Karlsruhe Institute of Technology

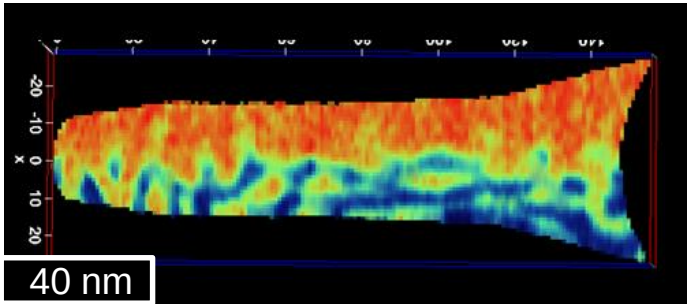
CHALMERS

Institute for Applied Materials

Atomsondentomografie der Metall-Aluminiumoxid-Grenzfläche auf NiAlCr-Legierungen

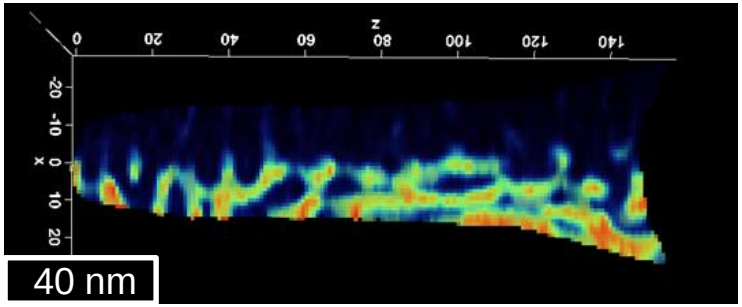
Torben Boll*,Olof Bäcke, Kinga A. Unocic, Bruce A. Pint, Krystyna Stiller

Al



APT

Ni

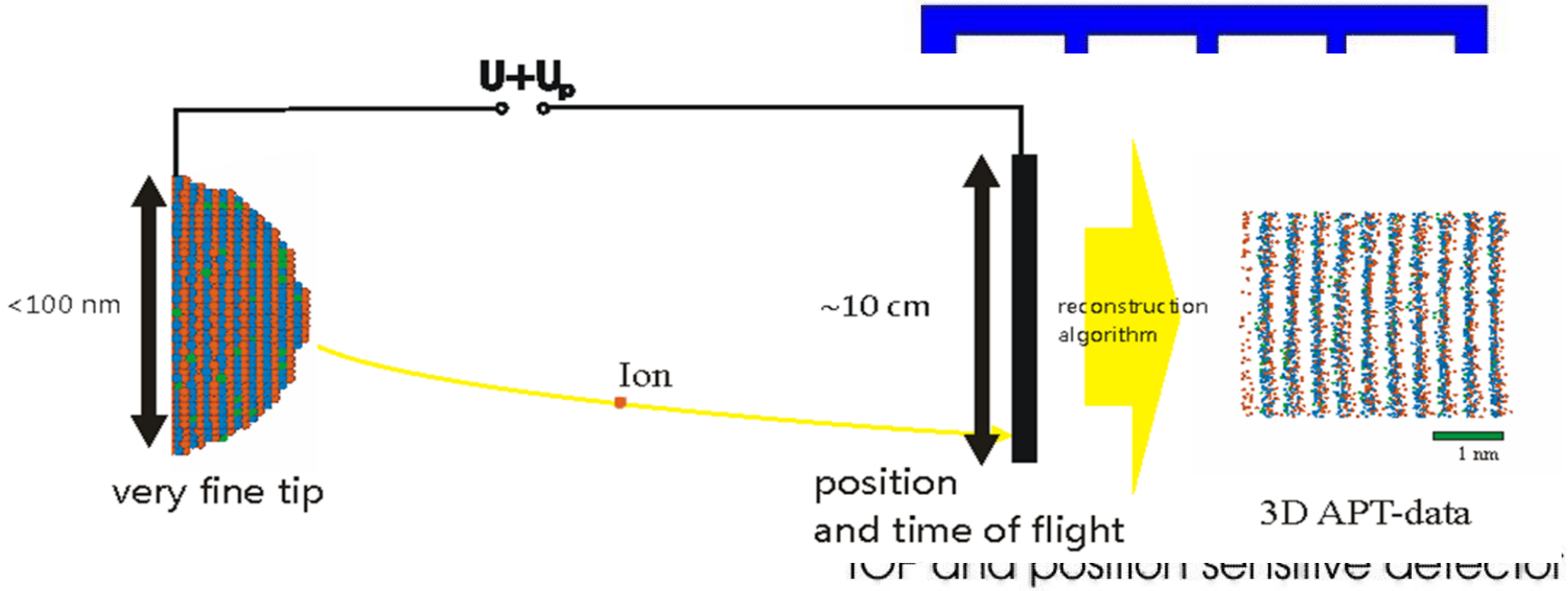


Aalen 15.09.2017

Überblick

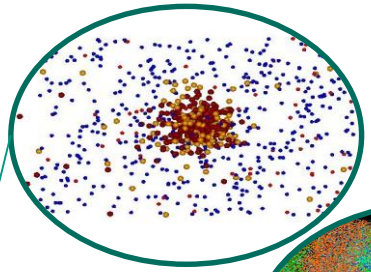
- Ni-Basis-Legierungen: Hohe Temperaturen (z.B. Flugzeugturbinen)
- Stabiles schützendes Oxid: α-Al_2O_3
- Weitere Elemente wie Cr oder reactive Elements (e.g. Y,Hf) werden hinzugefügt um die Mikrostruktur und die Chemie an den Korngrenzen (KG) und Phasengrenzen (PG) zu ändern
- TEM kann qualitativ Segregation an KG bestimmen
- Atomsonde (APT) für quantitative Analysen von KG und PG

Atom Probe Tomography (APT)

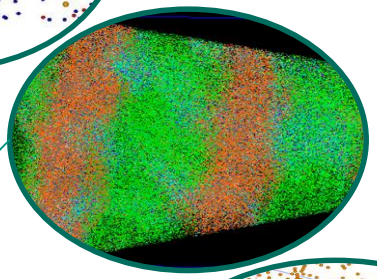


- Hohes Feld, fast stark genug um einzelne Atome zu evaporieren
- Zusätzlicher Puls (Laser oder Spannung): Atom wird evaporiert
- Aus der Flugzeit wird das Masse-zu-Ladungsverhältnis berechnet
- (x,y)- Koordinaten sind vom Detektor bekannt, z wird aus der Ankunftsreihenfolge errechnet

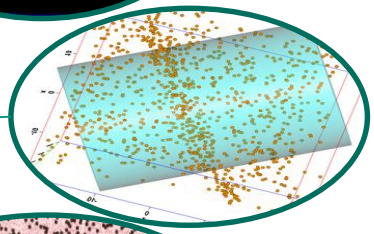
Was kann APT für mich tun?



