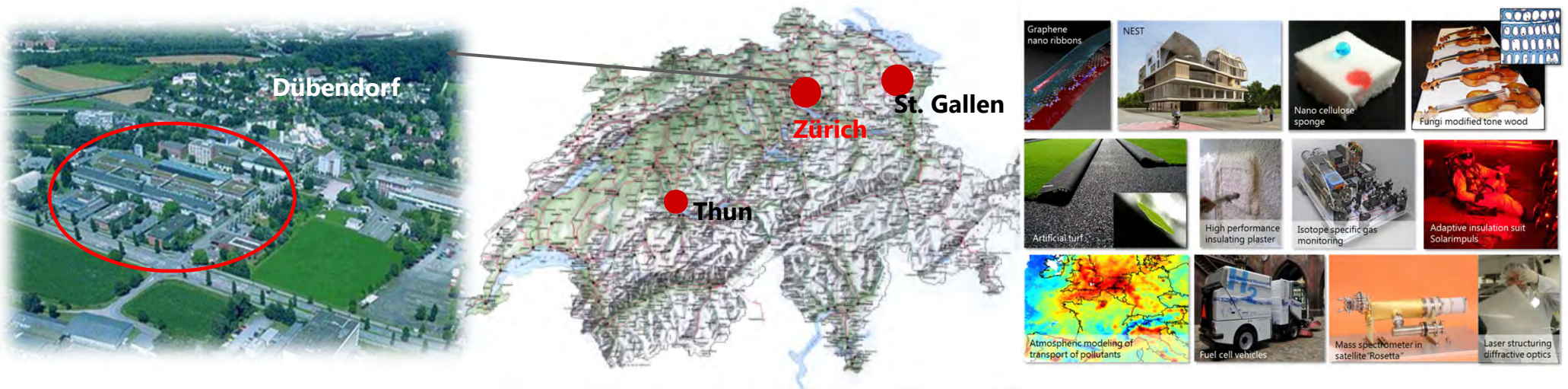


Willkommen
Welcome
Bienvenue



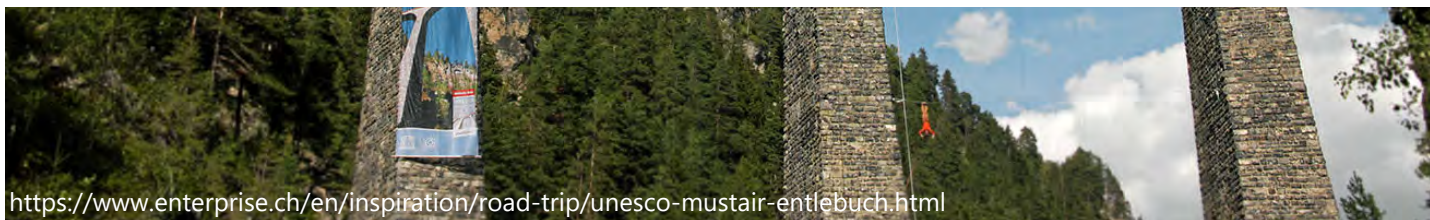
Empa

The Place where Innovation Starts



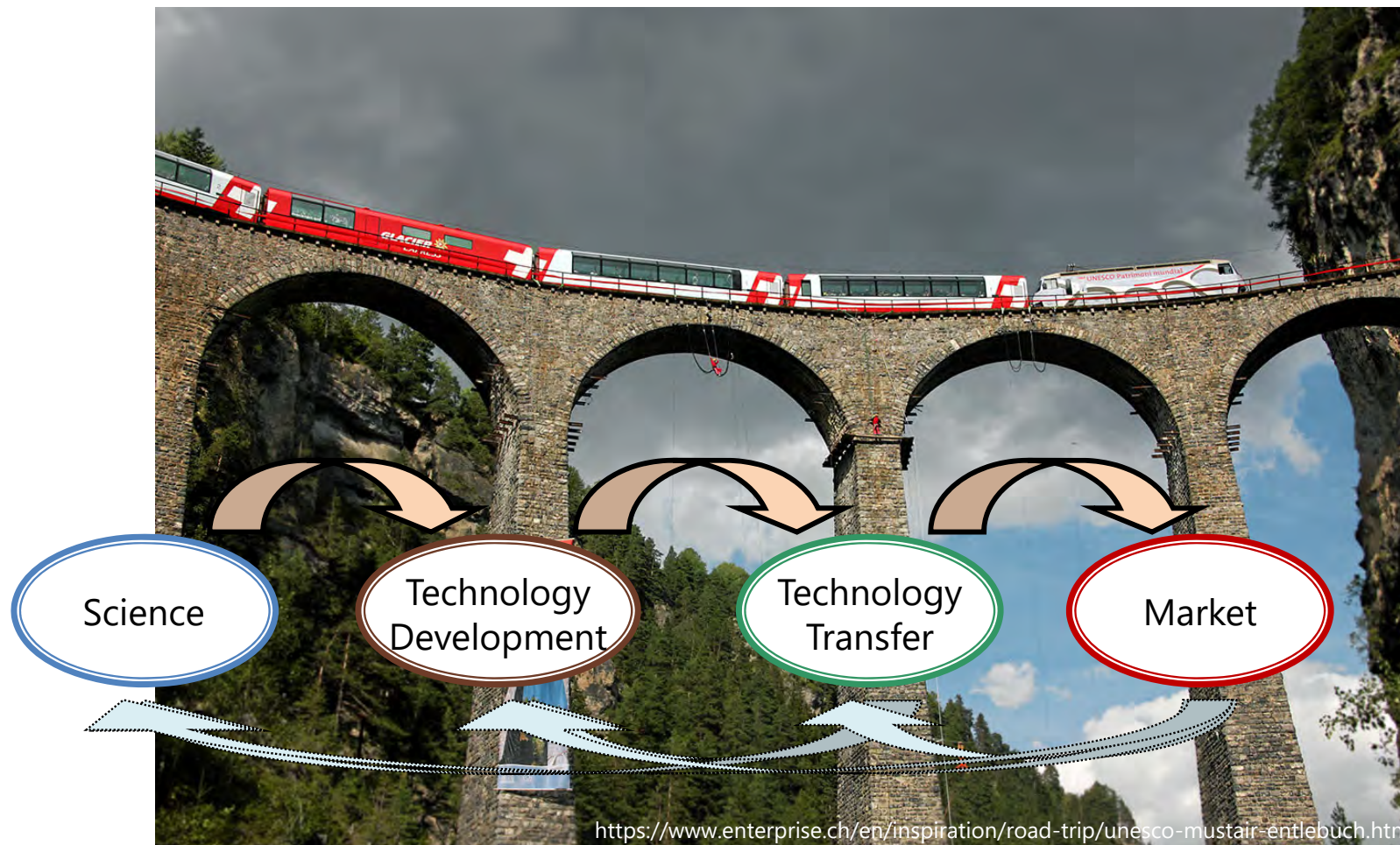


Bridging fundamental science with application, and universities with industry

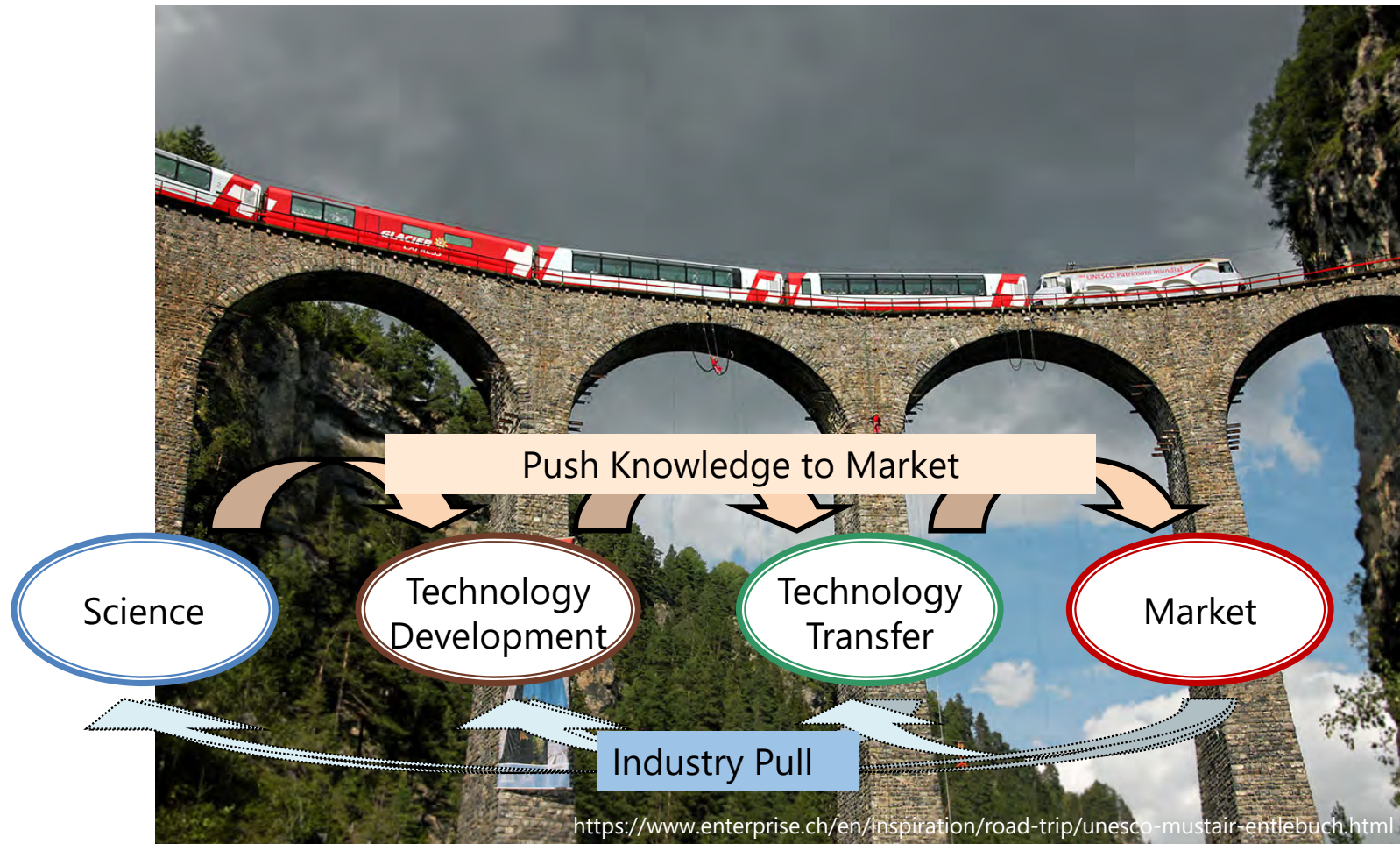


<https://www.enterprise.ch/en/inspiration/road-trip/unesco-mustair-entlebuch.html>

Bridging fundamental science with application, and universities with industry



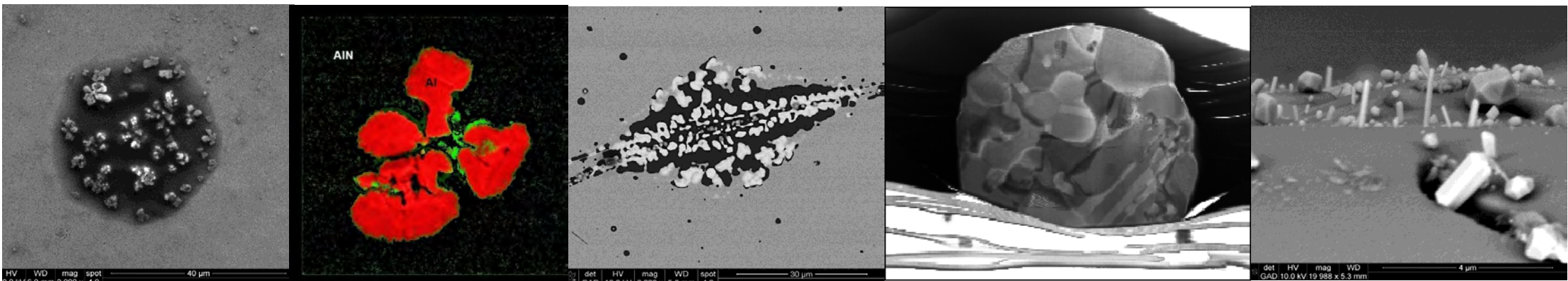
Bridging fundamental science with application, and universities with industry



Exploring nanomultilayers for joining technology applications *and > 15 years of collaboration with WUT*

Jolanta Janczak-Rusch

Empa, Swiss Federal Laboratories for Materials Science and Technology,
Joining Technologies & Corrosion, 8600 Dübendorf, Switzerland

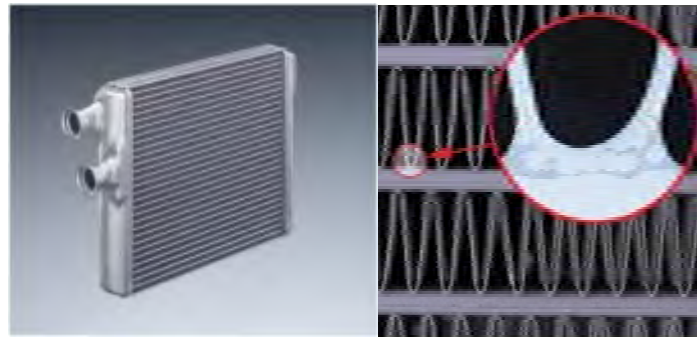


Content:

our journey through the exploration of nanomultilayers

- **Case Study 1:** Development of **Al-Si** brazing filler based nanomultilayers (NMLs)
 - *How to overcome the technological limits by brazing of Al heat exchangers?*
- **Case Study 2: Ag-Cu** brazing filler/**AlN** barrier NMLs
 - *Melting, phase separation, melting behavior, control of the mass transport*
- **Case Study 3: Ag/AlN** nanomultilayers
 - *Anomalous fast diffusion under oxygen atmosphere*
- **Case study 4: Cu/W** nanomultilayers
 - *Brazing and in-situ formation of nanocomposites*

Case study 1: AlSi/AlN nanomultilayers



Courtesy: MAHLE Behr GmbH & Co.,
Stuttgart

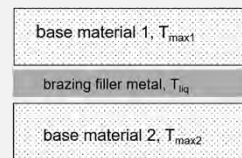
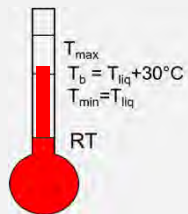
Source: www.innovaltec.com/brazed-aluminium-heat-exchangers/

Brazing of Al heat exchangers *Industrial request (2003-2008)*

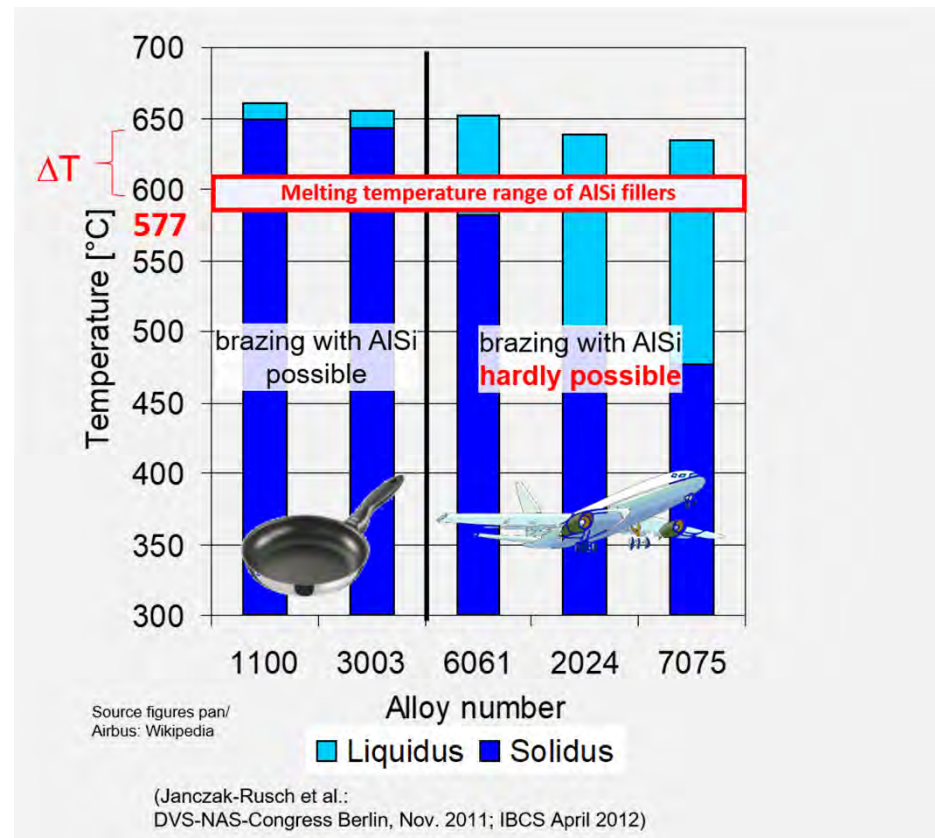
How to overcome the technological limits by brazing Al heat exchangers?

