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# Understanding Phase Transformations on the way to $Nb_3Sn$

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with contributions of Jonas Lachmann, Alexander Walnsch, Stefan Martin, Mario Kriegel

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# Freiberg, not Freiburg in Germany

sources: Wikipedia, TUBAF

# Topics of Physical Metallurgy group

Metastable phases and related microstructures; thermodynamic description, **Calphad Method**

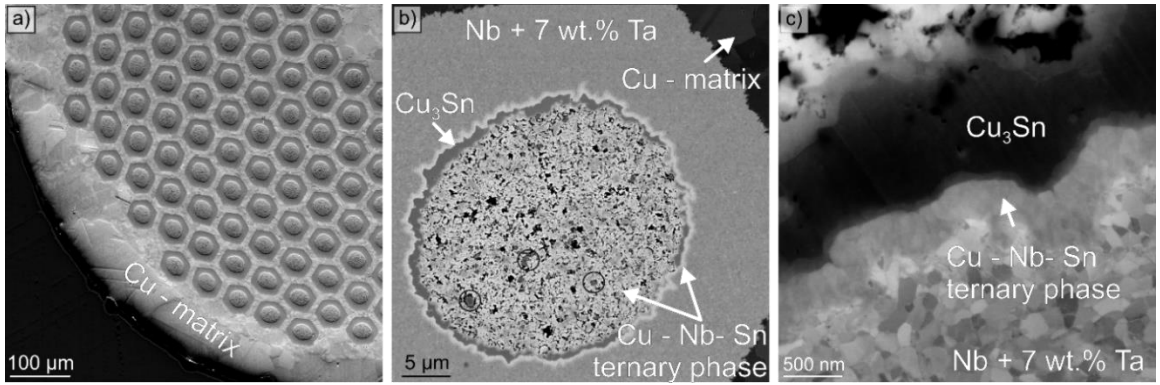
X-ray diffraction and electron backscatter diffraction (EBSD) on defective phases and peculiar microstructures

methodology

Phase transformations in alloys with interstitial elements; heat treatment of steel and cast iron

**Intermetallic phase** formation during Sn based soldering and in Al alloys

systems




A. Walnsch,
Internship @CERN
in the meantime
PostDoc in our group

Intermetallics 80 (2017) 16–21

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# The crystal structure of $(Nb_{0.75}Cu_{0.25})Sn_2$ in the Cu-Nb-Sn system

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# 2018-2023 FCC/HFM-funded research activities

Topics

- Phase equilibria in the Cu-Nb-Sn system, thermodynamic modelling
- Model diffusion couples to mimic processes in cables
- Influence of elements as Ta, Hf, O on phase formation and microstructure
- Microstructure investigations of processed wires