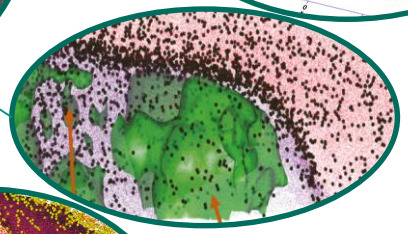
Keimbildung u. -wachstum



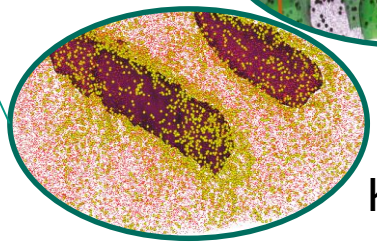
Dünne Schichten



Korngrenzen



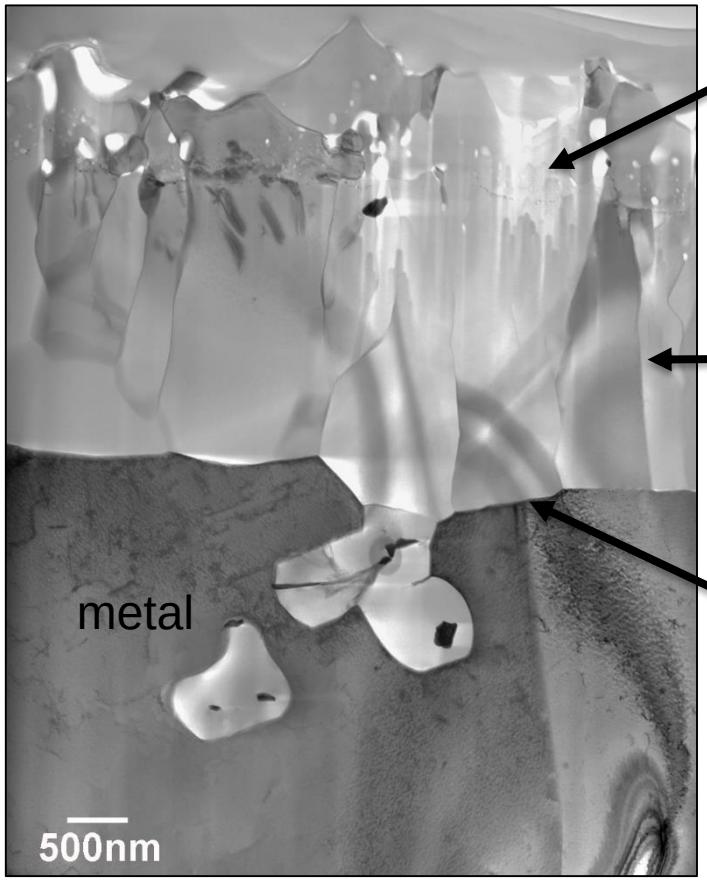
Phasengrenzen



Kohlenwasserstoffverbindungen (schwierig)

Überblick: NiAlCr

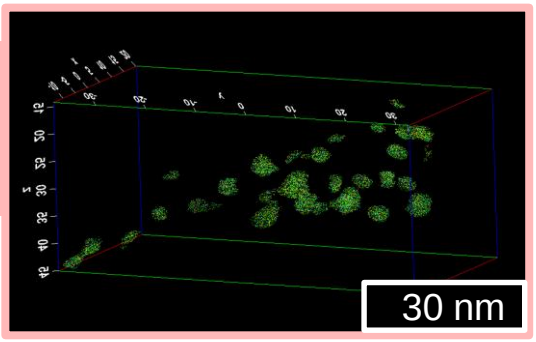
TEM



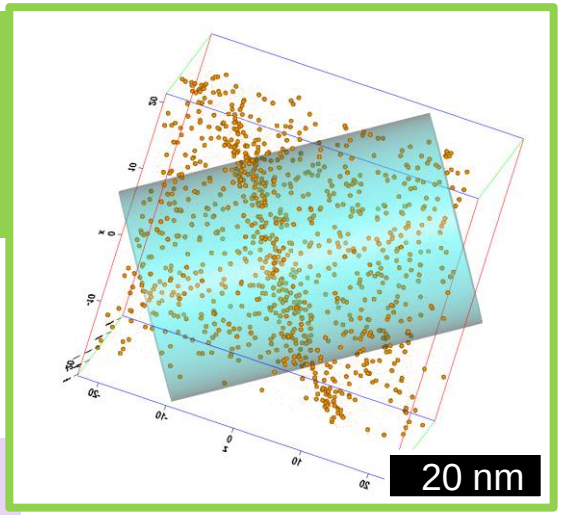
TEM of oxidized NiCrAl after 100h@1100°C

APT

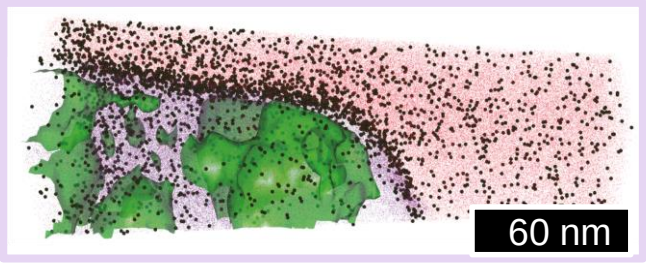
Outer oxide: Phase boundaries (PB) between different oxides



Inner oxide: Grain boundaries (GB) between different Al_2O_3-grains

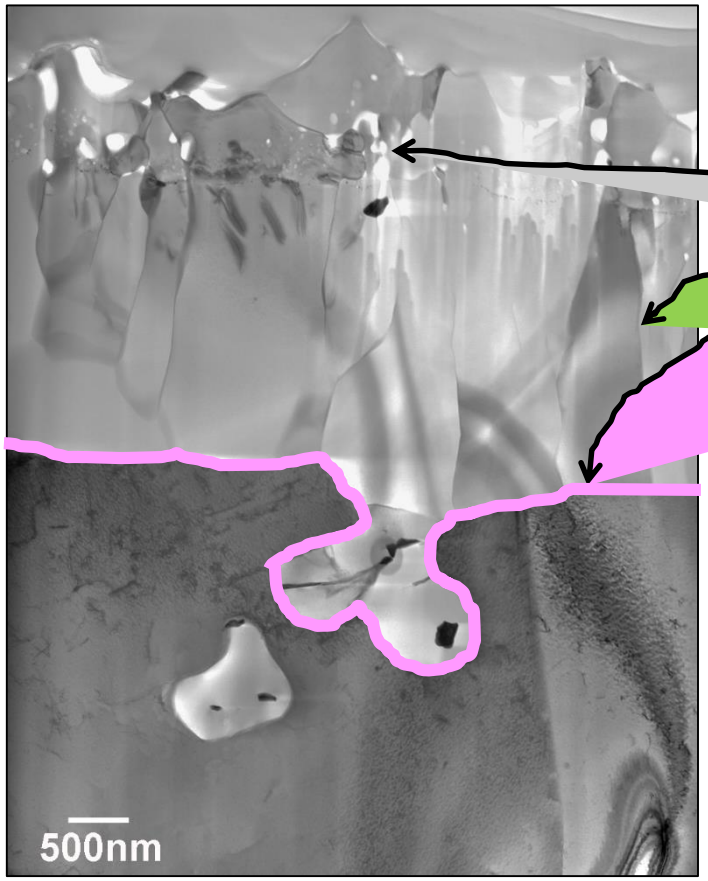


Phase boundary (PB) between the metal and the Al_2O_3-grains



Überblick: TEM

YHfB: $Ni_{60.6}Cr_{16.8}Al_{22.2}Y_{0.015}Hf_{0.037}B_{0.33}C_{0.028}$
YHfTi: $Ni_{61.9}Cr_{15.2}Al_{22.6}Y_{0.020}Hf_{0.043}Ti_{0.31}C_{0.017}$
YHf: $Ni_{62.3}Cr_{15.1}Al_{22.5}Y_{0.03}Hf_{0.04}C_{0.024}$



