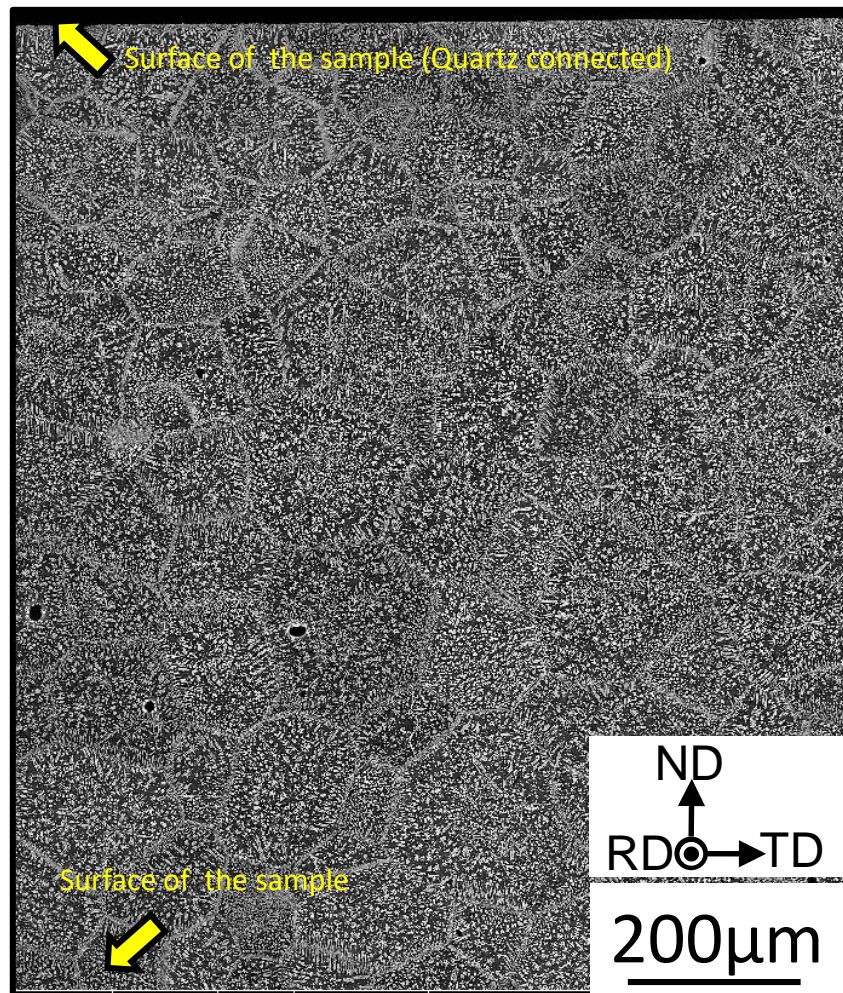
Bottom part of the sheet



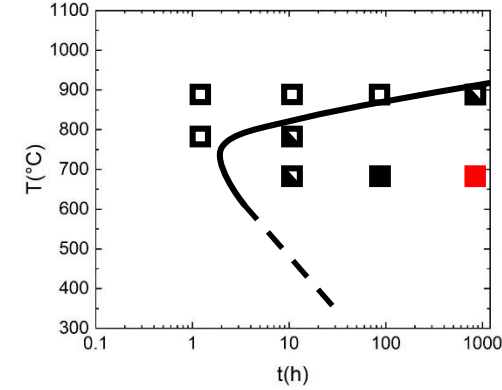