Brazing of **Al-alloys** with **AlSi filler metal alloys** is limited by the relatively high brazing temperature

- AlSi brazing filler alloys ($T_b > 577^\circ\text{C}$) are general purpose fillers



- Small ΔT between core and filler metal \rightarrow challenging process control, heat effects on the base metal
- Limited number of suitable core metals for brazing
 - Brazeability of high strength alloys? Innovation?
 - Solidus temperature of some high strength alloys as low as 485°C
- Need of energy- and cost-saving, environmentally friendly processes



Melting Point Depression (MPD) in nanoparticles and thin films

MPD of nanolayers

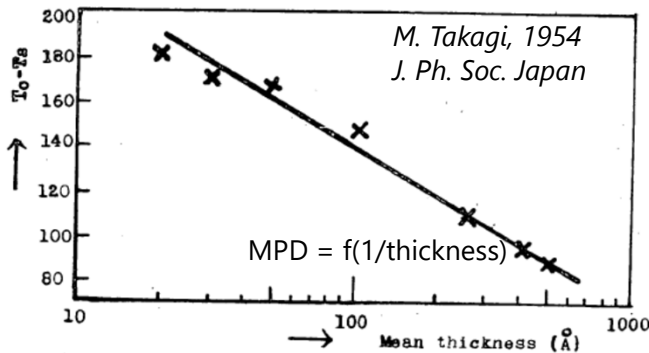
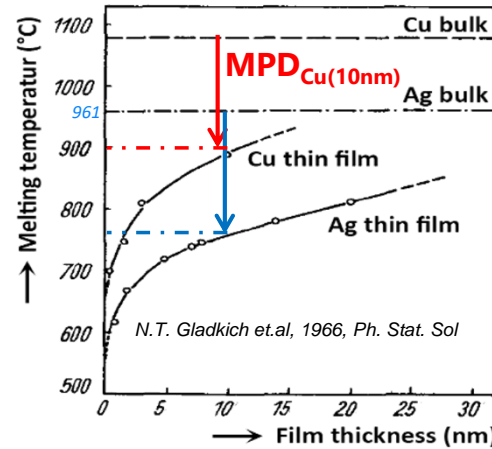
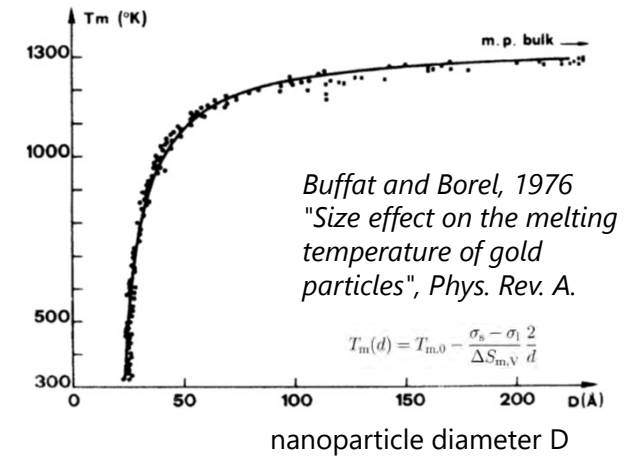


Fig. 3. $(T_0 - T_s)$ for various thickness of Pb films.



$$MPD = T_{m \text{ bulk}} - T_{m \text{ nano}}$$

MPD of nanoparticles



- ✓ It is well-known that the melting point of nanostructures decreases with decreasing size in the nano-scale regime (i.e. <20 nm)
 - Large surface to volume ratio of nanometals alters thermodynamic and thermal properties
- ✓ MPD in order of tens to hundreds of degrees can be achieved for metals with nanodimensions.

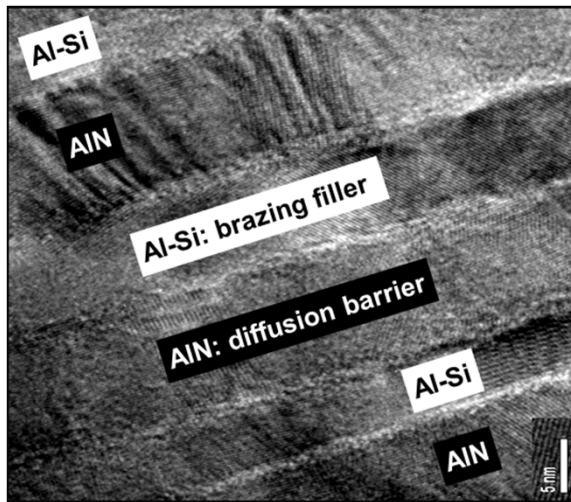
Can we reduce the brazing temperature by using a nanostructured brazing filler metal (alloy) ?

Nanolayered AlSi12 brazing filler (AlSi/AlN NML): methods

NMLs deposition

Magnetron sputtering

- alternated deposition of AlSi and AlN layers
- reactive sputtering of AlN



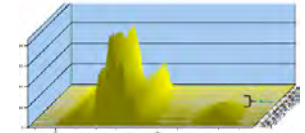
TEM image of cross-section of as-deposited AlSi12/AlN_{10nm} (10x) NML

Behr-/ EMPA- Patent DE102008050433.5

Determination of the melting point

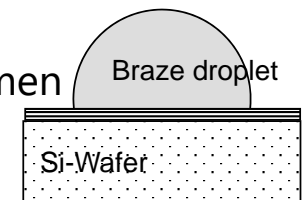
- **HT XRD investigation:** in-situ measurements (PANalytical X'Pert PROMP Diffractometer, HT Chamber), T > 300°C, steps of 20°C using CuK α radiation

Criteria: disappearance of the strongest crystalline peaks of the brazing filler phases (e.g. 111 or 200)



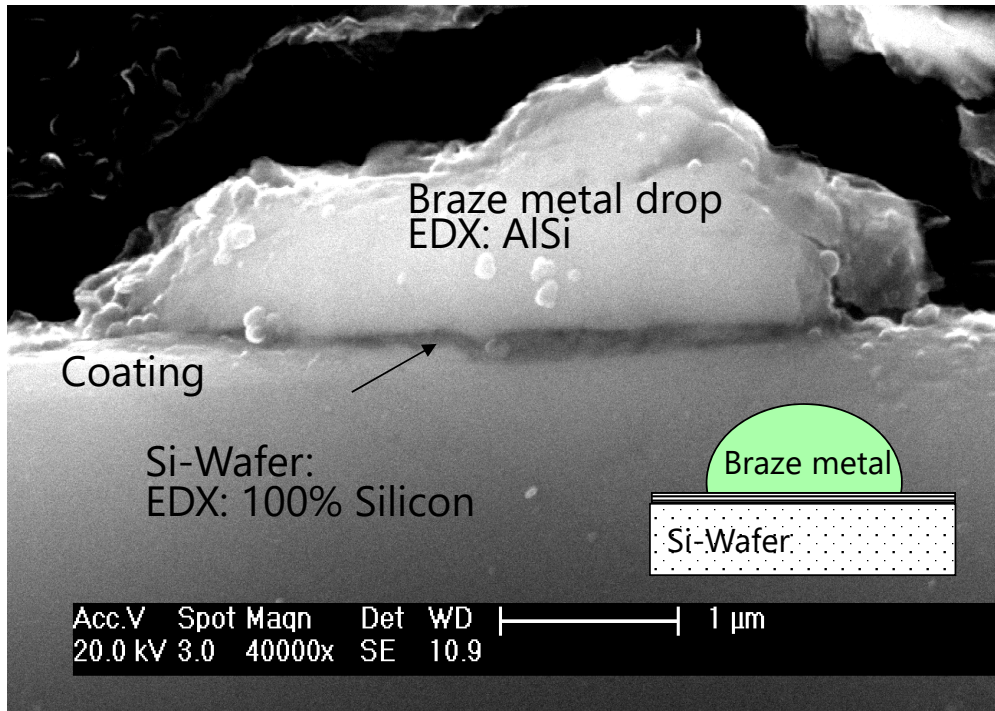
- **Melting experiments:** annealing (T=300 ... 600°C, hold time: 5min, heating rate: 5K/min, Ar 6.0) followed by SEM investigations and thickness measurements

Criteria: appearance of braze droplets at the specimen surface, change in multilayer thickness

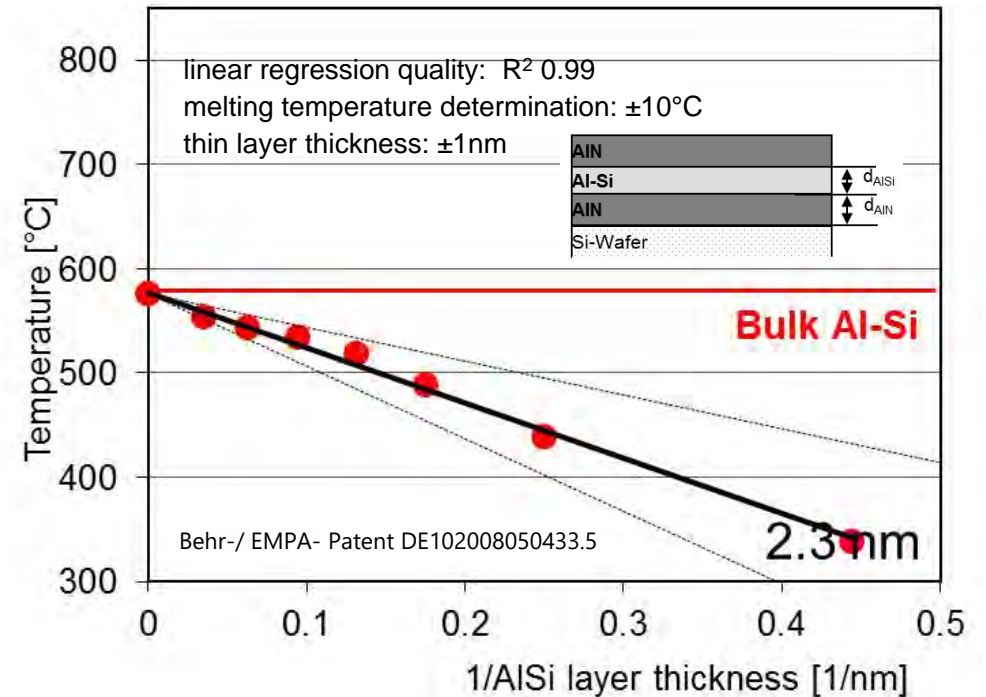


- Correlation of the results of both investigations

Melting of nanolayered AlSi12 brazing filler (AlSi/AlN NML)



Melting temperature of confined AlSi12 films



Ultra-thin Al-Si_{12at.%} films confined between AlN layers exhibit size-dependent MPD and allows brazing at reduced temperatures!

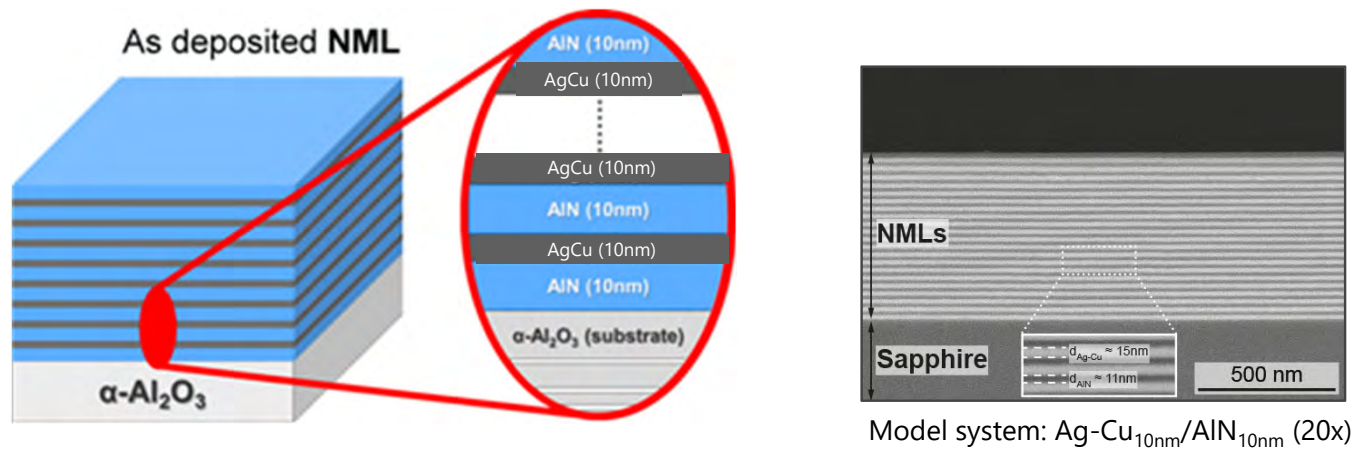
Lessons learned from the case study 1

- NMLs offer great opportunities (not only for joining applications)
- **Al-Si/AlN:** a significant MPD can be achieved for nano Al-Si brazing filler
 - It scales with the filler thickness
- The relationships are very complex: many interplaying parameters (meaning also huge design opportunities)
 - Significant effect of the diffusion barrier material observed

Main practical question related to joining with NMLs:

how to intensify the outflow of the braze material to the NML surface?