TEM of oxidized NiCrAl after 100h@1100°C

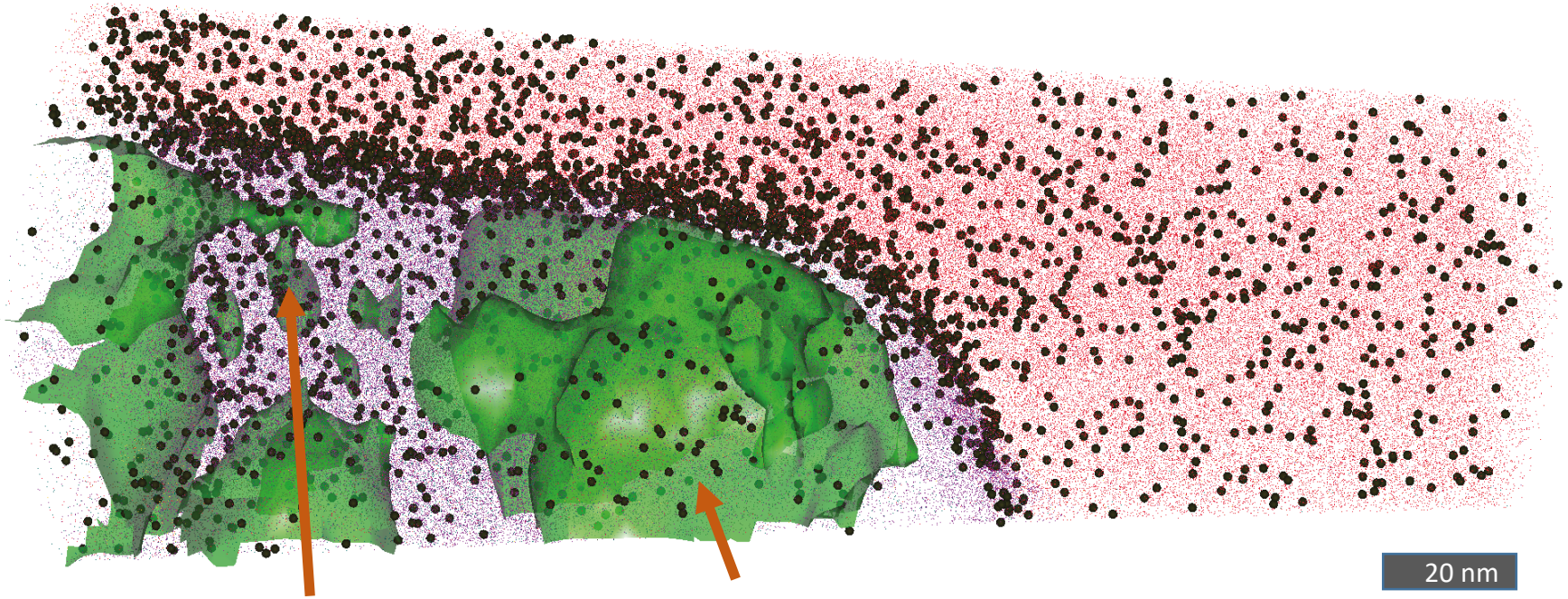
	Metal-Al_2O_3	Al_2O_3-Al_2O_3	Al_2O_3-M_yO_x
Hf	Y	Y	Y
Y	N	Y	Y
Ti	N	N	N
B	N	N	N
Ni		Y	
Cr		Y	

APT: Quantitative Enrichment at different interfaces

BOLL, T., UNOCIC, K. A., PINT, B. A. & STILLER, K. (2017). Microscopy & Microanalysis

Atom Probe: YHfTi M/O Phasengrenze

HfO Cr Ni (isosurface) Al / AlO



Cr-rich grain
$Ni_{59}Al_{16}Cr_{24}Ti_{0.1}$

Al-rich grain
$Ni_{70.2}Al_{21.3}Cr_{5.9}Ti_{0.7}$

- Hf aber kein Ti or Y an Metall-Oxid-Grenzfläche
- γ/ γ` Nanostruktur im Metall. Ti-Anreicherung in γ`

CHALMERS OAK RIDGE National Laboratory KNMF Karlsruhe NANO MICRO Facility KIT Karlsruhe Institute of Technology

Atom Probe: YHfTi M/O Phasengrenze

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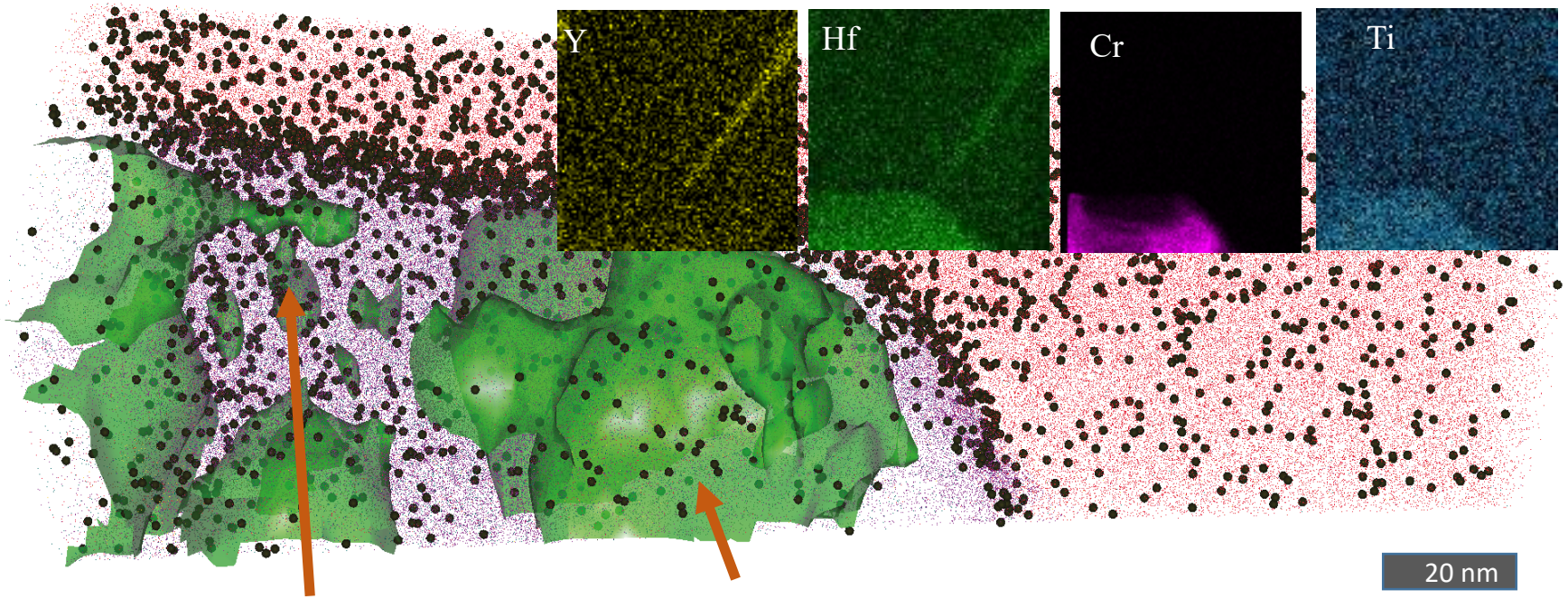