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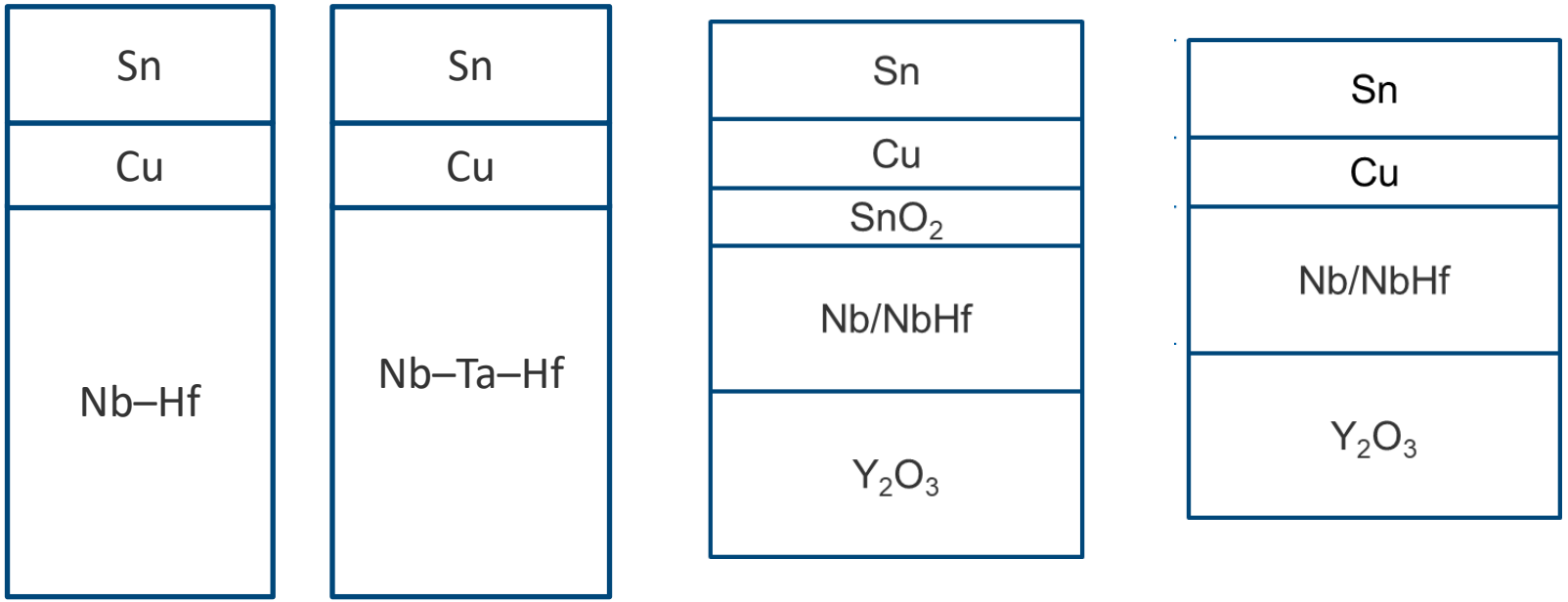
J. Lachmann

# Main: Model diffusion couples with different architectures




- Solid substrate (Nb, Nb alloy, alternatively largely inert $Y_2O_3$); covered by electroplating or PVD
- Also a few bulk alloys have been considered