Case study 2: Ag-Cu/AlN nanomultilayers



Collaboration with INMAT WUT and IMIM PAN
PhD Thesis Vinzenz Bissig Empa/WUT

Fundamental study on Ag-Cu/AlN nanomultilayers

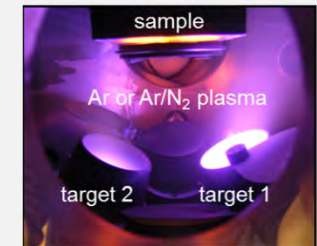
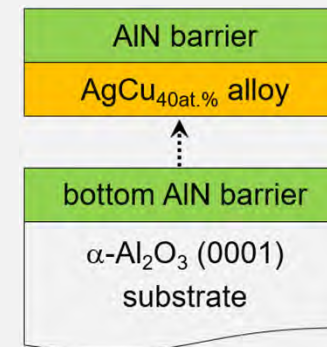
Materials

- Silver-based braze alloy have wide range of brazing applications (relatively low T_b)
- Silver-copper eutectic alloy Ag-28Cu (wt.%) is a “key” brazing alloy (representative of virtually every other braze/solder filler alloy).

Name	Analysis (wt%)	Melting point °C	EN 1044	DIN
Eutectic	Ag-28Cu	778	AG 401	L-Ag 72

- Representative equilibrium phase diagram: a simple eutectic system with partial solid solubility in the terminal phases
- Two-phase structure with silver and copper rich lamella
- The solubility of copper in a silver rich phase and vice versa is negligible at room temperature

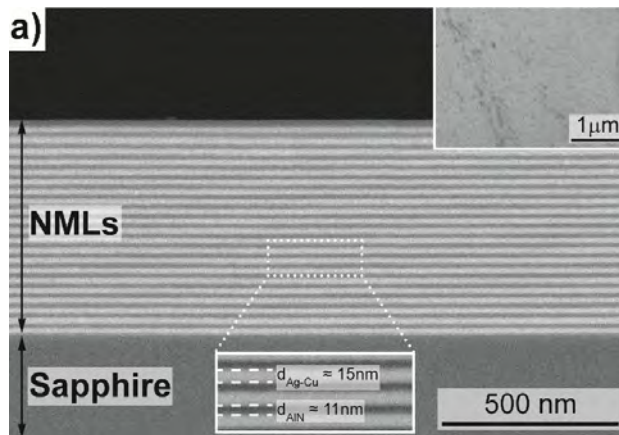
Deposition of Ag-Cu_{40at.%} /AlN NMLs



- Ag-40at.% Cu target, applied power: 25 W
- AlN: reactive sputtering of Al in Ar/N₂(g), 0.3 Pa
- Substrate: α -Al₂O₃(0001)
- Single layer thickness: 4-15 nm
- Number of repetitions: 1, 10, 20

As-deposited Ag-Cu_{40at.%}/AlN NML

Morphology by HRSEM

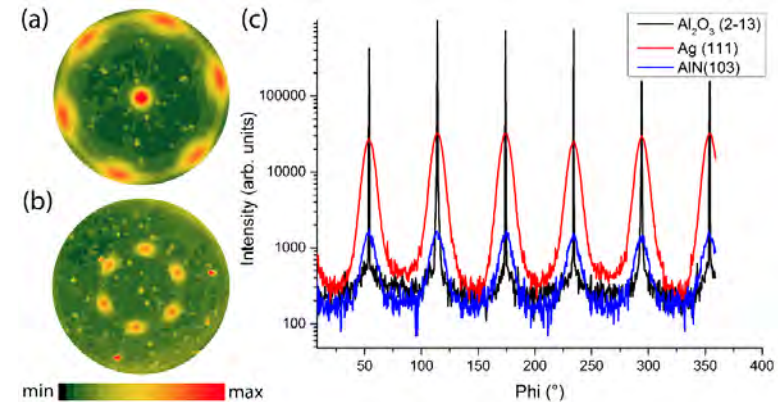


NML cross-section (Ag-Cu: light grey)

SEM micrographs of (a) a cross section and (b) the surface of an as-deposited Ag-Cu_{40at.%}/AlN NML, as produced by magnetron sputtering.

Texture by XRD

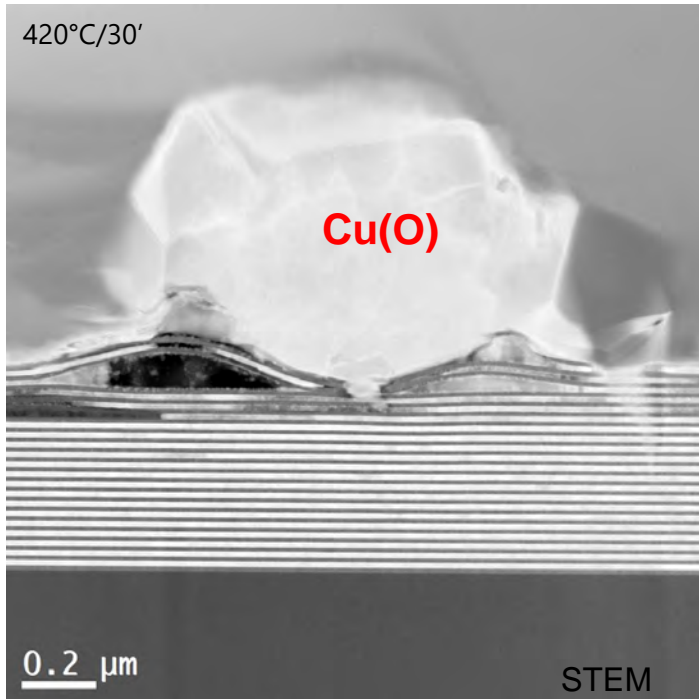
Ref./Source: J. Janczak-Rusch, et.al, Phys. Chem. Chem. Phys., 2015, 17, 28228-28238



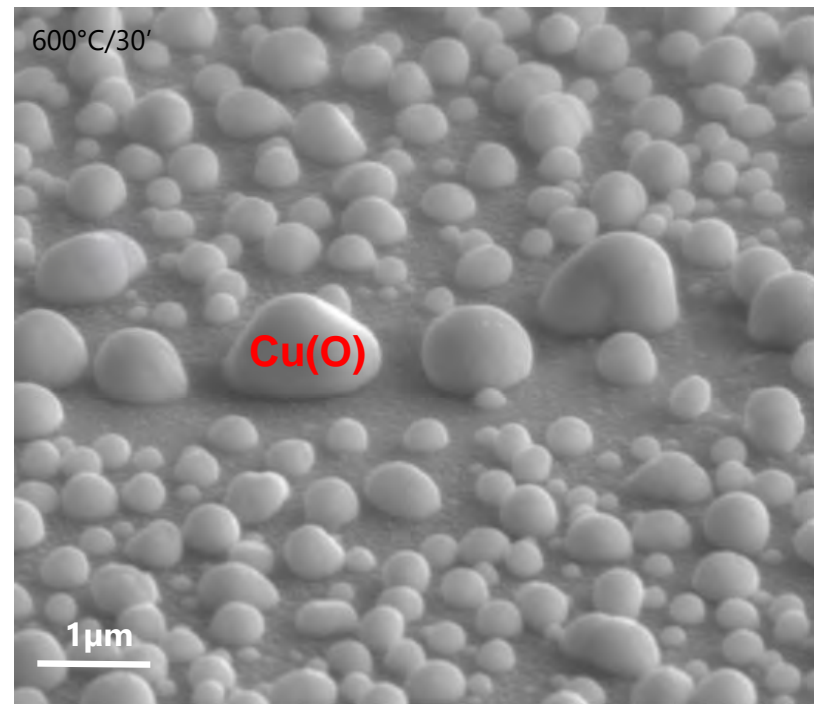
XRD pole figures for (a) the Ag(111) and (b) the AlN(0103) family of planes, as recorded from the AgCu_{8nm}|AlN_{10nm} NML in the as-deposited state. (c) Phi-scans of the α -Al₂O₃(2-1-13), Ag(111) and AlN(10-13) reflections.

- Alternating nanocrystalline Ag-Cu_{40at.%} and AlN layers are of uniform thickness
- Ag-Cu_{40at.%} layers consist of fcc matrix of Ag nano-grains supersaturated with Cu
- Strong in-plane and out-of-plane texture, due to the orientation relationship $\text{Ag}\{111\}\langle 110\rangle \parallel \text{AlN}\{0001\}\langle 1010\rangle \parallel \text{Al}_2\text{O}_3\{0001\}\langle 1010\rangle$.

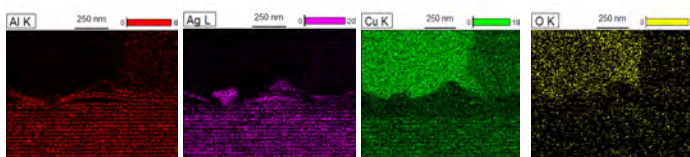
AgCu/AlN after isothermal annealing (420° and 600°C/30')



NML cross section (TEM image)



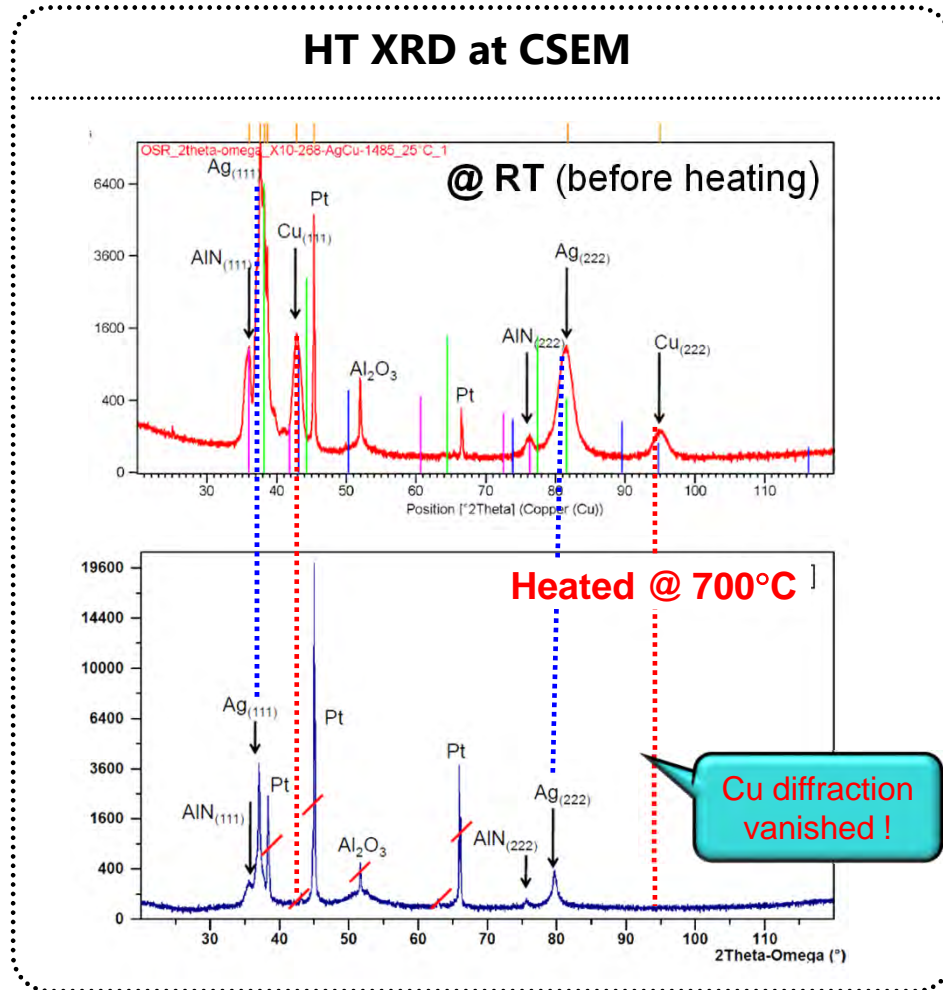
NML top surface (AlN as a top layer)



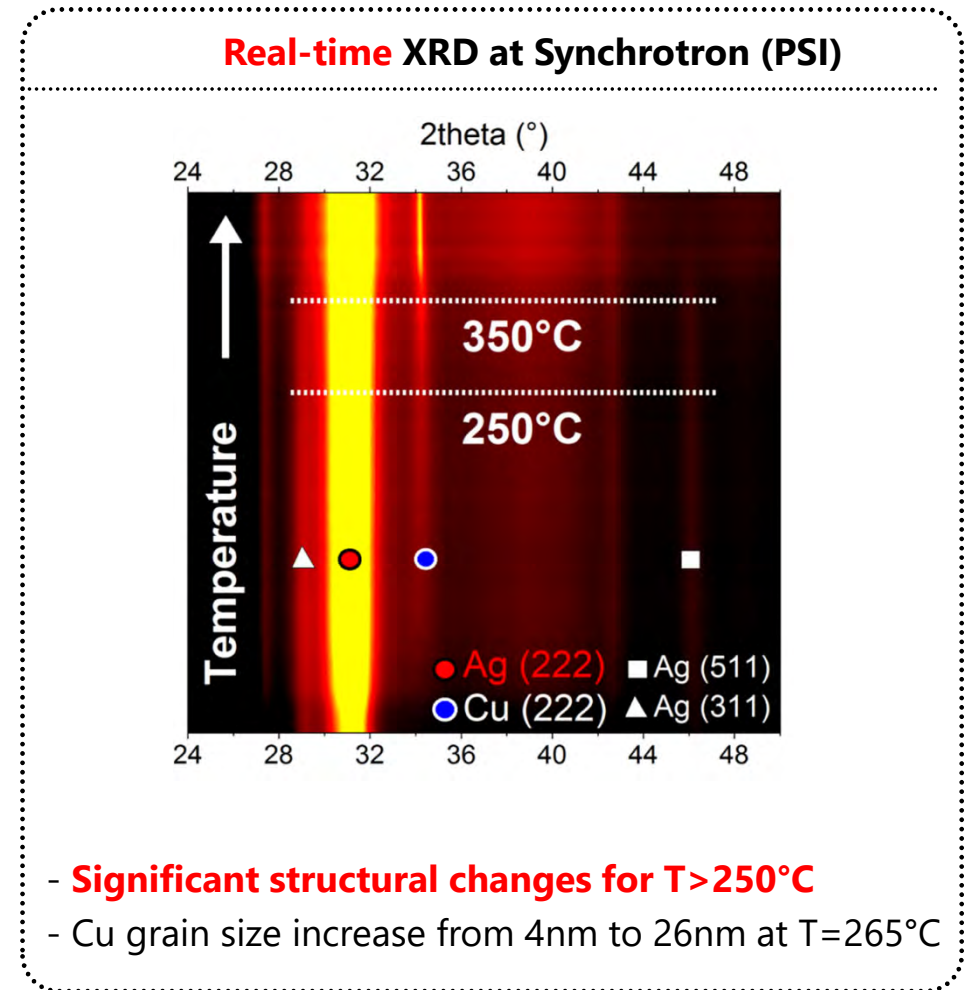
TEM investigations: WUT, group of Prof. M.Lewandowska

Massive "outflow" of Cu (only)