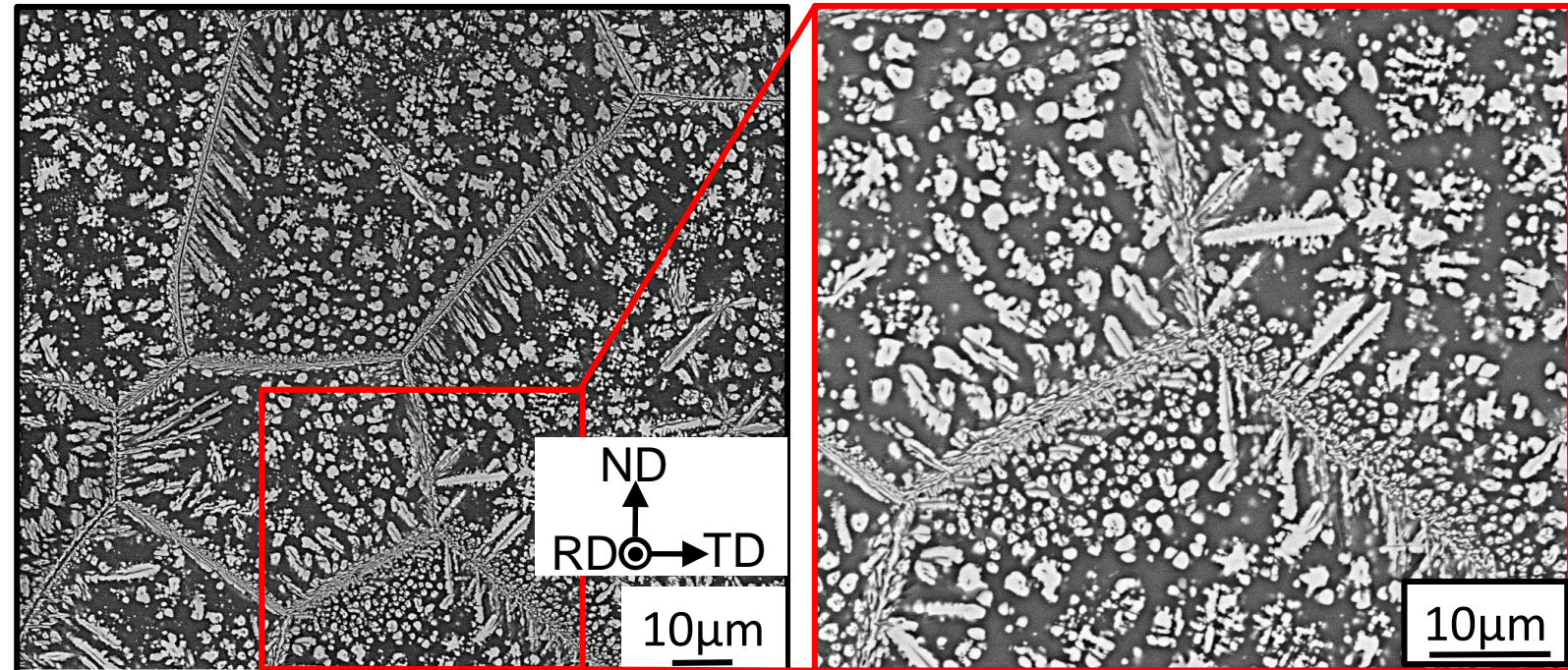
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