# Methods for investigated developing microstructures

optical microscopy

SEM

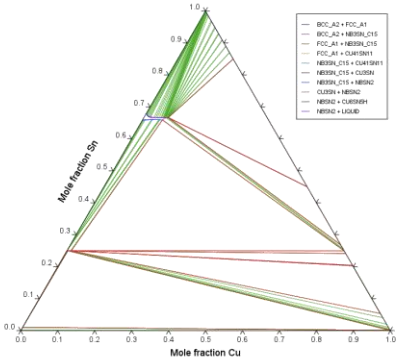
TEM

XRD

Calorimetry

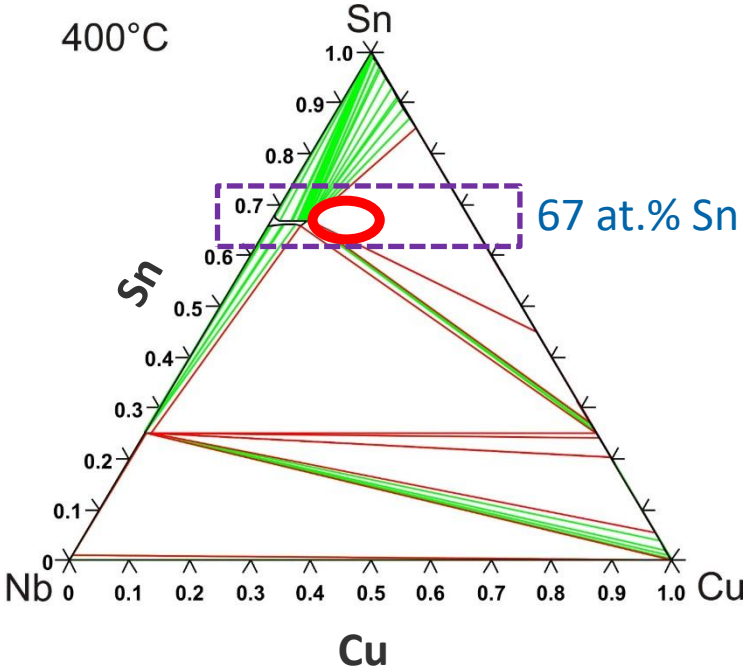
&

thermodynamic modelling



# Phase equilibria in the Cu-Nb-Sn system, thermodynamic modelling

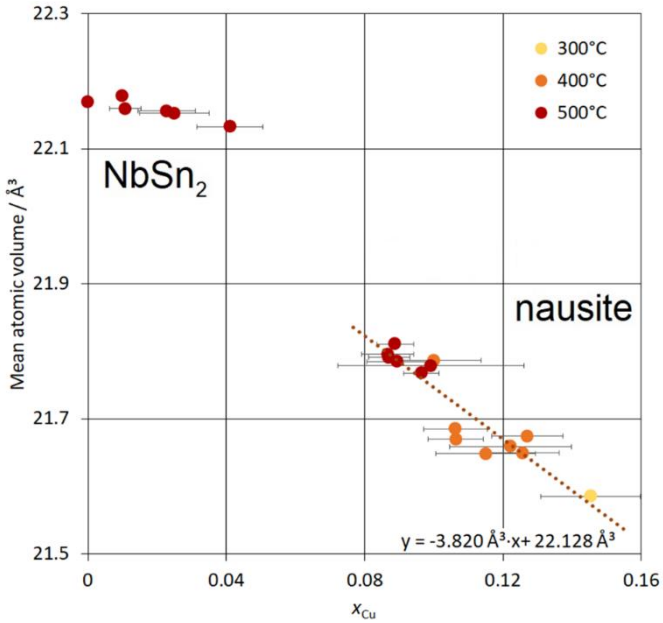
## $NbSn_2$ and nausite ($Nb_{1-x}Cu_xSn_2$); towards a new CALPHAD description of the Nb-Cu-Sn system

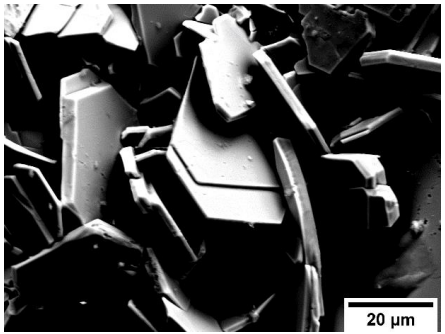


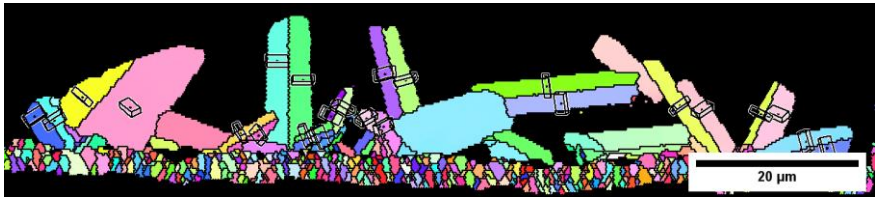

- Related crystal structures (polytypes); reliable distinction necessary
- XRD/EBSD methodology adapted