Melting point depression of confined Ag-Cu by HT-XRD:



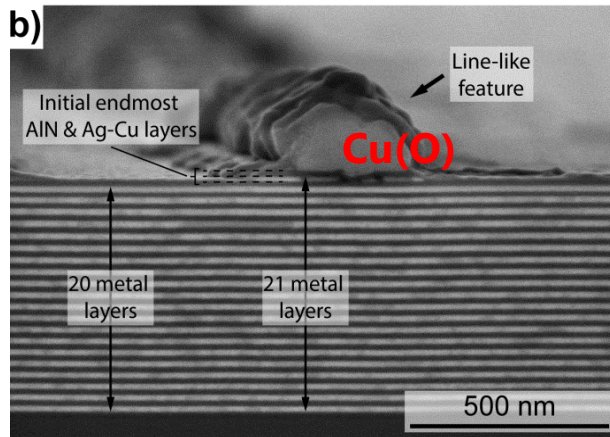
J. Janczak-Rusch/Exploring nanomultilayers for joining technology applications/22



Ref./Source: J. Janczak-Rusch, et.al, Phys. Chem. Chem. Phys., 2015, 17, 28228-28238

Melting behavior of nanoconfined Ag-Cu/AlN

SEM after heating (420°C)



Cu surface protrusions

EDX point measurements			
As-deposited NML			
	Ag (at.%)	Cu (at.%)	O (at.%)
Bare NML	19.0±0.3	12.4±0.5	1.4±0.8
NML after fast heating to 420 °C			
Bare NML	20.3±0.3	10.8±0.4	0.8±0.7
Protrusion	0.7±0.1	48.7±1.1	45.4±3.7

Ref./Source: J. Janczak-Rusch, et.al, Phys. Chem. Chem. Phys., 2015, 17, 28228-28238

J. Janczak-Rusch/Exploring nanomultilayers for joining technology applications/23

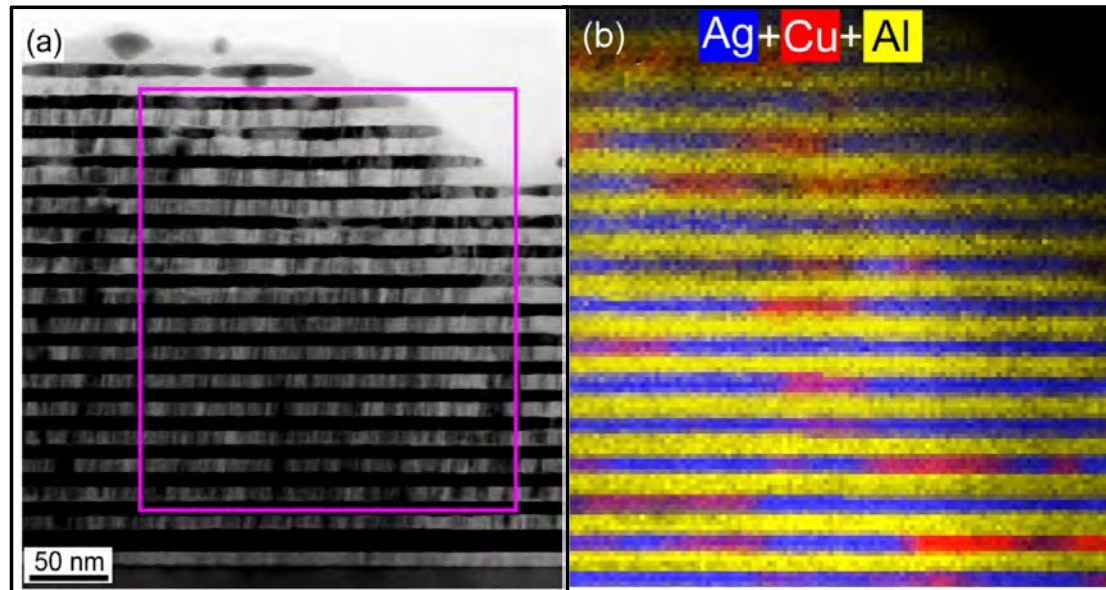
Findings (XRD, SEM, TEM, EDX)

Non-eutectic melting behavior of confined Ag-Cu alloy !

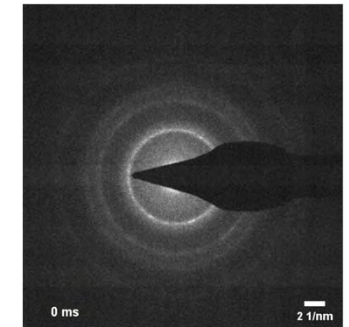
Melting Temperature of	Bulk materials	in NML (layer thickness: 10nm)
Ag-Cu (eutectic)	778°C	heterogeneous melting (420-810°C)
Cu	1084°C	420-560°C
Ag	961 °C	> 700°C

**What is the reason for the observed behavior ?
Why the phase with a higher melting point (Cu) melts first?**

Phase separation in Ag-Cu_{40at.%}/AlN NML



TEM (bright field)/EDS mapping of Ag-Cu_{8nm}/AlN_{10nm} NML after prolonged annealing at $T > 250^{\circ}\text{C}$

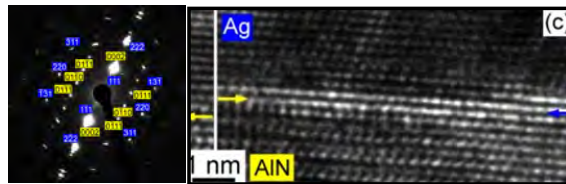


HT TEM AlN_{10nm}/AgCu_{16nm}/AlN_{4nm}, $T=350^{\circ}\text{C}$, 0.4s

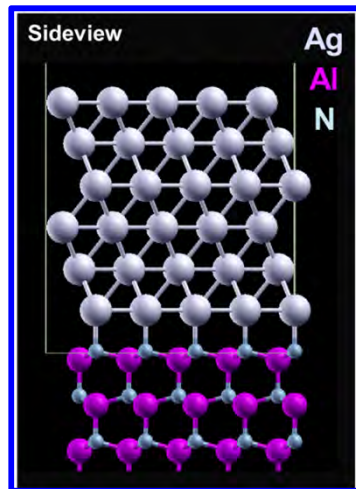
- Phase-separation with coarsening of Ag and Cu domains driven by large enthalpy of mixing and reduction of grain boundary energies
- Onset of phase separation becomes thermally activated at around 250°C , but does not induce noticeable degradation of the NML structure
- It is a very fast process (~ 0.4 s); observed also for confined Al-Si alloys

Interface's structure in Ag-Cu/AlN (DFT and HR TEM)

Ag/AlN interfaces

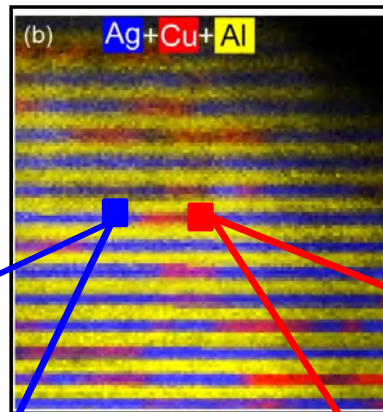


semi-Coherent (stable)



5x5x3-Ag unit cell on 5x5x3-AlN(0001)

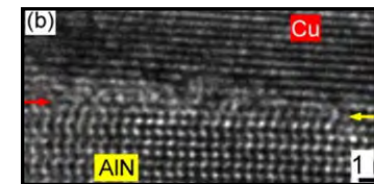
Phase-separated Ag-Cu



Refs: *Phys Chem Chem Phys* 17 (2015) 28228,
Appl. Phys. Lett. 101 (2012) 181602

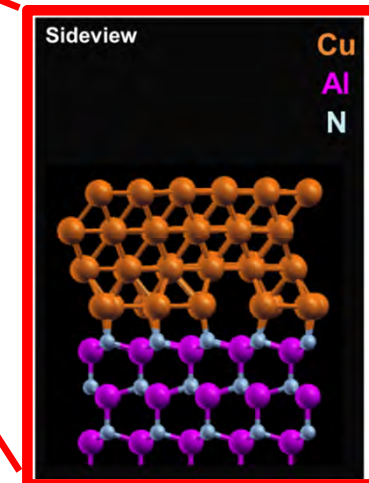
Lower enthalpy of defect formation and higher mobility along **Cu/AlN** interface, (preferential Cu migration)

Cu/AlN interfaces



HR-TEM

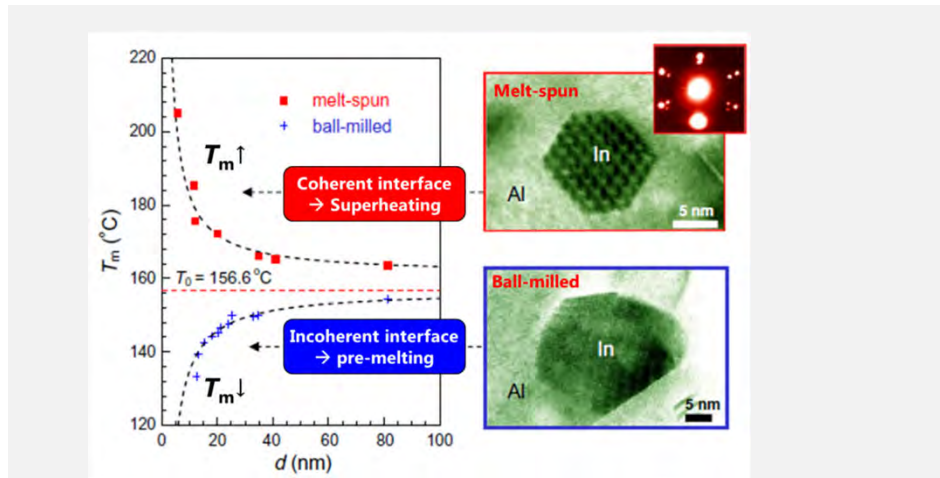
Incoherent (unstable)
with strong in-plane disorder



5x5x1/6x6x3-Cu unit cell on 5x5x3-AlN(0001)

The effect of the interface structure on solid-liquid transition

In particles embedded in a Al matrix (experimental study)

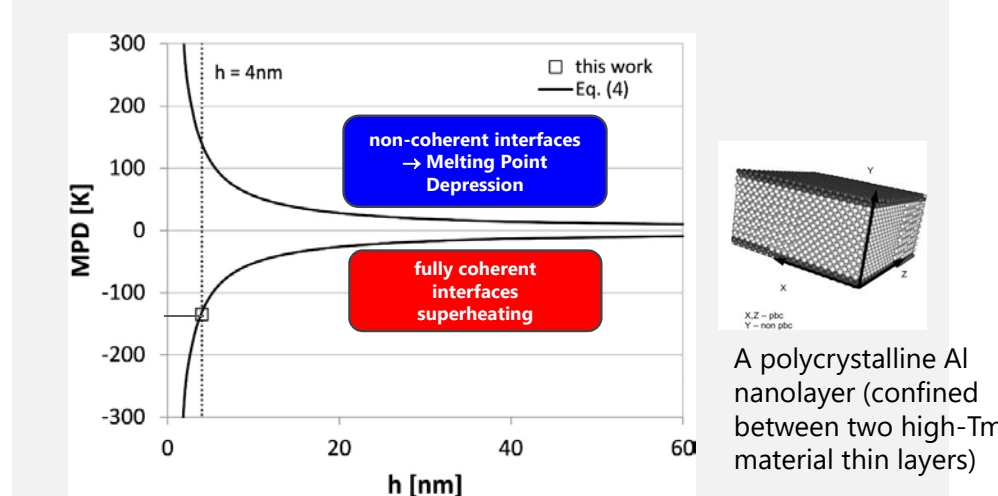


Effect of interface structure on melting of nano-confined metals

Ref: Lu & Jin, *Curr. Opin. Solid State Mater. Sci.* 5 (2001) 39

Nanoparticles coated by or embedded in a high melting point matrix with incoherent interface, exhibit a melting point higher than that of the bulk counterpart (superheating)

Confined Al thin films (molecular dynamics simulations)



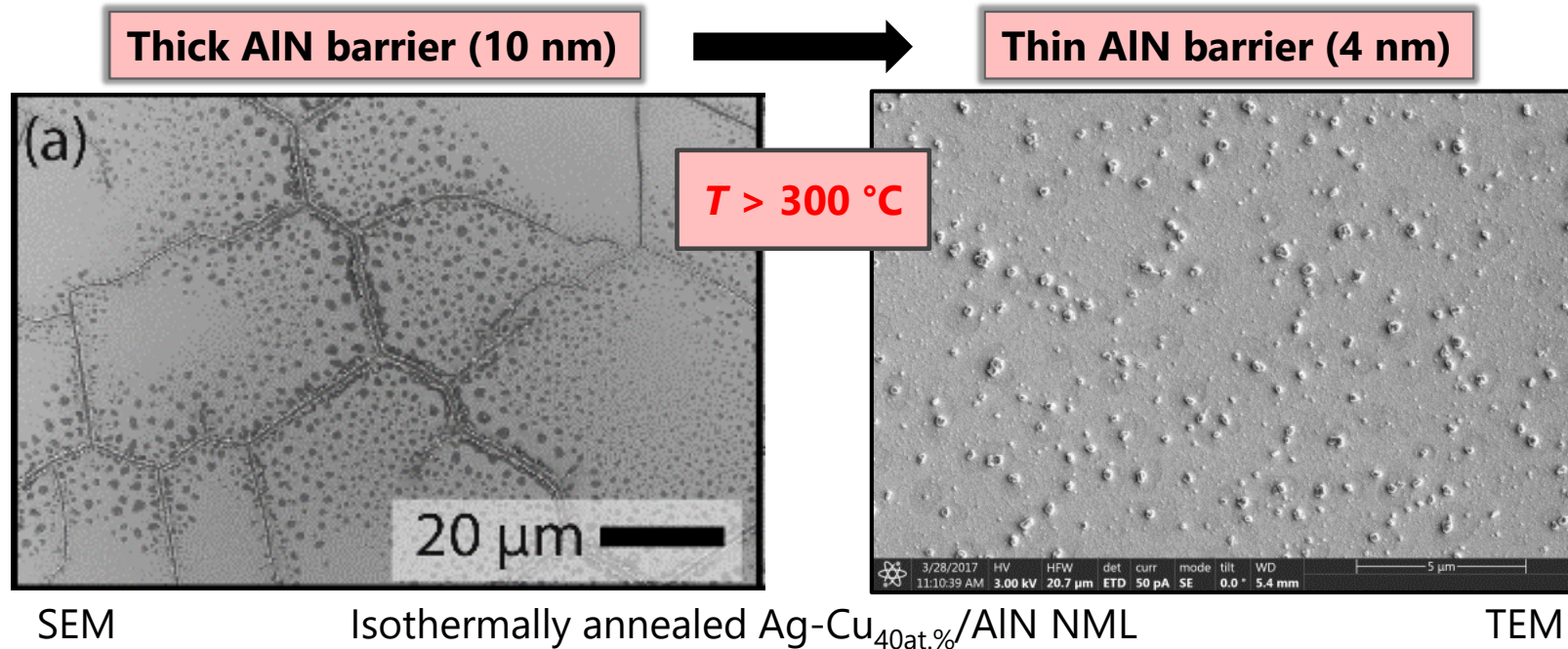
Effect of interface structure on melting point of confined thin aluminium films

Ref: T. Wejrzanowski, M. Lewandowska, K. Sikorski, K. J. Kurzydowski, 2014.

doi:10.1063/1.4899240.