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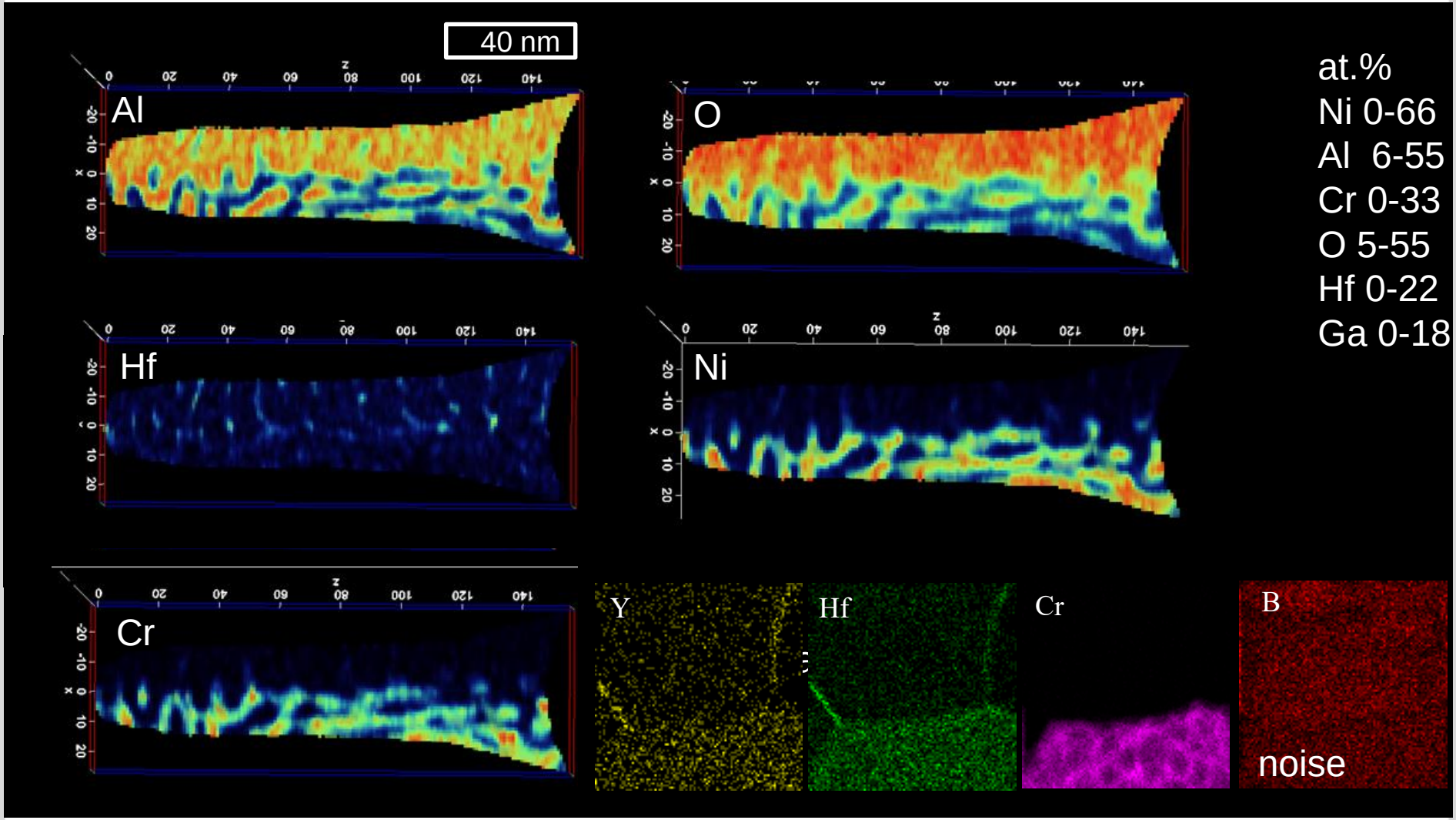
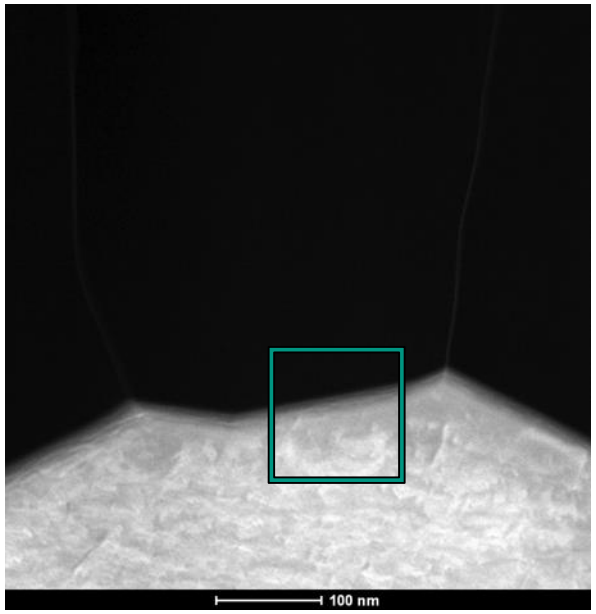
Atom Probe:YHfB: M/O Phasengrenze