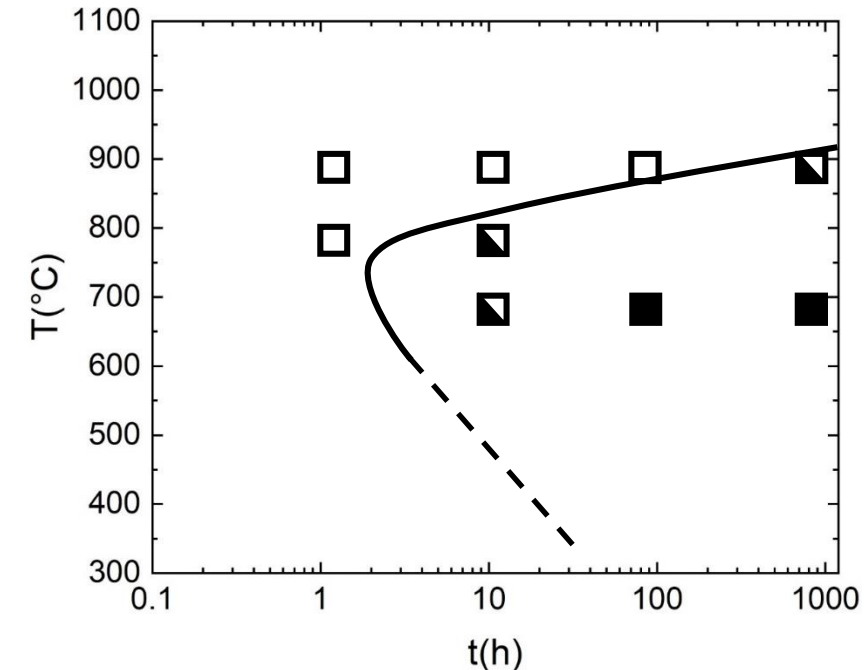
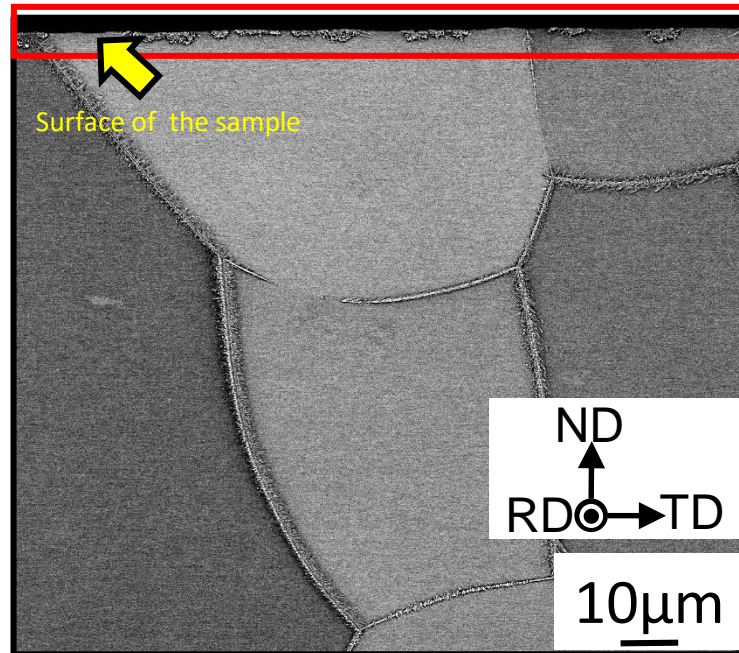
## Sheet overview