The coherent intercrystalline interface suppress the transition of solid aluminum into liquid, while free-surface gives MPD. Al thin film of 4nm thickness confined between coherent interlayers shows a MP of 131 K by MD higher than defect free infinite crystal

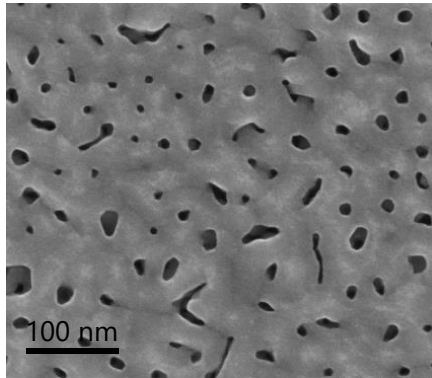
The effect of the barrier layer thickness



- Fast, patterned outflow of Cu
- Void formation
- Fracturing of AlN barrier layers

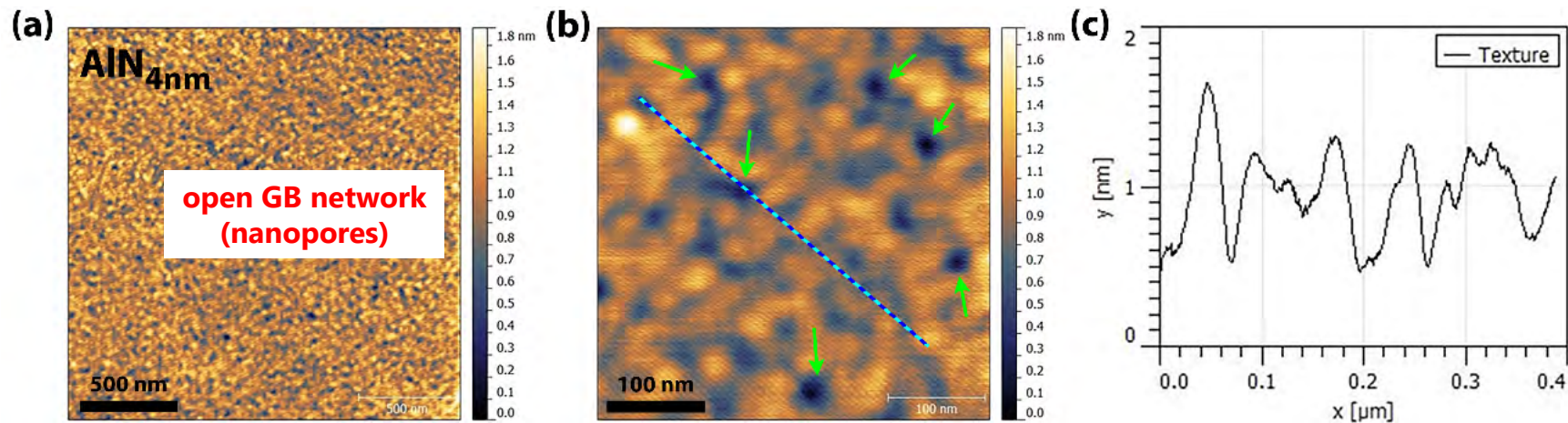
- Fast, homogenous outflow of Cu and Ag
- No void formation
- Sintering of AlN barrier layers

Directional mass transport in NMLs with ultra-thin barriers

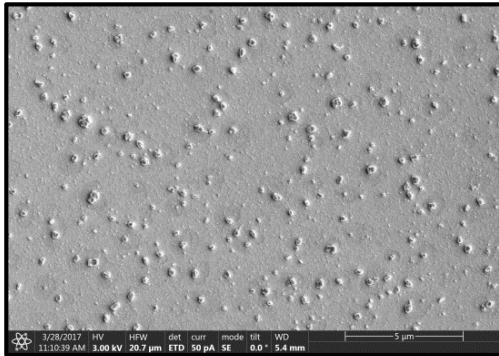


Surface of thin AlN barrier (4 nm)

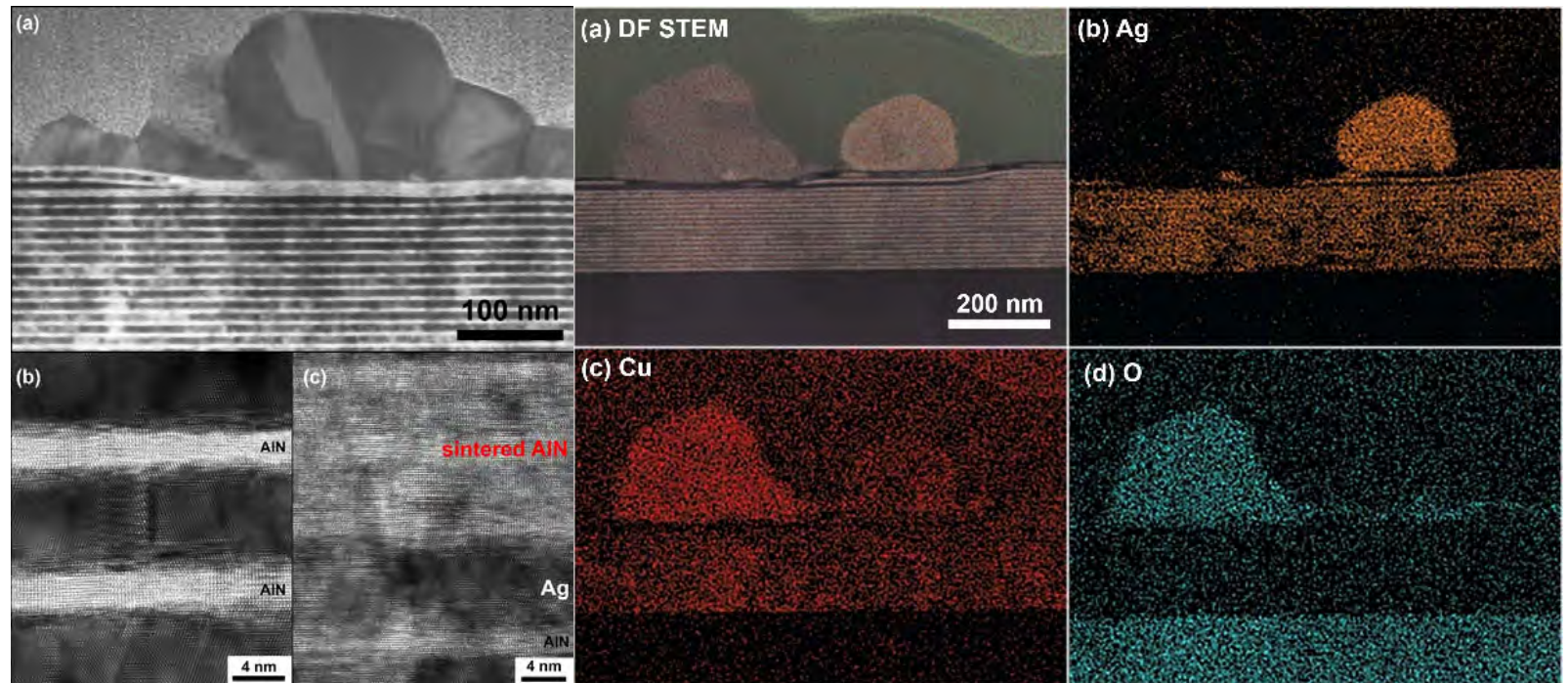
Ultra thin AlN nanolayers deposited by magnetron sputtering show **open grain boundary structure**. The open nano-channels promote the diffusion of confined metals (Cu and Ag) from the entire NML towards the surface.



Metal outflow in Ag-Cu_{40at.%}/AlN NML with a thin barrier (4nm)



The NML surface is densely and homogeneously covered with submicron and nanometer sized Ag and Cu droplets ($T > 300\text{ }^{\circ}\text{C}$)

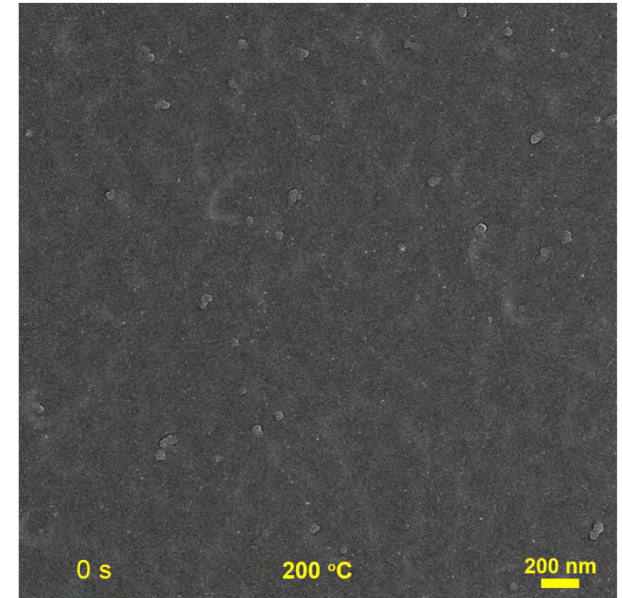


STEM micrographs and EDS elemental maps for the AgCu_{8nm}/AlN_{4nm} NML cross-sections after 5 mins of isothermal annealing at 500 °C

Confined voids, as created by mass transport of Cu and Ag, have been closed by the deformation and partially sintering of the remaining AlN barrier layers!

Lessons learned from the case study 2

- Nano-confined alloys of bulk eutectic composition can show non-eutectic melting (heterogeneous process).
 - Sputter deposited Ag-Cu/AlN NMLs undergo **phase separation** of Ag and Cu (a very fast process) nano/grains, as driven by large positive enthalpy of mixing.
- Melting point depression strongly depends on the structure of local interfaces and energetics.
 - The Cu nano-grains form incoherent AlN interface with AlN, resulting in random in-plane texture and high melting point depression of $\sim 500^\circ\text{C}$.
- The outflow of the confined material can be tuned

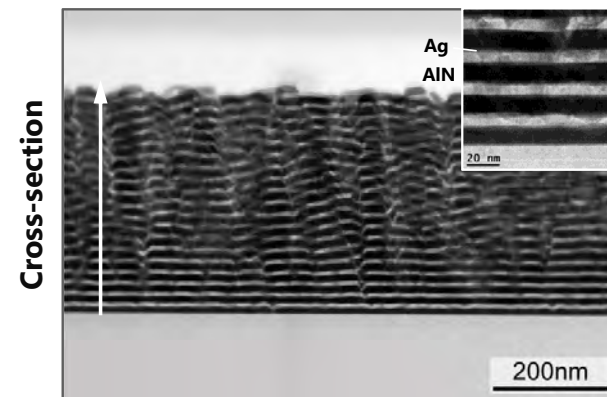
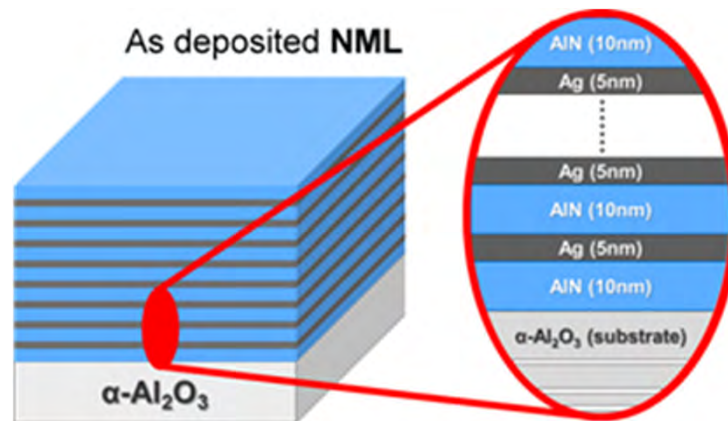


Phase transformations in AlN/Ag-Cu_{18nm}/AlN sandwich 200-400°C (in-situ TEM observations)

Ref: V. Araullo-Peters et. al. ACS Appl. Mater. Interfaces 116, 6605-6614

Does a nanoconfined “eutectic” alloy offer any advantages in terms of MPD?

Case study 3: $\text{Ag}_{5\text{nm}}/\text{AlN}_{10\text{nm}}$ (20x) nanomultilayer

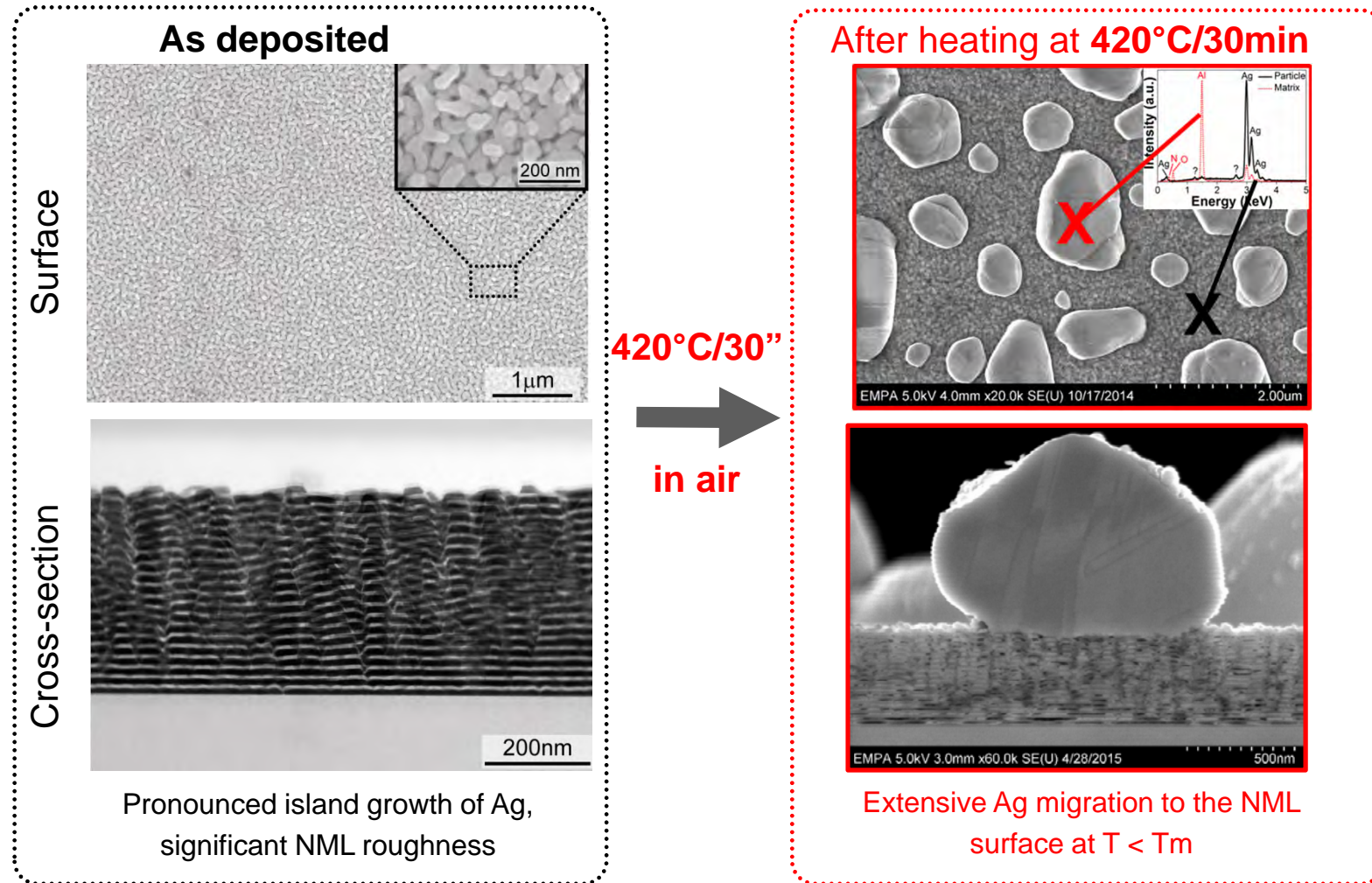


Model system: $\text{Ag}_{5\text{nm}}/\text{AlN}_{10\text{nm}}$ (20x)