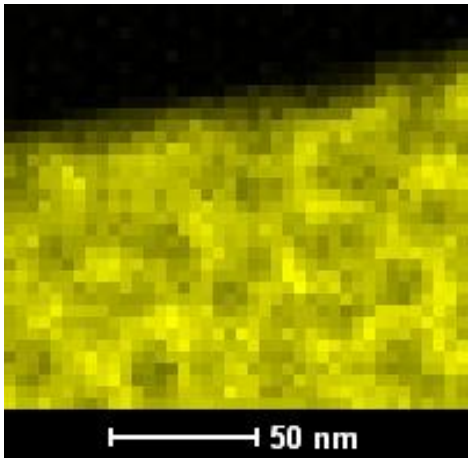
Unerwartete Rauheit der Grenzfläche

HAADF STEM

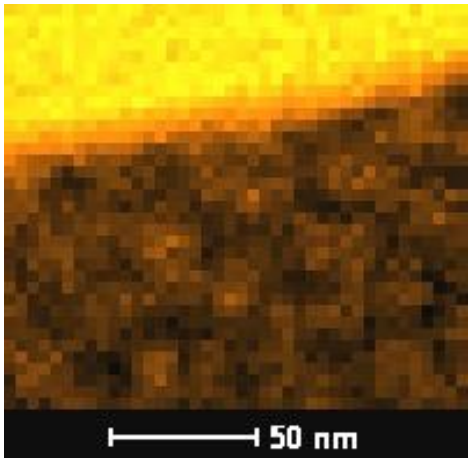


HAADF STEM

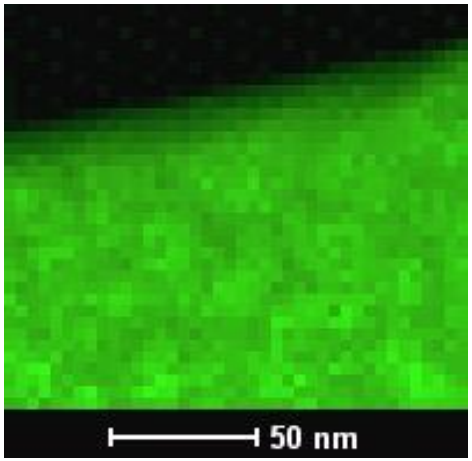
Cr



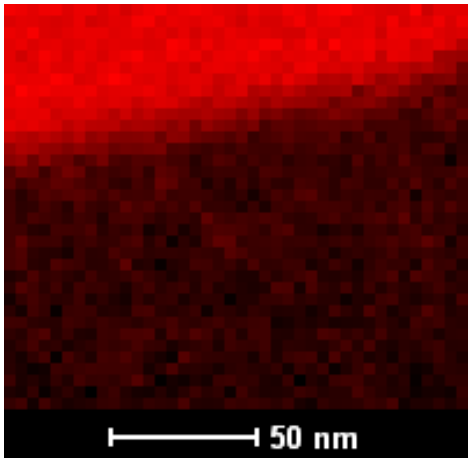
Al



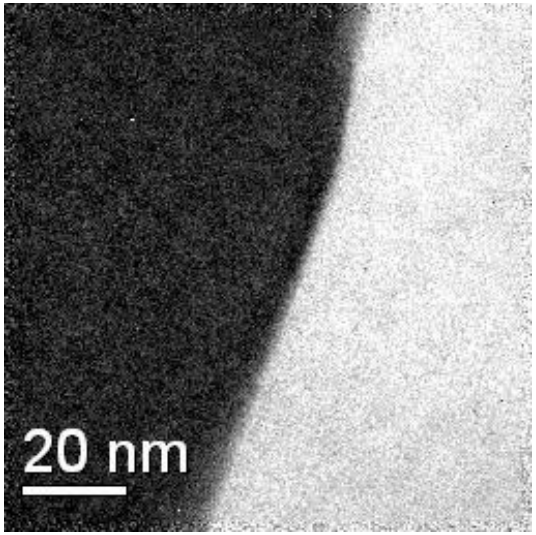
Ni



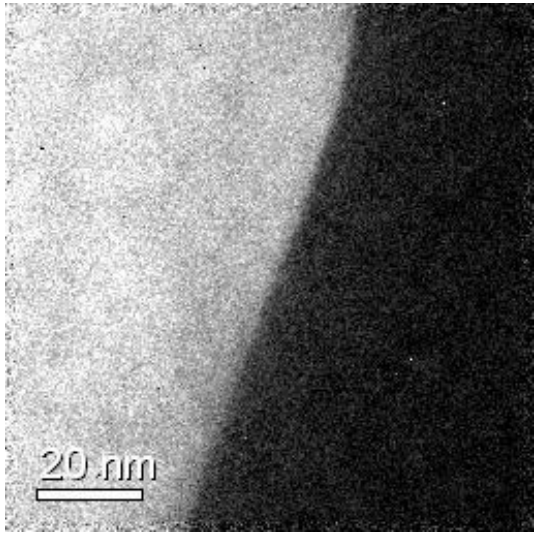
O



TEM



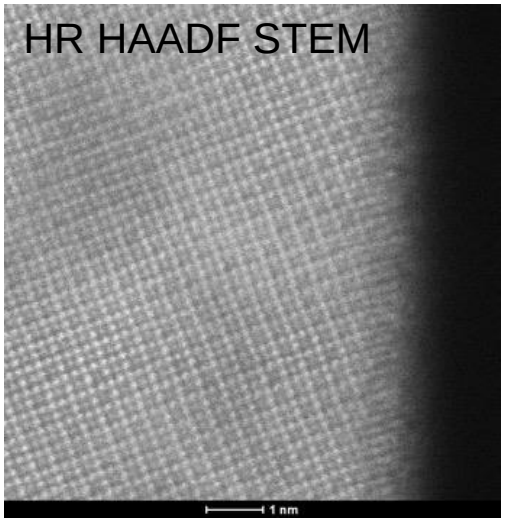
EFTEM - O

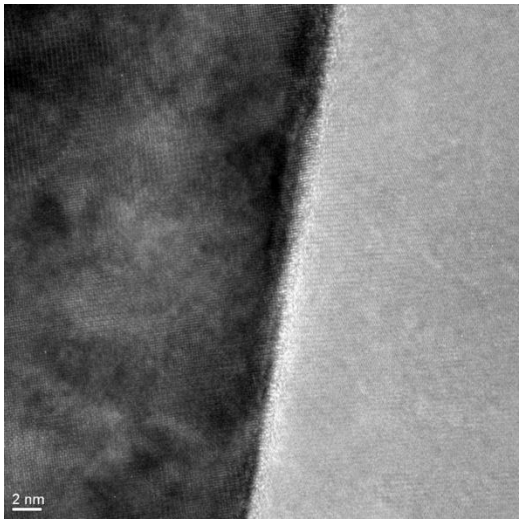


EFTEM - Ni

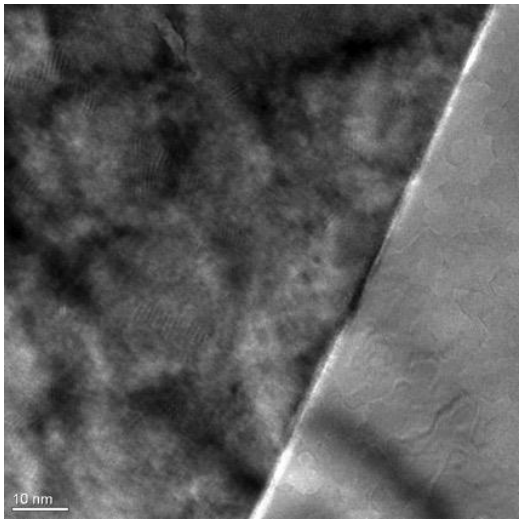
Auch mit EFTEM ist keine O im Ni zu sehen