## Middle part of the sample

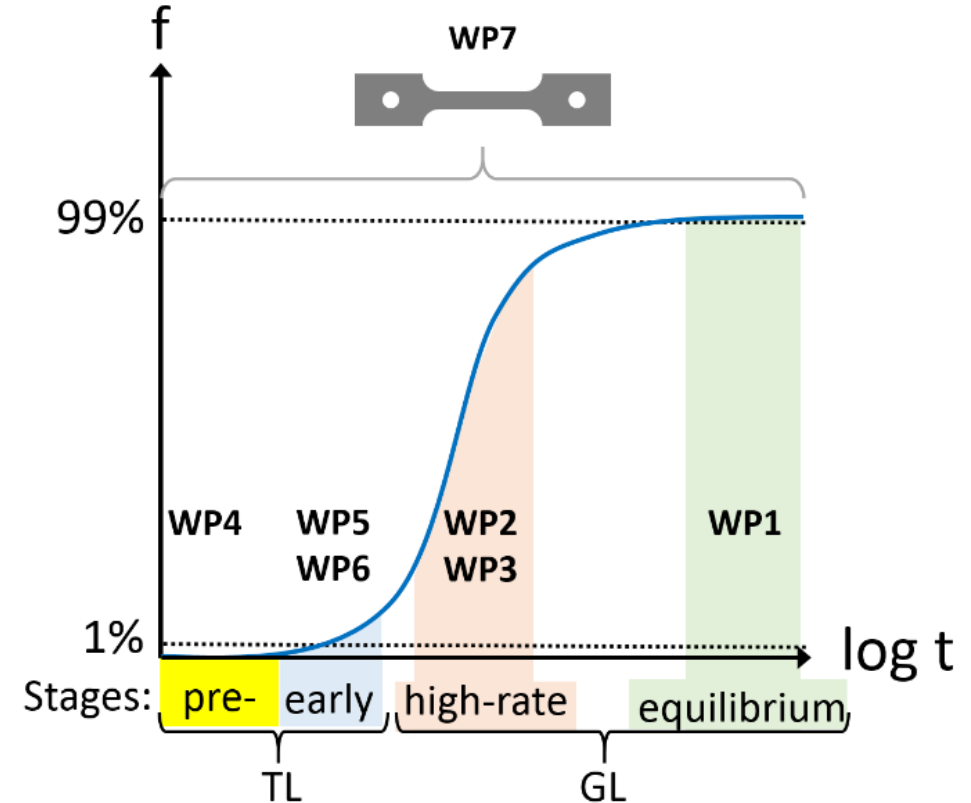
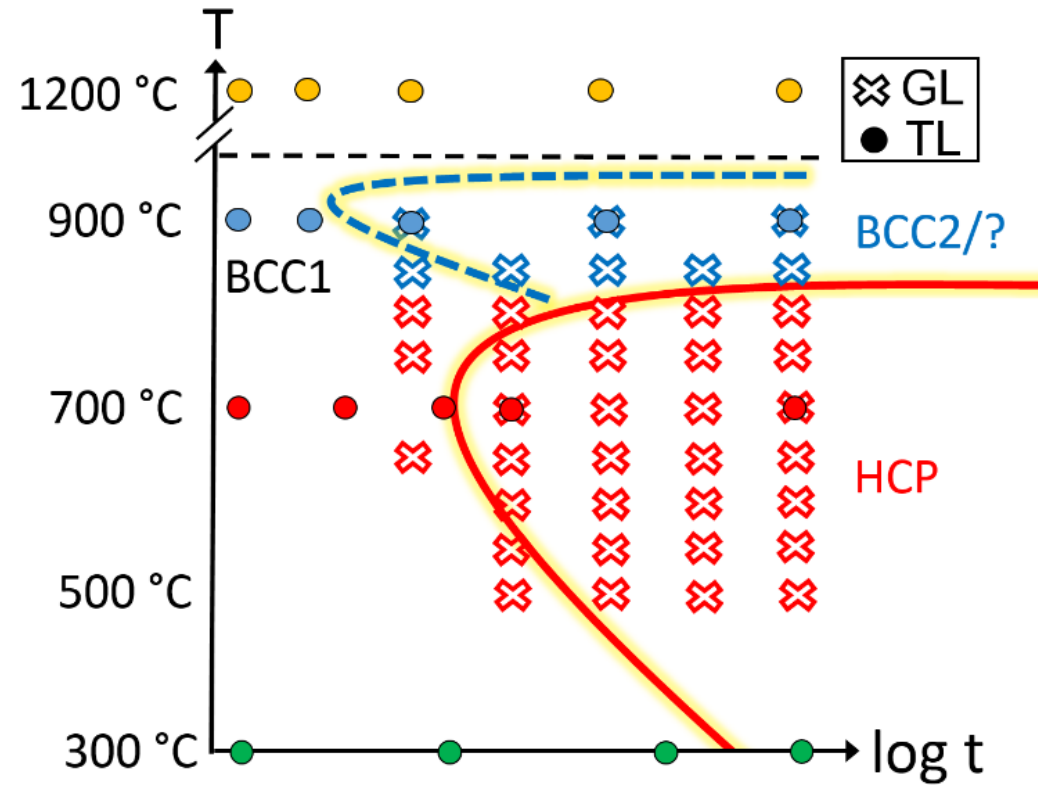






- Even low amount of interstitial elements (contamination) like Oxygen or Nitrogen during annealing cause forming of precipitates on the surface of the annealed samples.
- At 900°C after 10h and also 100h no precipitate was observed, but after 1000h precipitates were observed.
- At 800°C after 1h, no precipitate was observed but after 10h the precipitates could be seen once again along the grain boundaries.
- At 700°C after 10h, precipitates were observed along almost all the grain boundaries and after 100h there were present also within the grains.