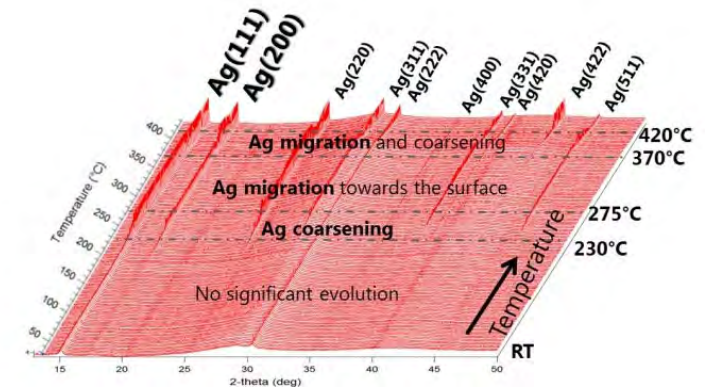
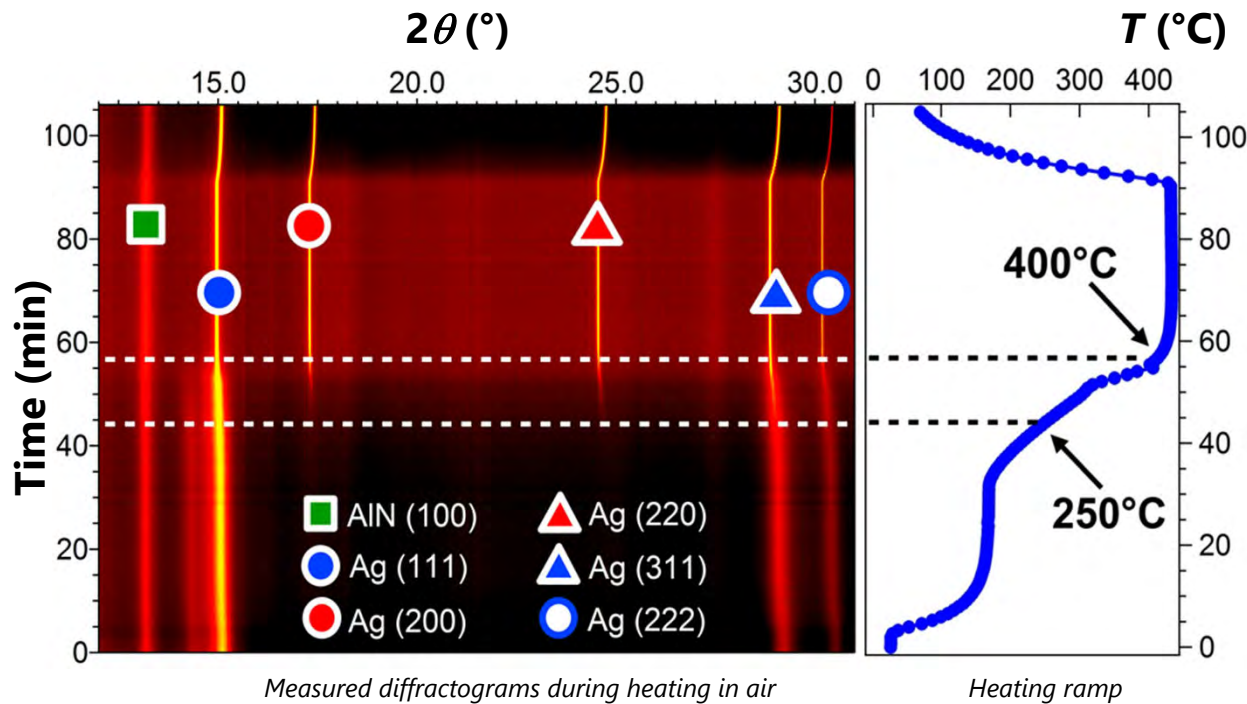
Collaboration with WUT

Ref: J Mater Chem C 4 (2016), 4927

The behavior of Ag/AlN nanomultilayers upon heating



In-situ tracing of the outflow of nanoconfined Ag during heating (XRD at the synchrotron)



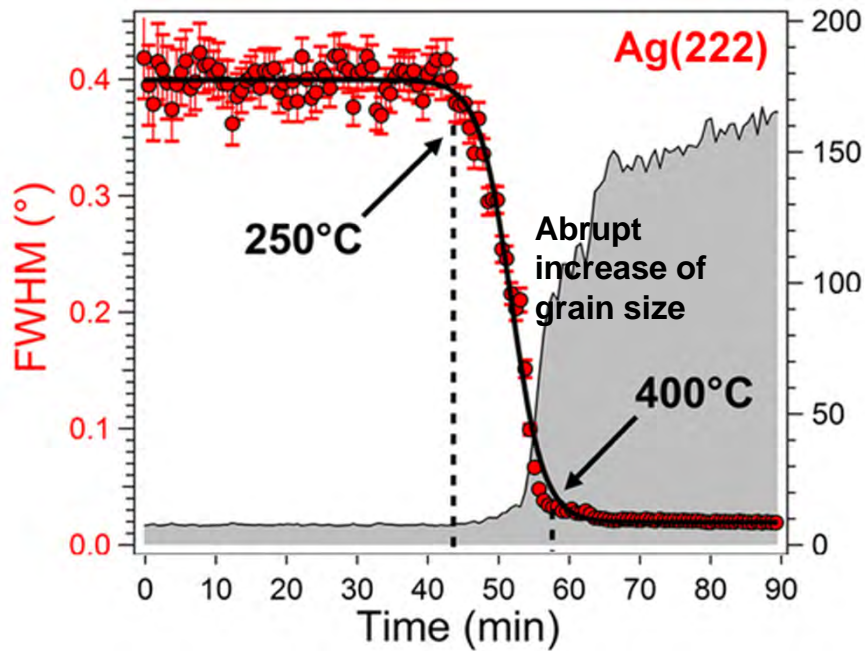
M. Chiodi et. al. Massive Ag migration through metal/ceramic nano-multilayers. *J.,Mat. Chem.C*, 4[22]2016(4927-4938).

- Initially very broad peaks become narrower upon heating
- Appearance of Ag (200) & Ag (220) reflections (enhanced polycrystallinity!) at $T > 250^\circ\text{C}$

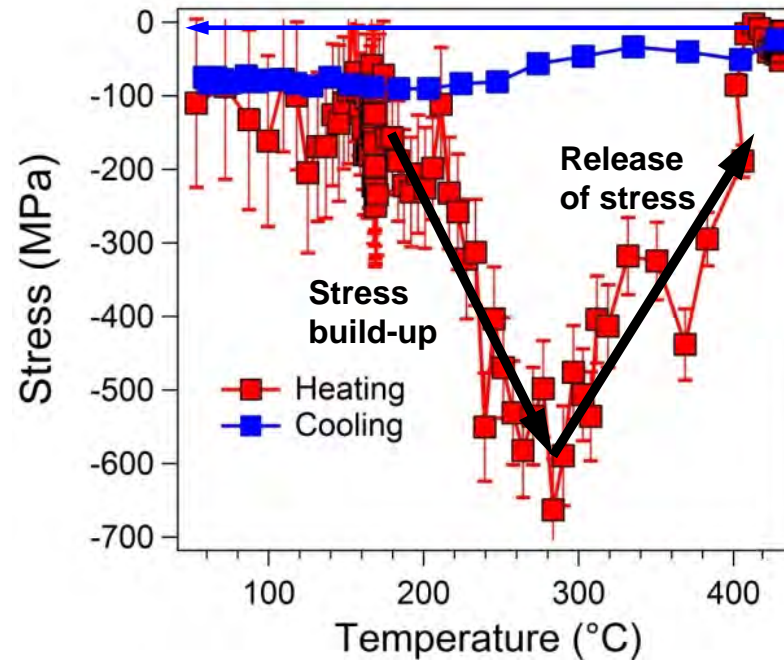
In-situ XRD at the synchrotron during heating of Ag/AlN NMLs in air evidences very fast outflow of Ag to the surface at temperatures as low as 250°C !

In-situ tracing of Ag outflow during heating

Ag grain-size evolution



Average stress evolution



Ag_{5nm}/AlN_{10nm}

Uniform Stress Deformation Model applied to Ag(111) and Ag(222)

$$\beta_{hkl} \cos \theta_{hkl} = \frac{K\lambda}{D} + \frac{4\sigma \sin \theta_{hkl}}{Y_{hkl}}$$

Sherrer formula Stress-related broadening

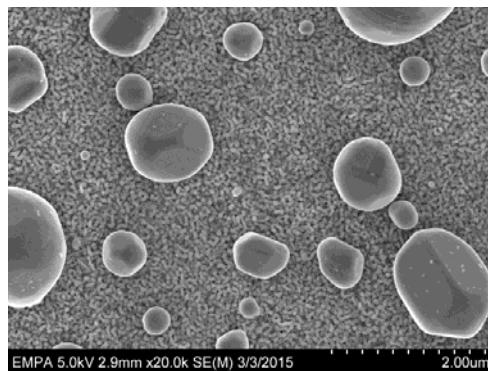
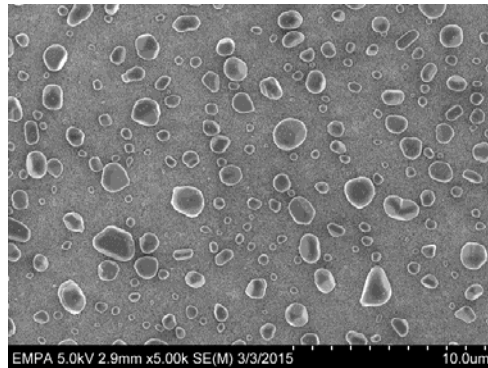
β is the measured **FWHM**
 θ is the diffraction **angle**

M. Chiodi et. al. Massive Ag migration through metal/ceramic nano-multilayers. *J.,Mat. Chem.C*, 4[22]2016(4927-4938).

Ag migration is related to the stress-release in the NML

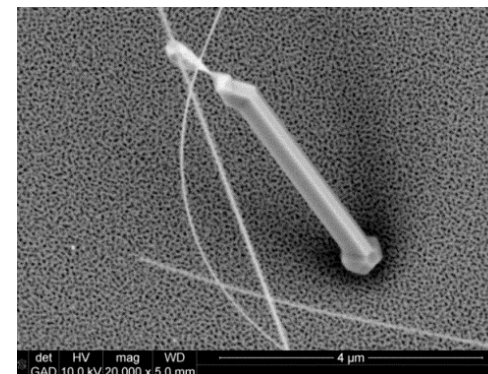
The effect of atmosphere on the Ag outflow and thermal stability of Ag/AlN NMLs

Heating in Air



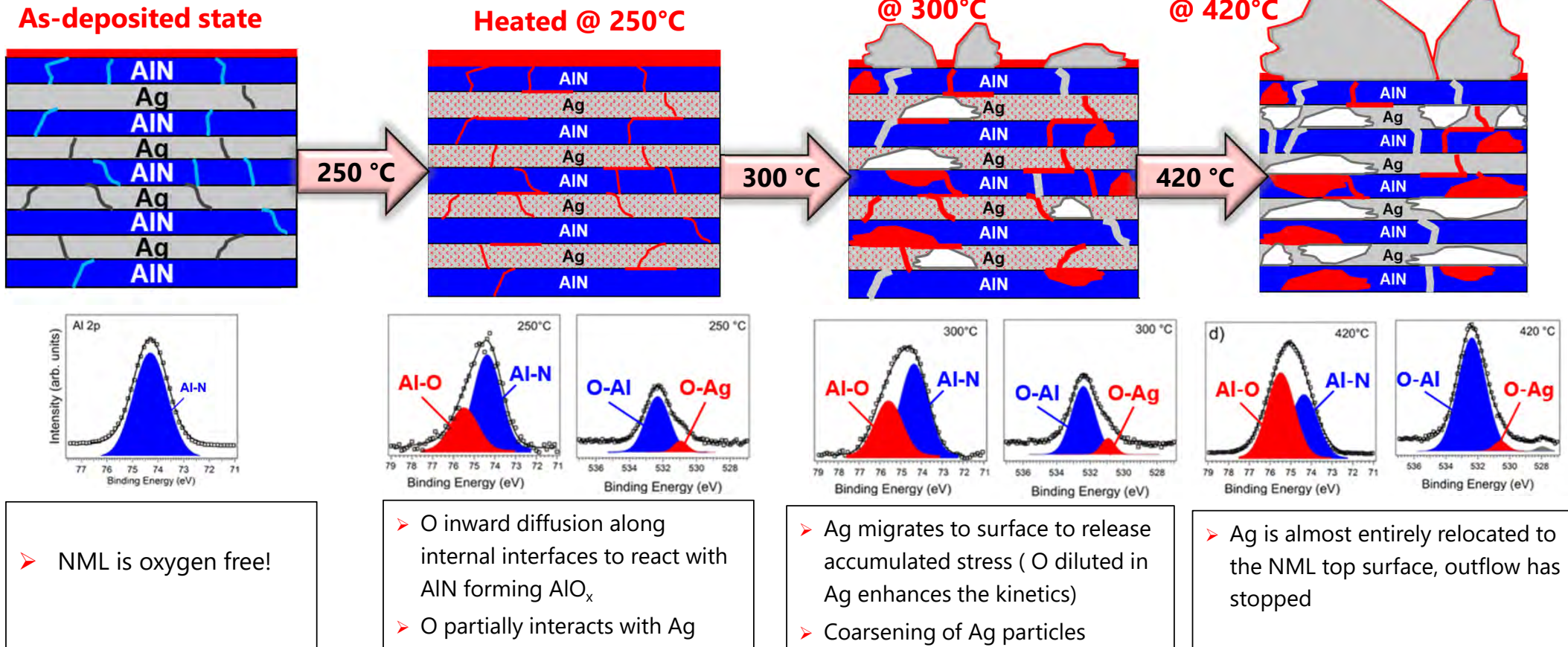
High number density of surface particles (no whiskers)

Heating in vacuum (or single layers)



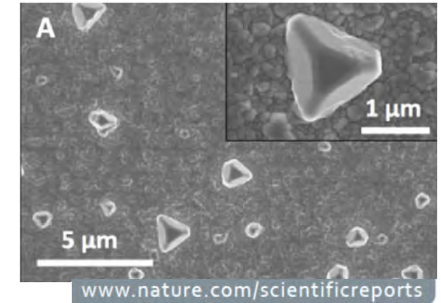
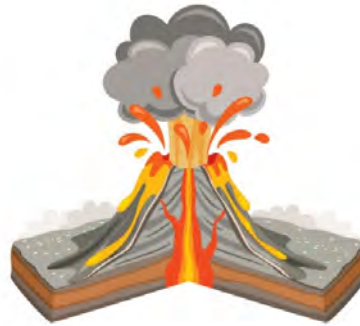
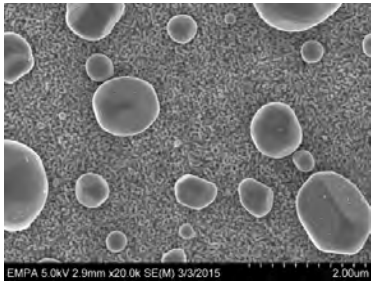
Whisker formation by slow solid state diffusion

Effect of O₂(g) on thermal stability of Ag/AlN by ex-situ XPS



Interaction of O with Ag enhances enthalpy of Ag vacancy formation and thereby the Ag atomic mobility!