# Basis assessment of phase equilibria

- various samples containing $NbSn_2$ and/or nausite
- Further data

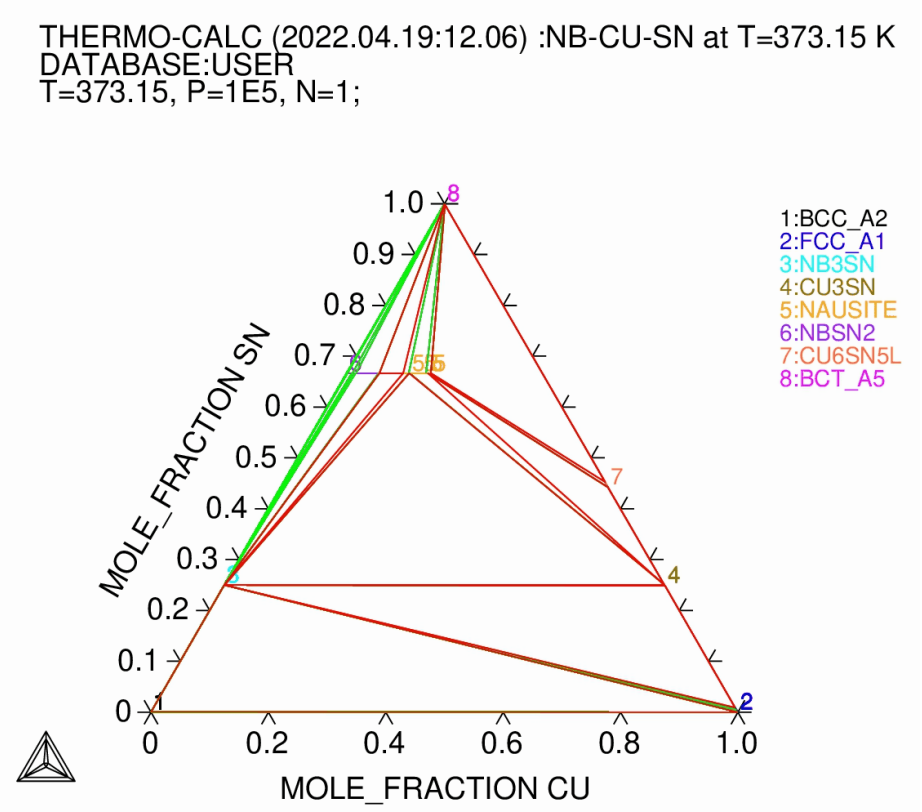



[1] J. Lachmann, M. J. Kriegel, A. Leineweber, S.-L. Shang, Z.-K. Liu, “Thermodynamic re-modelling of the Cu–Nb–Sn system: Integrating the nausite phase“, Calphad 77 (2022)

# New (public) CALPHAD database Cu-Nb-Sn including nausite (supplemented by DFT calculations)



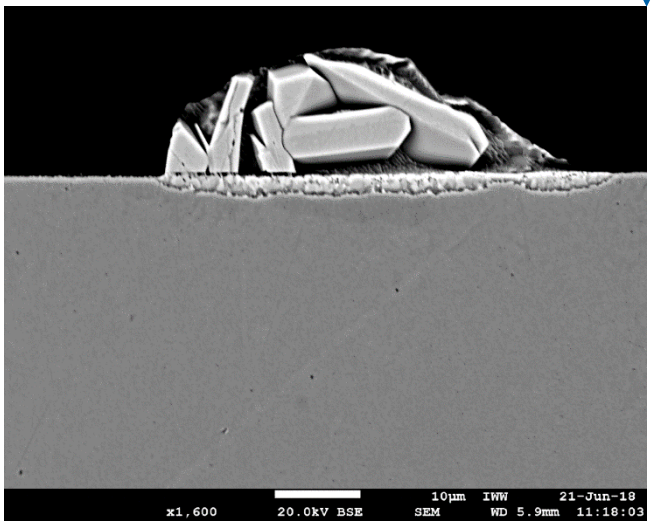

Further activities including Ta, Hf; to be published (there is, e.g. (Ta, Cu)$Sn_2$)



[1] J. Lachmann, M. J. Kriegel, A. Leineweber, S.-L. Shang, Z.-K. Liu, Thermodynamic re-modelling of the Cu–Nb–Sn system: Integrating the nausite phase, Calphad 77 (2022) 102409

## Comments on diffusion couple techniques

- Access to large-scale microstructures and simplified diffusion geometry
- Fight against unwetting effects in the case of melting
- Learning process for proper design to avoid this




Nb+plated Sn

# Influence of elements as Ta, Hf, O on phase formation and microstructure

## Diffusion couple design

200°C } formation of $Cu_6Sn_5$

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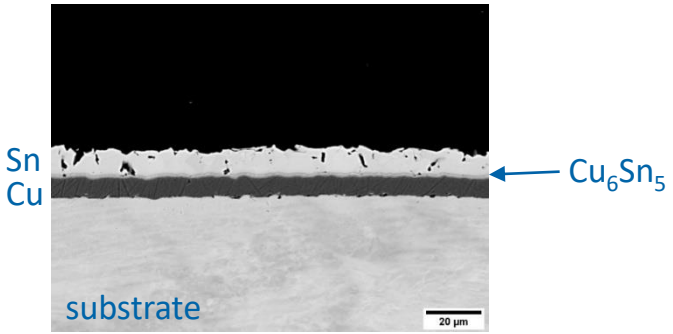
400°C } formation of nausite
450°C

