Synthesis and characterization of single phase solid solution HfNbTaTiZr

T. Hanemann¹, A. Srinivasan Tirunilai², K.-P. Weiss¹, A. Kauffmann², J. Freudenberger³, M. Heilmaier²

¹Institute for Technical Physics (ITEP), Karlsruhe Institute of Technology (KIT), Eggenstein-Leopoldshafen, Germany
²Institute for Applied Materials (IAM-WK), Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany
³Leibniz Institute for Solid State and Materials Research Dresden (IFW Dresden), Dresden, Germany

Institute for Applied Materials (IAM)
SPP-Subgroup meeting HEA mech. Properties, Dresden, 09.11.2018
Background and Motivation

- Investigate deformation behavior of bcc HEAs
  - HfNbTaTiZr as model system (RT ductility, cold workability)

- Special emphasis on solid solution strengthening
  - Interaction of dislocations with local atomic arrangement
  - Influence on deformation mechanisms, e.g. twinning

- Single phase solid solution
- Reproducible initial microstructure

[Rao et al., Acta Mat. 125, 2017]
Project context

Materials synthesis/characterization of initial state

Mechanical Testing
RT – 4.2K

Comparison with deformation theories

\[ \delta = \sqrt{\sum x_i \left(1 - \frac{r_i}{\bar{r}}\right)^2} \]
Material synthesis

Arc melting

Homogenization

Rotary swaging

Recrystallization
Microstructure - ascast

- Single phase bcc structure
- Inhomogeneous distribution of elements between dendritic and interdendritic regions

→ Homogenization
Material synthesis

Arc melting

Homogenization

Rotary swaging

Recrystallization

Set1:
1100°C, 48h, fused silica, furnace cooling

Set2:
1200°C, 48h, fused silica, water quenching
Microstructure - Homogenized

- Set1: 1100°C, 48h, fused silica, furnace cooled

- Single phase bcc by XRD
- Grain boundary phase in BSE
Microstructure - Homogenized

- Set1: 1100°C, 48h, quartz tube, furnace cooled

- Single phase bcc by XRD
- Grain boundary phase in BSE → Zr/Hf/O-enriched
Explanations for secondary phase formation

1. Stabilization of Zr/Hf-rich phase by oxygen:
   - Increased O-content after homogenization
   - No clear correlation between second phase and O-content

2. Decomposition of solid solution during slow cooling:
   - Homogenization at single phase field
   - Decomposition at $T < 985^\circ$C

→ water quenching

[Senkov et al., Acta Mat. 68, 2014]
Microstructure - Homogenized

- Set2: 1200°C, 48h, fused silica, water quenched

→ Single phase bcc by XRD and BSE
Material synthesis

Arc melting

Homogenization

Rotary swaging

Recrystallization

Set1: 1100°C, 48h, fused silica, furnace cooling

Set2: 1200°C, 48h, fused silica, water quenching
Microstructure – Rotary Swaged

- Deformation to $\varphi=1.39$
- Evolution of $<110>$ texture
  - $\rightarrow$ expected for bcc
Material synthesis

Arc melting

Homogenization

Rotary swaging

Recrystallization

Set 1: 1100°C, 48h, fused silica, furnace cooling

Set 2: 1200°C, 48h, fused silica, water quenching

?
Summary

- Cooling rate after heat treatment in single phase field critical for single phase retention → water quenching
- Necessity of microstructural investigation on different length scales
Outlook

- Investigation of chemical homogeneity on smaller length scales
  → e.g. APT

- Complete standard material synthesis route
  → Reproducibility?

- Mechanical tests at RT down to 4.2K
Thank you for your attention

Many thanks to:

DFG
Deutsche Forschungsgemeinschaft