A new phenomena of nano-volcanic eruption of Ag originating in Ag-O interactions



April 2016, Empa, WUT:
massive, oxygen enhanced Ag migration in sputtered Ag/AlN NMLs (here: Ag_{5nm}/AlN_{10nm} after annealing at 420°C)

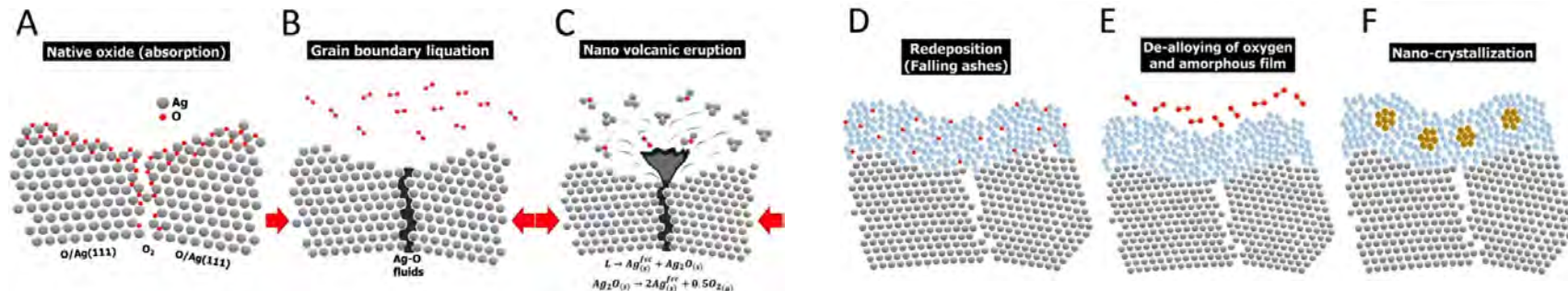
Chiodi, et. al., *Massive Ag migration through metal/ceramic nano-multilayers: an interplay between temperature, stress-relaxation and oxygen-enhanced mass transport*, J. Mater. Chem. C, 2016

https://www.freepik.com/premium-vector/volcano-eruption-with-lava_9433373.htm

Ag nano-volcanic eruption: abundant Ag hillock formation by several interactions between Ag and O, which can be exactly analogized to a volcanic eruption and the deposition of ash.

Oct. 2016, Osaka University and Cheng Kung University: abundant Ag hillock formation on sputtered Ag films at elevated temperatures (here: 1μm thick Ag thin films sputtered on Ti-coated Si wafers and annealed at 250°C/ 5 min)

Sk. Lin, S. Nagao, E. Yokoi, et al., *Nano-Volcanic Eruption of Silver*, Scientific Reports (6)2016



The phenomenon involves grain boundary liquation, the ejection of transient Ag-O fluids through grain boundaries, and the decomposition of Ag-O fluids into O₂ gas and suspended Ag and Ag₂O clusters.

www.nature.com/scientificreports

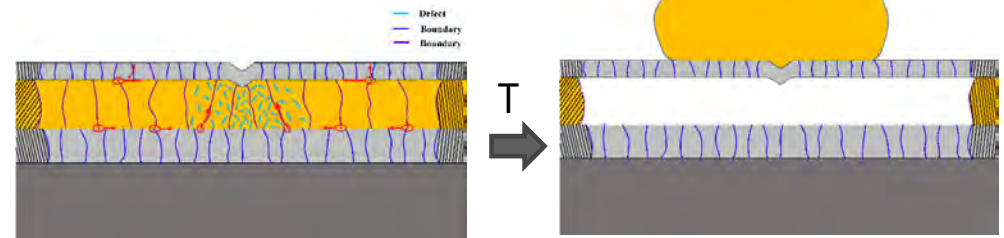
Lessons learned from the case study 3 (Ag/AlN)

- Fast and **extensive migration** of nano-confined Ag at temperatures $T > 250^{\circ}\text{C}$ (well below bulk melting point of 962°C) can be evoked by **oxygen**.
- Migration of the nanoconfined metal is related to the stress-release in the NML

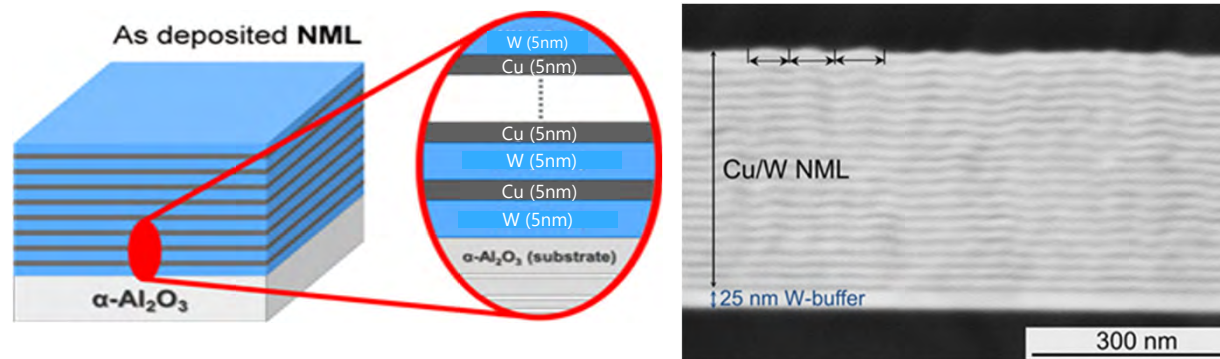
Application-oriented findings:

- New opportunities for the development of joining processes
 - Fast, low-temperature bonding processes with nano-confined brazing fillers are possible
 - no explicit need of melting of the brazing filler or using brazing filler alloys (higher MPD can be practically easier achieved for confined metals than for alloys)
 - use of air as brazing atmosphere can be a choice (low-cost processes)

Can we control the outflow of the confined metal for a localized bonding?



Case study 4: Cu/W nanomultilayer



How to make high-strength joints of refractory metals at $T < 750^\circ\text{C}$?

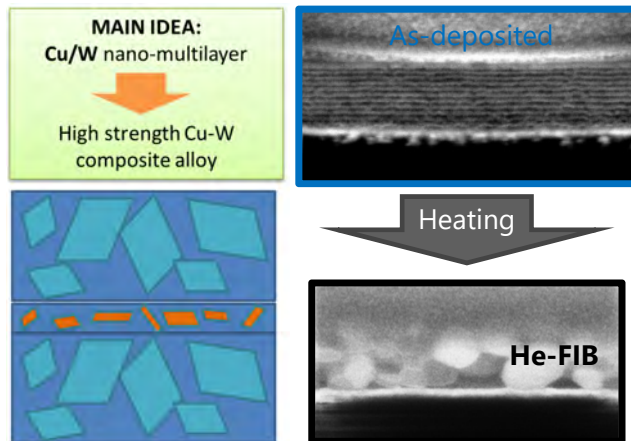
Industrial request (2014-2016) $\text{W}_{25\text{nm}} + (\text{Cu}_{5\text{nm}}/\text{W}_{5\text{nm}}) \times 100$

Refs.: F. Moczner, et. al. , J. of Mat. Sci. Eng. B 6 (2016) 226-230
 C. Cancellieri, et. al. J. Appl. Phys. 2016, 120, 195107
 F. Moczner, et. al. Acta Materialia 2016, 107, 345-353

Cu/W nanomultilayer as a brazing filler

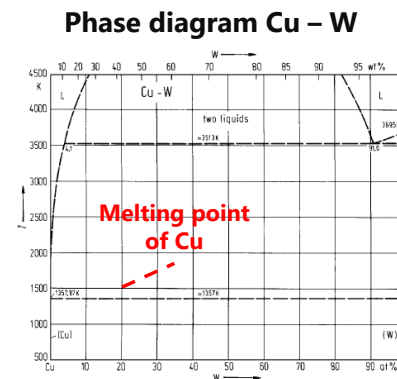
Requirements: high-strength joints at $T < 750^{\circ}\text{C}$

Approach



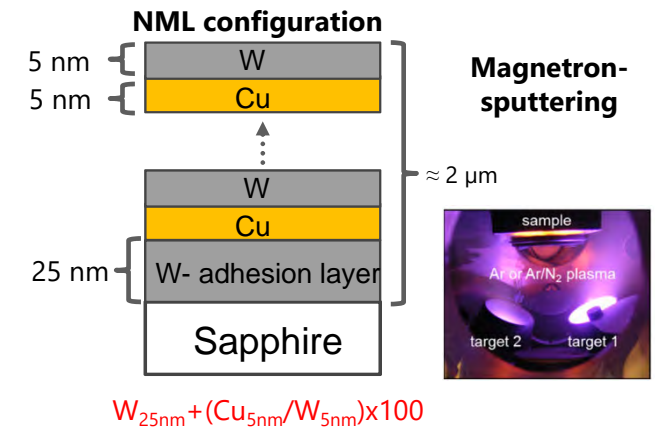
- Cu alloys reinforced with W-particles offer attractive mechanical properties
- NML approach with to join at relative low temperatures and to form in-situ a high-strength composite (transformation of NML to particular-based composite)

Material System



- Immiscible system
- No solid state phase transformations up to the melting point of Cu
- Large CTE mismatch

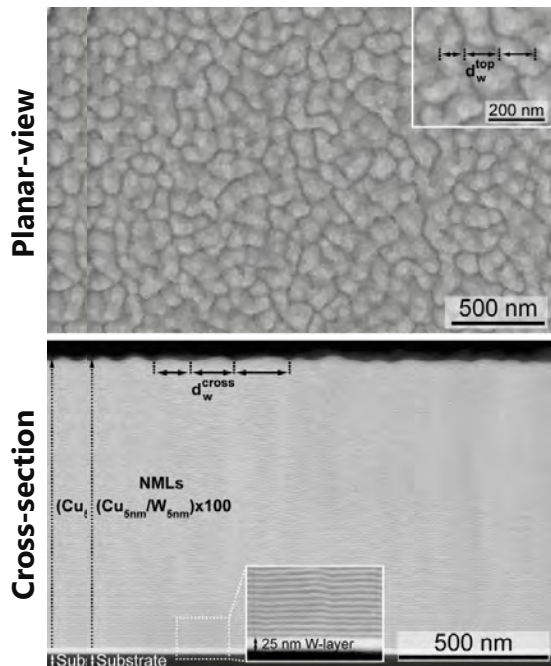
Methods



- Characterization via SEM, XRD
- Isothermal treatments experiments performed between $400\text{-}800^{\circ}\text{C}/100 \text{ min}$ (in vacuum)
- Isothermal *in-situ* HT-XRD ($675\text{-}725^{\circ}\text{C}$) to study kinetic of the structural transformation

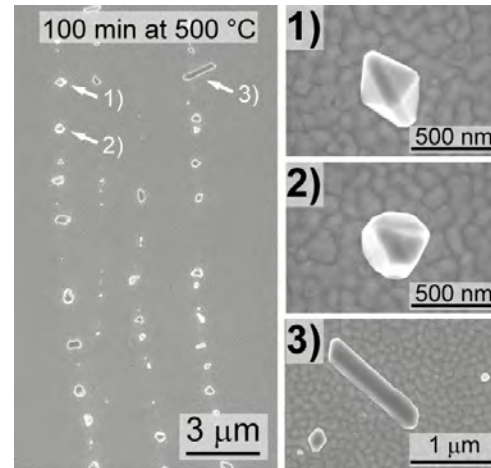
Thermal Stability of Cu/W (SEM investigations)

As deposited NML



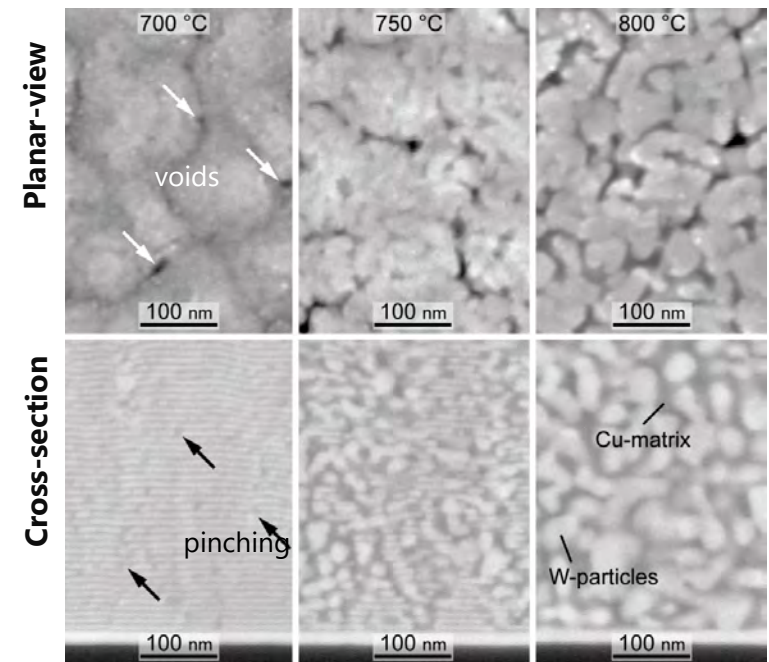
- Increase in layer waviness with increasing number of repetitions
- Semi-coherent interface between Cu and W layers