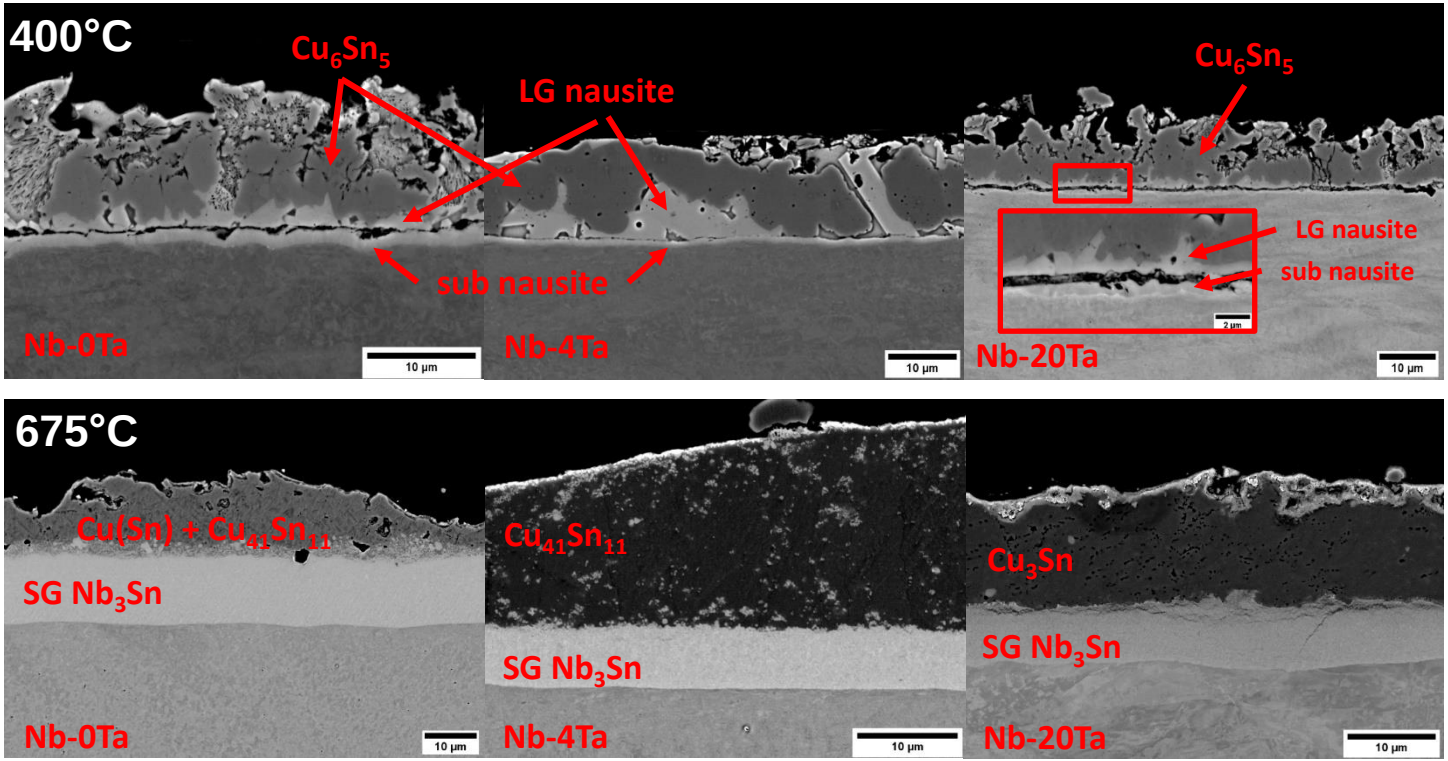
600°C } formation of $Nb_3Sn$
675°C
750°C

| Sn | Sn | Sn |
|---|---|---|
| Cu | Cu | Cu |
| Nb–Ta:<br>0 / 4 / 20<br>at.% Ta | Nb–Hf:<br>2 / 4 / 20<br>at.% Hf | Nb–Ta–Hf:<br>4–0<br>4–0.7<br>4–1<br>4–2<br>2–0.5<br>at.% Ta–Hf |