Regionen mit Moire-Effekt im Metall

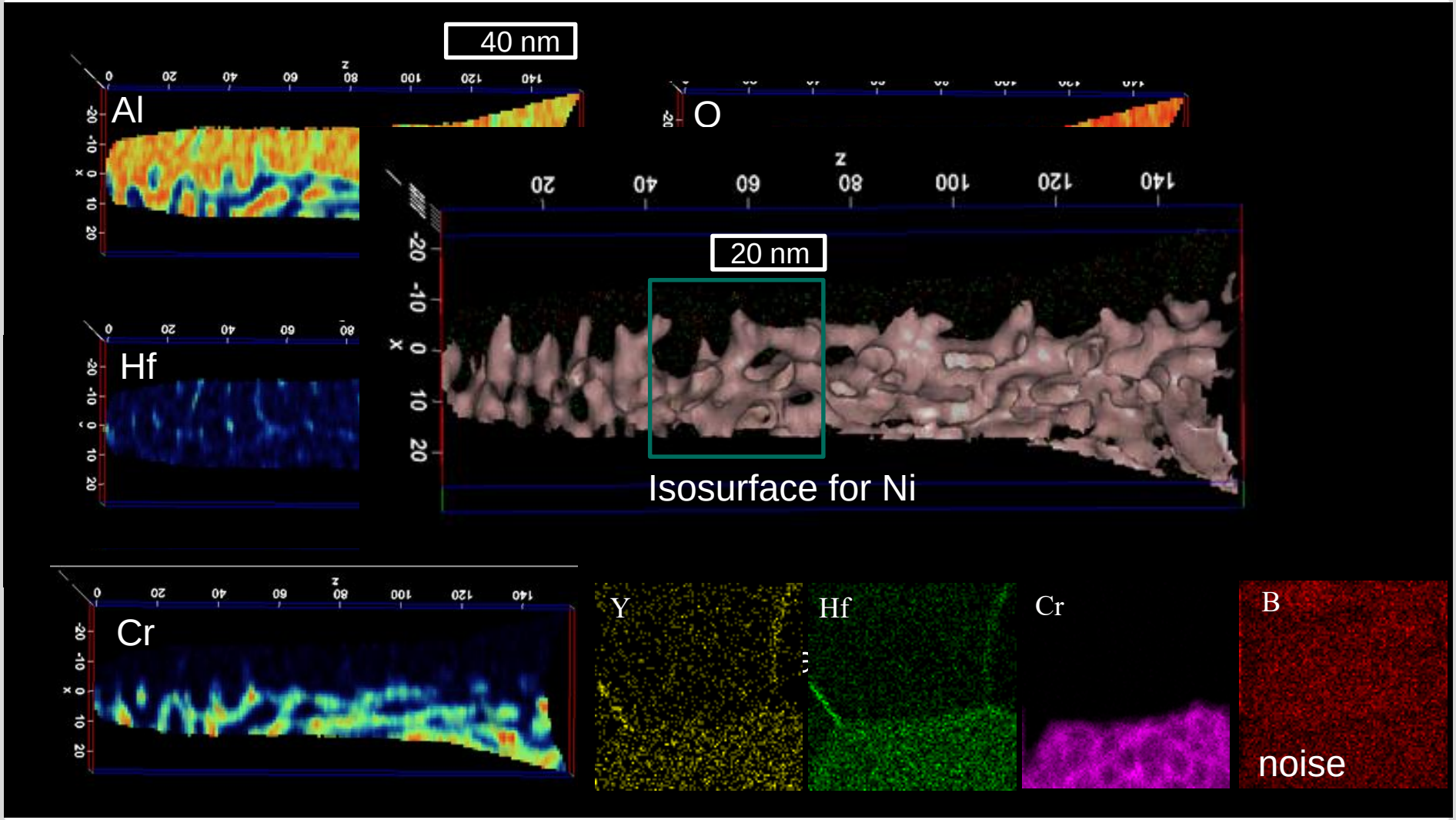




HRTEM

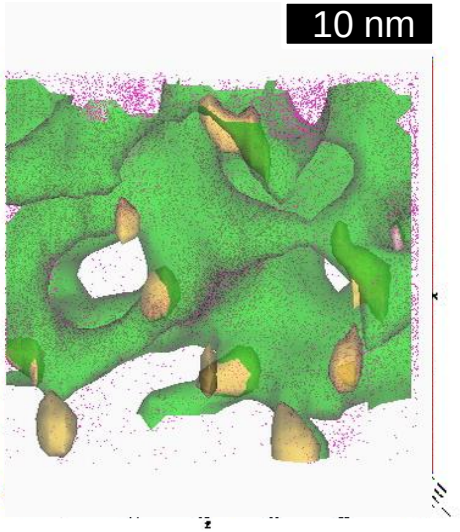


Atom Probe:YHfB: Metal-oxide interface

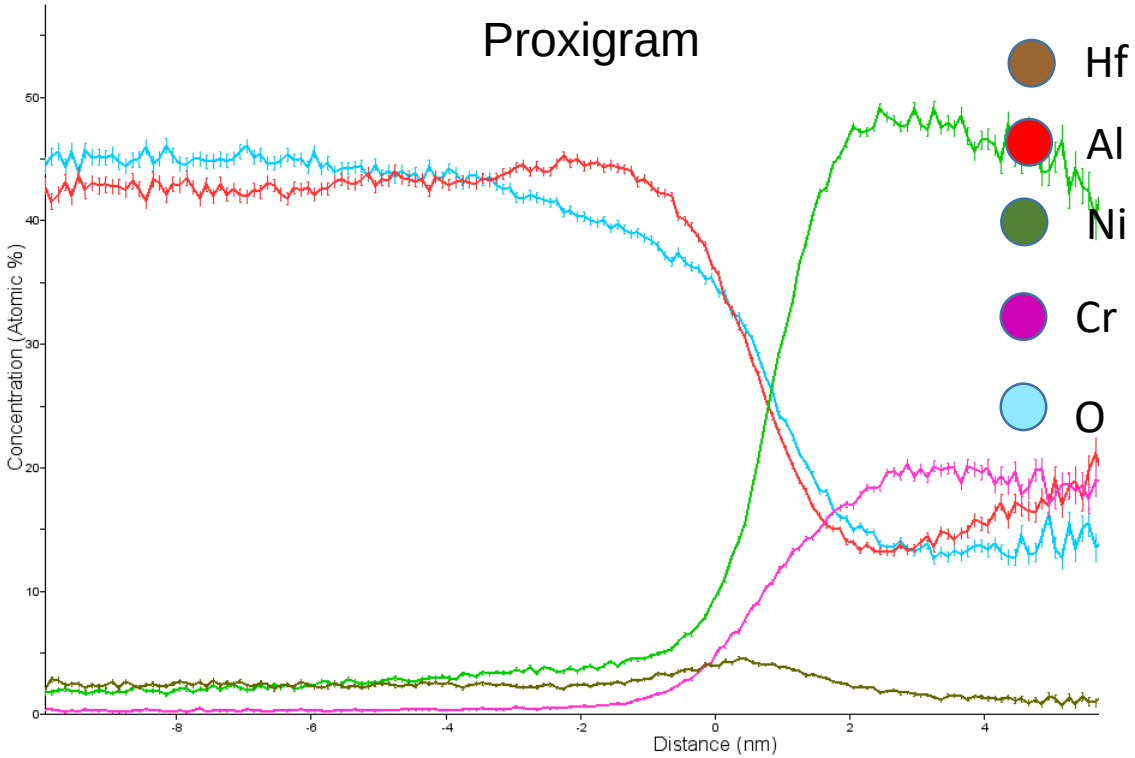


Unerwartete Rauheit der Grenzfläche

M/O Grenzfläche



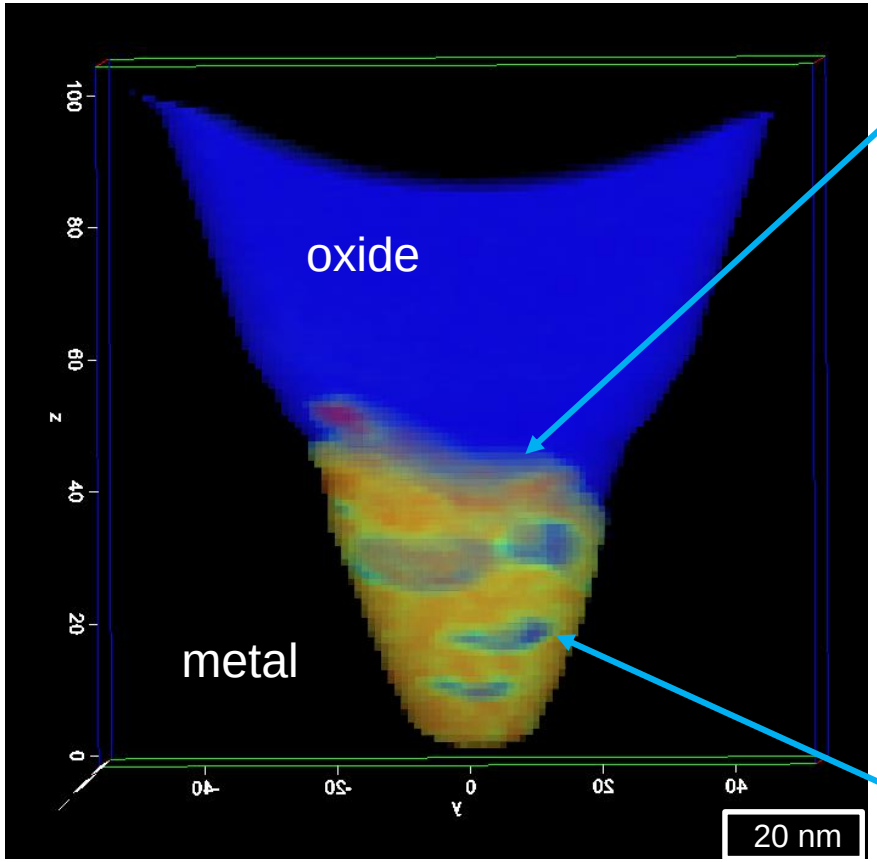
Green: 17 at.% Ni
brown: 2at.%Hf



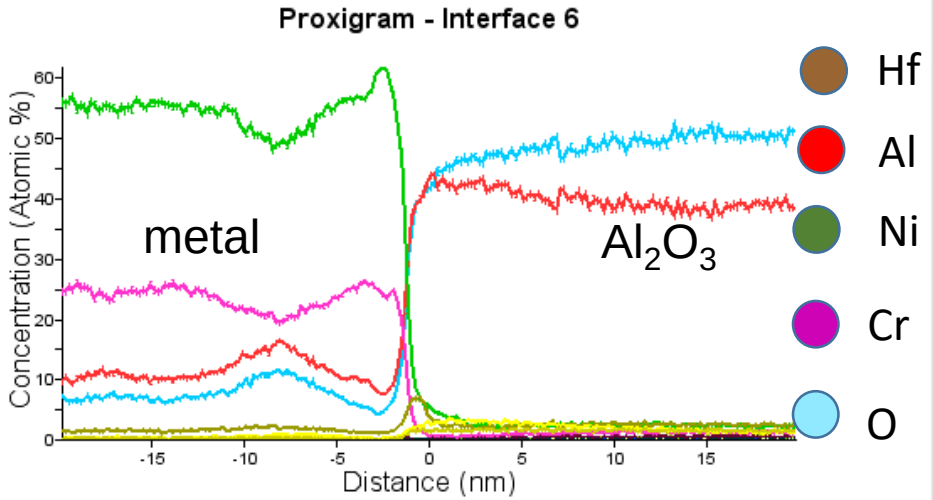
- Al_2O_3-Grenzfläche
- Al-Verarmung, Ni-Anreicherung im Metal nahe der PG
- Hf angrereichert an der PG
- Keine anderen Elemente sind angreichert