# Collaborations



Lehrstuhl Werkstoffwissenschaft  
materials science and engineering 23

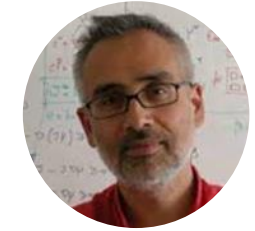
## International collaborations



J.-P. Couzinié



E.P. George



B. Appolaire

## National collaborations



S. Divinski



G. Wilde



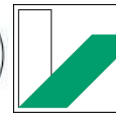
K. Durst



D. Raabe



F. Körmann



U. Glatzel



M. Feuerbacher

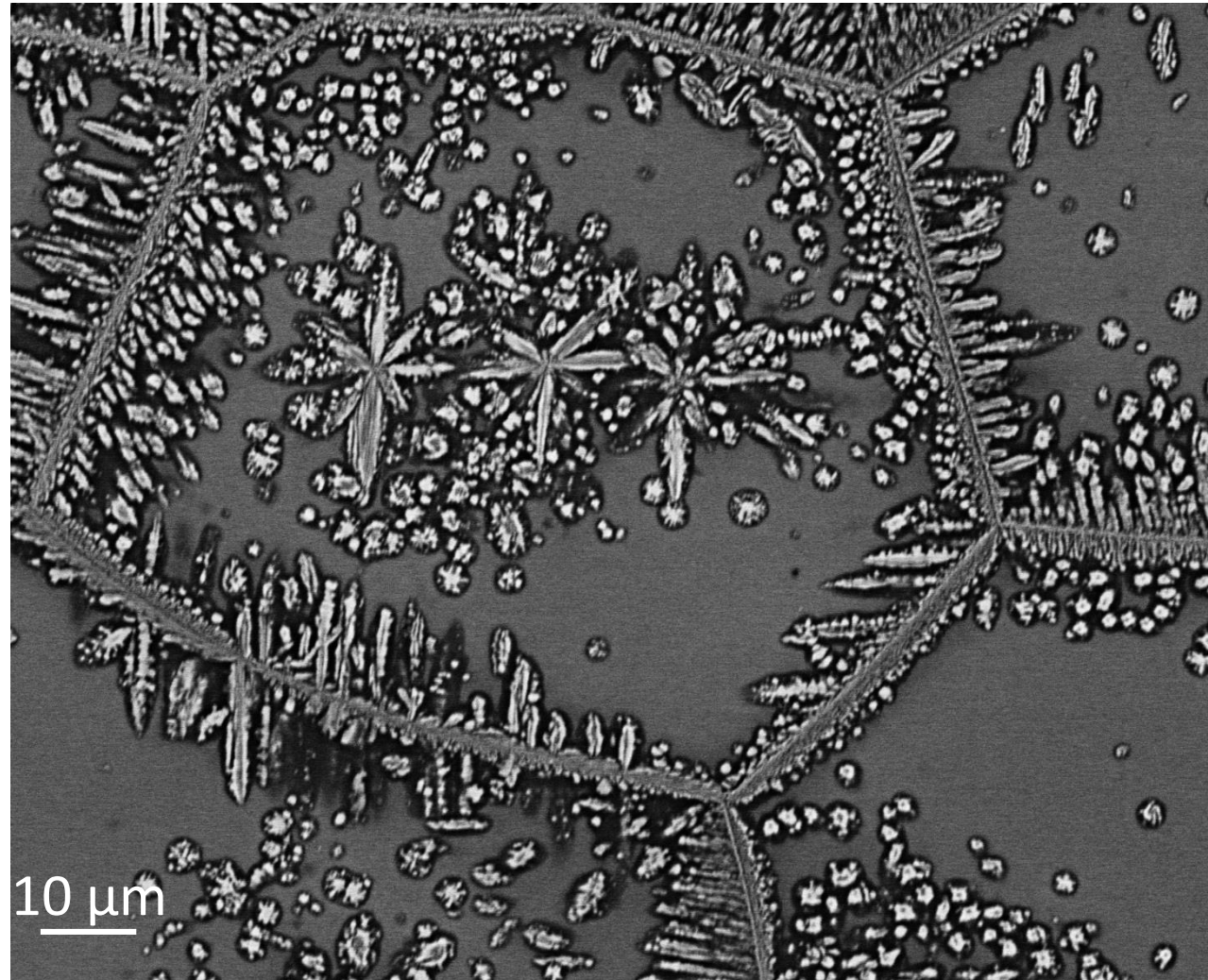


G. Eggeler



J. Frenzel





Thank you for your attention