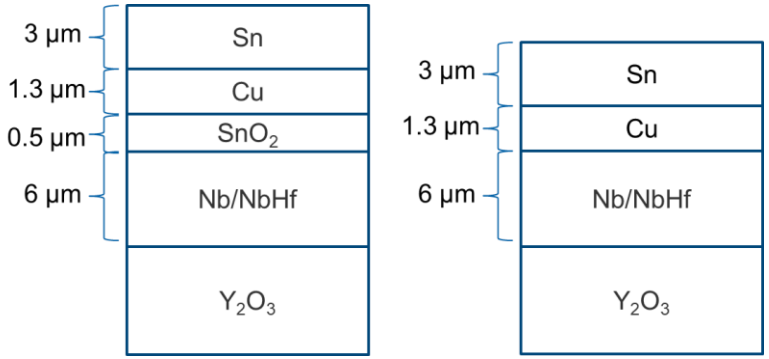

- Nb alloy substrates
- Cu and Sn deposited via PVD
- heat treatment in Ar atmosphere
- quenching in water
- embedding as cross-section

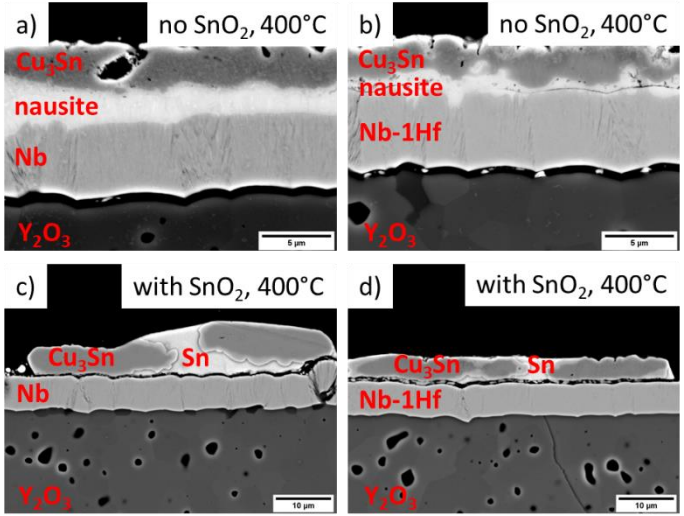
# E.g. investigations on the influence of Ta




- Ta decreases nausite and $Nb_3Sn$ layer growth

# Effect of O on phase formation and microstructures in the Cu-Nb-Sn-(Ta, Hf) system

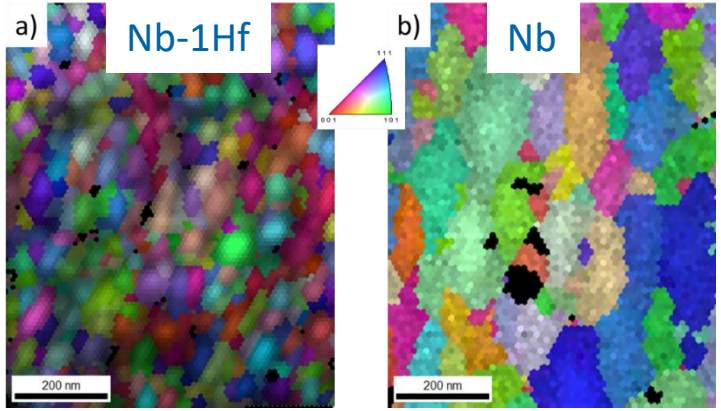



- deposition of Nb/Nb alloy on $Y_2O_3$ substrate using PVD
- deposition of $SnO_2$ using magnetron sputtering
- Cu, Sn again using PVD
- heat treatment in Ar atmosphere

675°C

$Nb_3Sn$ grown from ($SnO_2$ free)




$Nb_3Sn$ size not determinable in case of samples with $SnO_2$

## Summary

- Better understanding of thermodynamics and kinetics of phase formation
  (a) CALPHAD description
  (b) coarse and fine scale versions of $Nb_3Sn$ and of other intermetallic phases
  (c) $SnO_2$ as O source; O is there and reduces grain size. But in particular Hf may have an effect by its own.
- Further development of diffusion couple techniques; further concepts to be tested in the future…?!

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