APT: YHfB- M/O-Grenzfläche

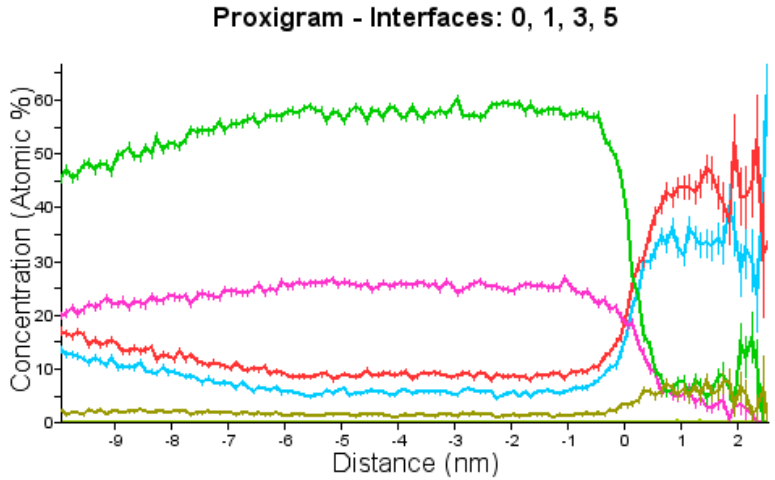
Ni



Isosurface 34 at.% O

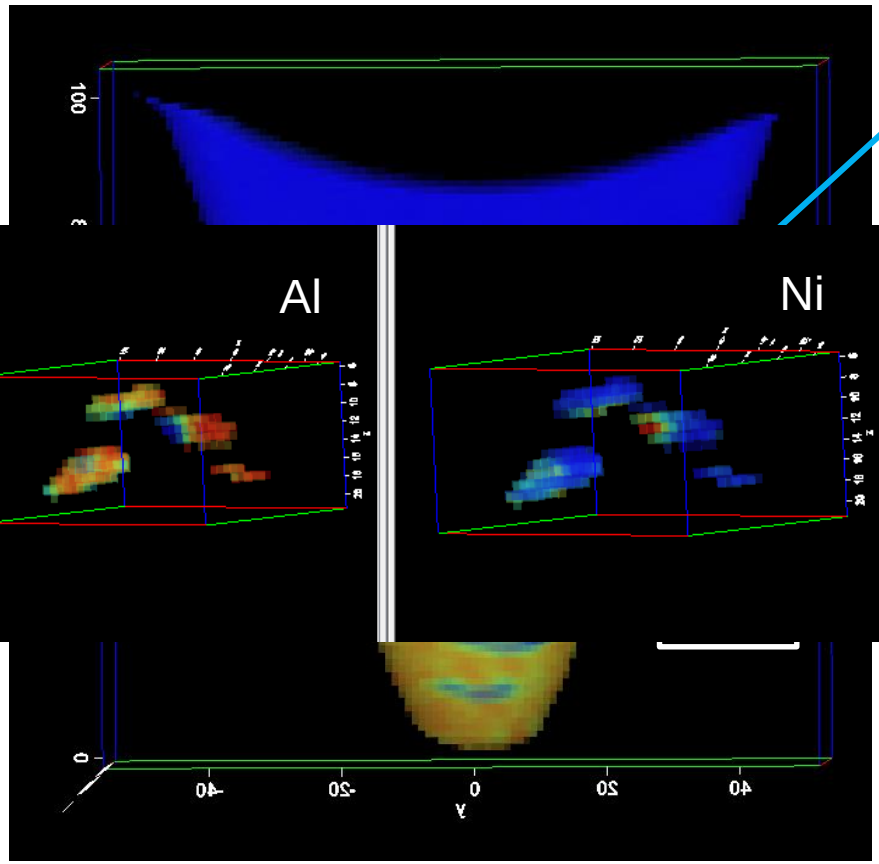


Isosurfaces 13 at.% O

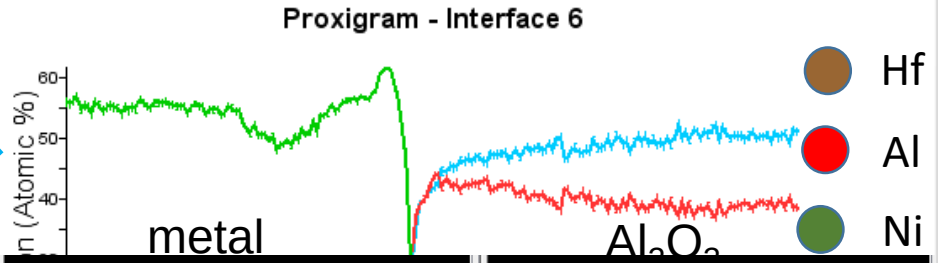


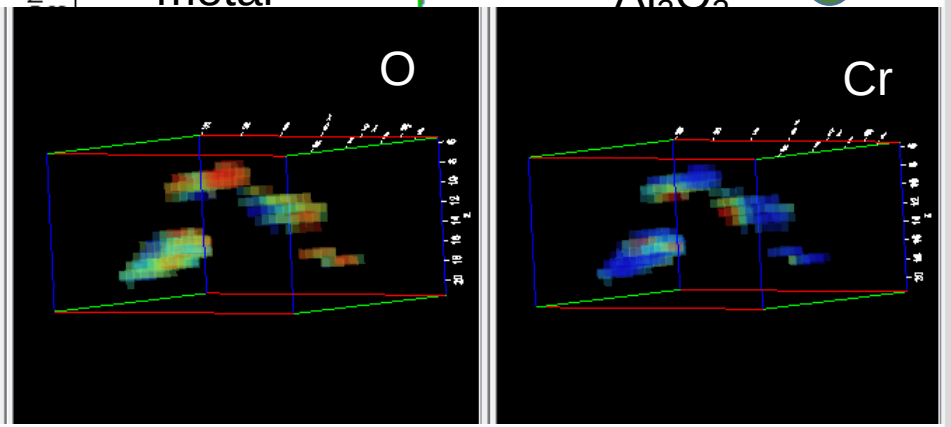
Patikel sind kein reines Al_2O_3, sondern enthalten auch NiO und CrO

APT: YHfB- M/O-Grenzfläche



Isosurface 34 at.% O





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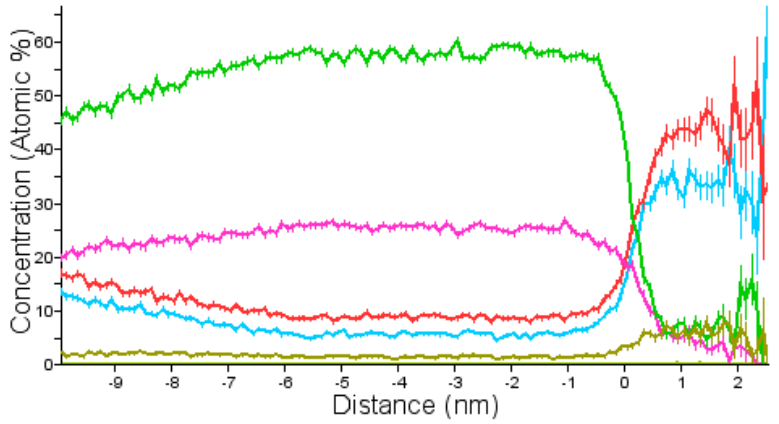

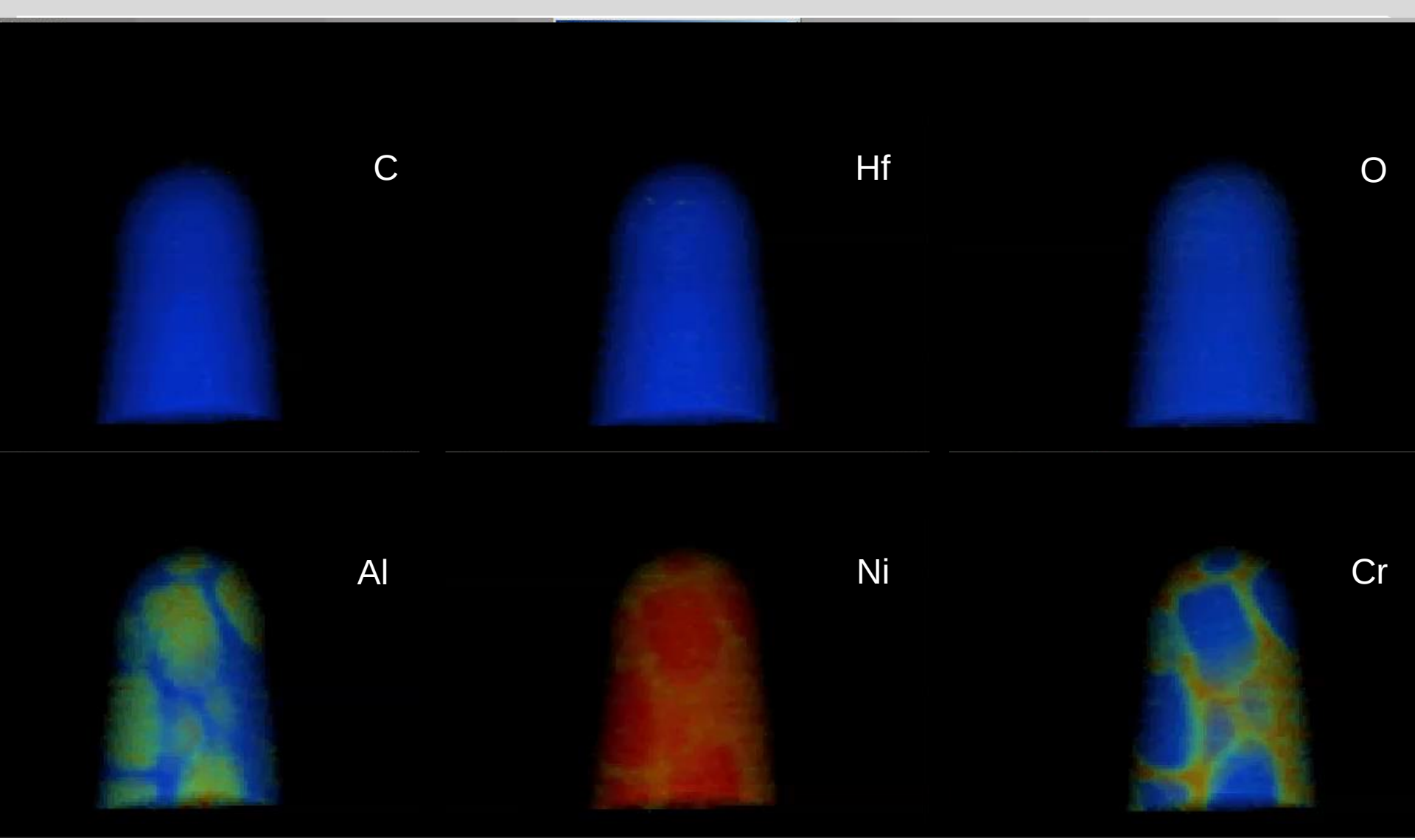

- Oxidation von oben links
- Oxygen diffundiert entlang γ/ γ` PG
- γ` (Ni_3Al): $Ni_{61}Al_{23.5}Cr_{9.3}O_{4.0}$ γ (NiAlCr):$Ni_{52.5}Cr_{32.3}Al_{10.0}O_{3.2}$

Zusammenfassung

Grenzflächen

- Ti & B weder an KG noch PG
- Hf an M/O-PG, O/O-PG und Oxid-KG
- Y an O/O-KG und O/O-PG,a ber nicht an M/O-PG
- Al_2O_3-Al_2O_3 KG enthalten Ni und Cr.
- Hf und Y beeinflussen den Transport von O, Ni and Cr in den KG

Der Oxidationsprozess

- Oxid-Metal Grenzfläche rauh
- Oxidation entlang γ/γ`-PGs in Al-reiche γ'-phase
- Kleine Oxidpartikel im Metall nicht nur Al_2O_3 sondern auch NiO und CrO
- Nur dank APT beobachtet

Vielen Dank für Ihre Aufmerksamkeit

An APT interessiert? - knmf.kit.edu, oder mich ansprechen.
KNMF gewährt sinnvollen Projekten APT-Zeit.