First structural changes



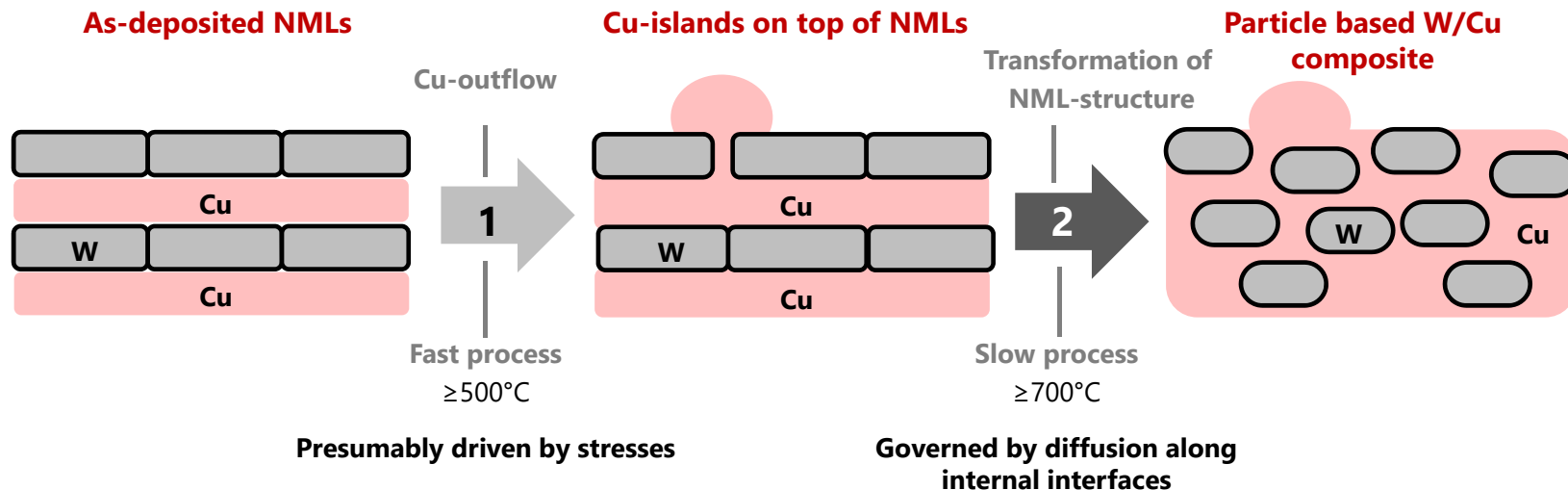
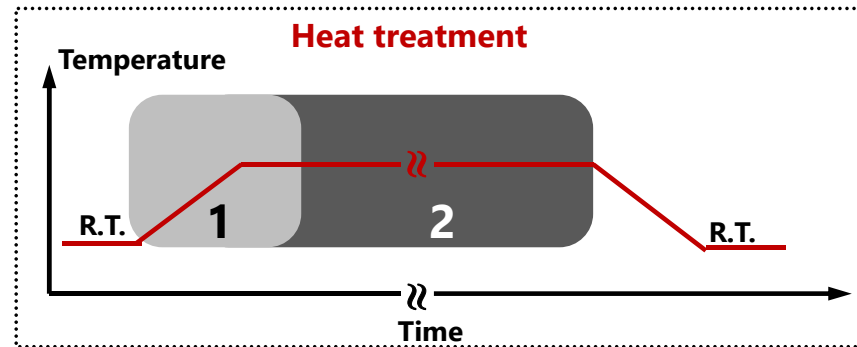
- Starting from 500°C a high number of lines with faceted Cu particles appear on the surface
- No severe defects (crack, voids)

Transformation from NML to nanocomposite

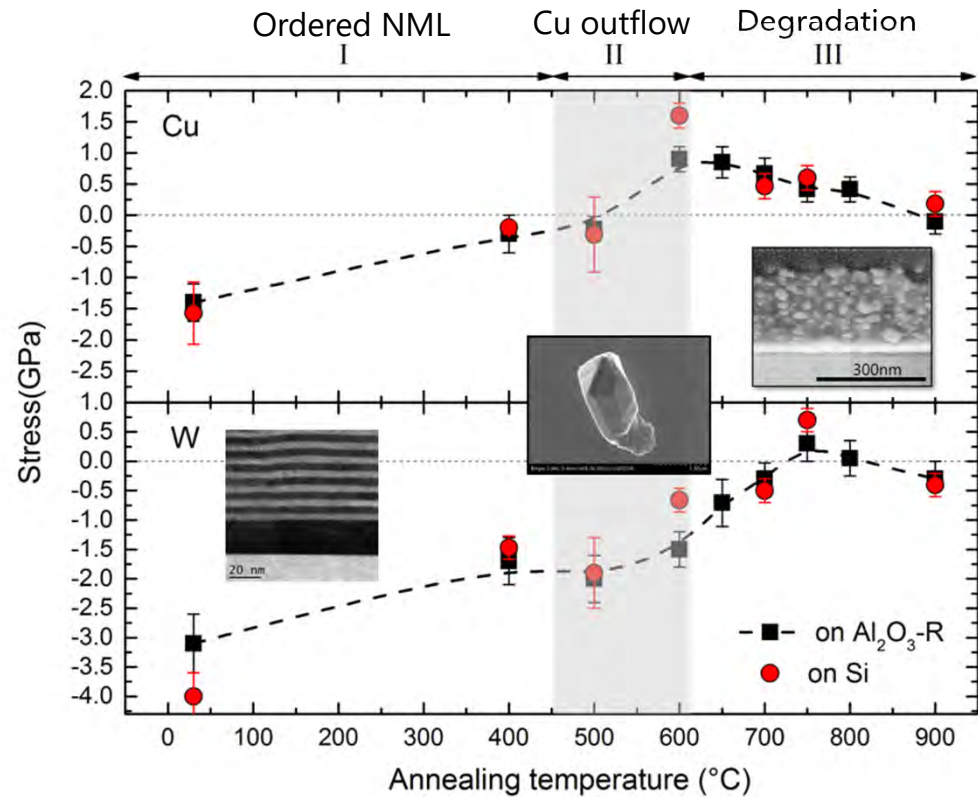


- $T > 650^\circ\text{C}$: Degradation of the NML (formation of voids at the surface and pinching of the layers) and formation of a spheroidized Cu-W nano-composite
- NML degradation progresses is completed at 800°C

Structural evolution of Cu/W upon heating



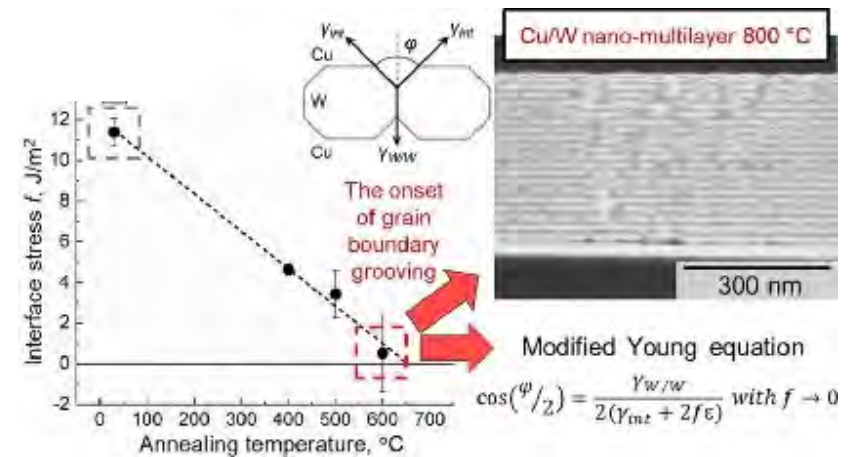
Effect of stress on GB diffusion in Cu/W NMLs



Refs.:

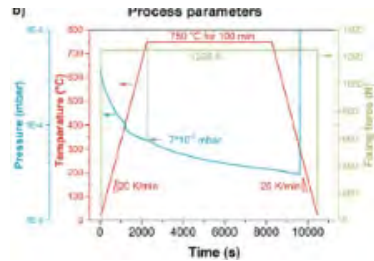
A. V. Druzhinin, et. al. Appl. Surf. Sci., 2020, 508, 14254

A.V. Druzhinin, et. al., Materialia, 2019, 7. 100400



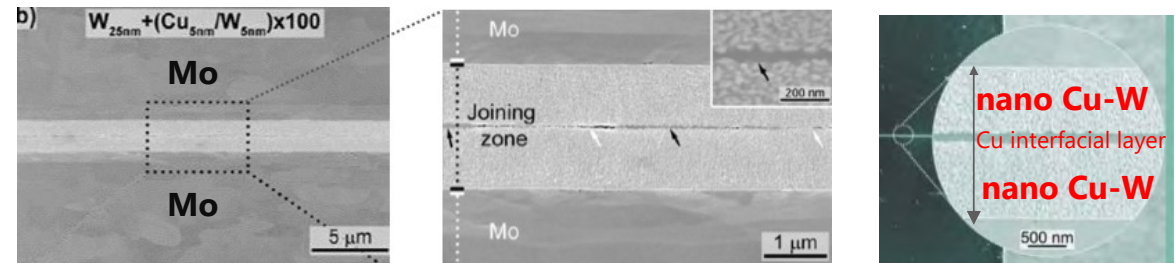
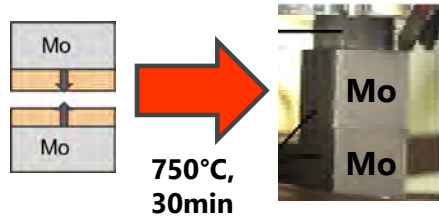
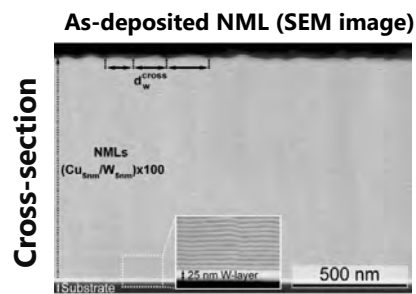
High compressive stresses in the W barrier layer increase the effective activation energy for Cu diffusion in W GBs (by a factor of ~1.5) and considerably slow down the diffusion kinetics

Joining with Cu/W nanomultilayers: proof-of concept



High strength refractory metal joints with Cu/W NML @ 750°C

Refs: F. Moszner et al, J Mater Sci Eng B 6 (2016) 226



In-situ formation of a nanocomposite in a joint during brazing process

High strength joints can be achieved with NMLs at reduced brazing temperatures

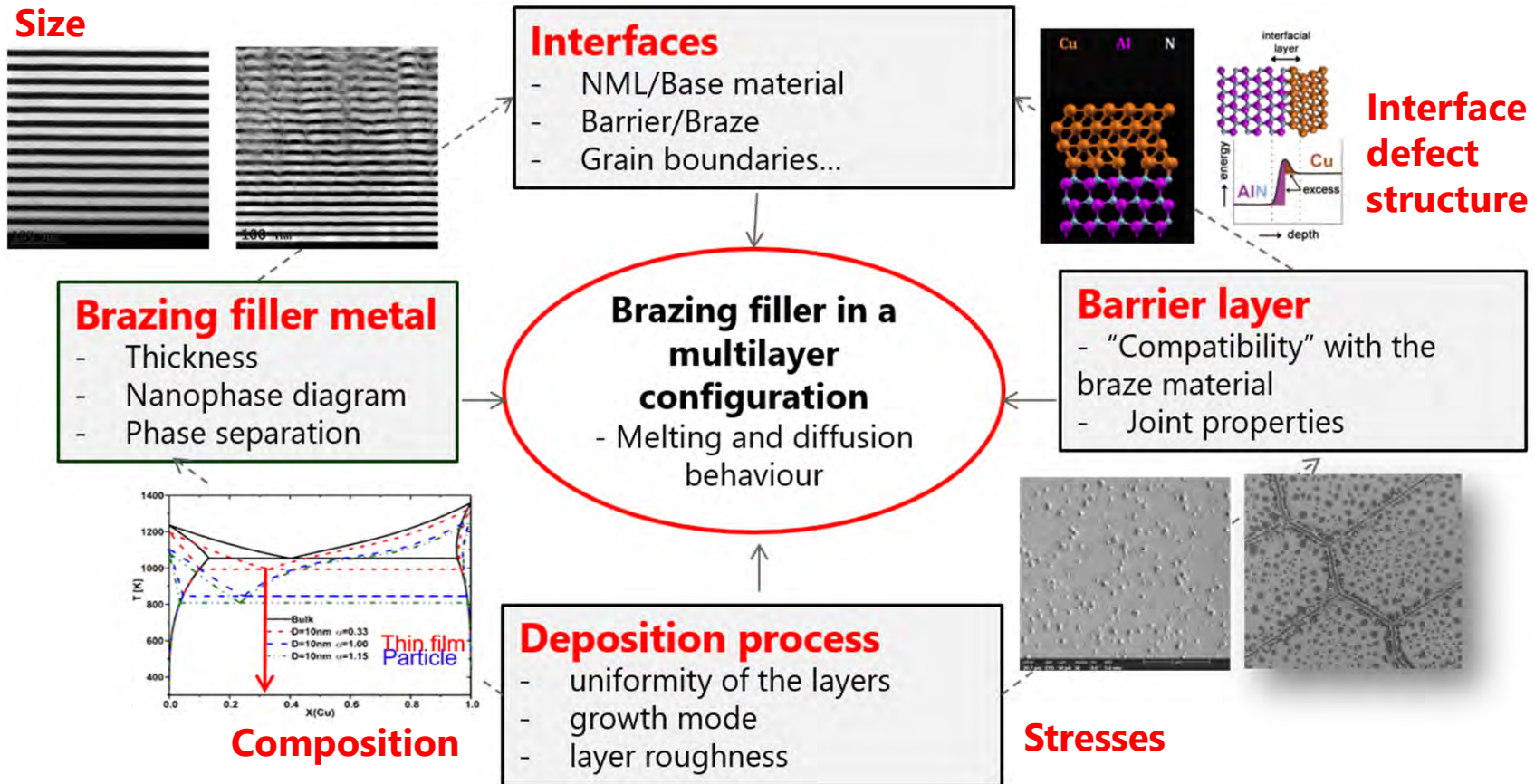
Lessons learned and outlook case study 4 (Cu/W)

- High strength nanocomposite can be in-situ formed during the bonding process when using NMLs of immiscible metallic systems such as Cu-W as a bonding material
- The temperature of the transformation process can be controlled by the interfacial stress

Outlook:

- Engineering of NMLs of immiscible metals for joining application and thermal management
 - NMLs with desired functional properties
- Deeper understanding of the behaviour NMLs of immiscible metals
 - In-situ control of internal stresses during the NML deposition
 - Combining experiments with modelling

NMLs offer a great “engineering tool”



but the new phenomena and complex relationships between the design parameters need to be fully understood

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Current Joining Team

- ✓ Dr. Lars Jeurgens
- ✓ Dr. Bastian Rheingans
- ✓ Tobias Burgdorf
- ✓ Dr. Hans-Rudolf Elsener
- ✓ Dr. Claudia Cancellieri

Former Team Members

- ✓ Dr. Vicente Araullo-Peters
- ✓ Dr. Vinzenz Bissig
- ✓ Dr. Mirco Chiodi
- ✓ Dr. Joanna Lipecka
- ✓ Dr. Frank Moszner
- ✓ Dr. Giancarlo Pigozzi
- ✓ Dr. Sebastian Siol
- ✓ Benjamin Lehmert

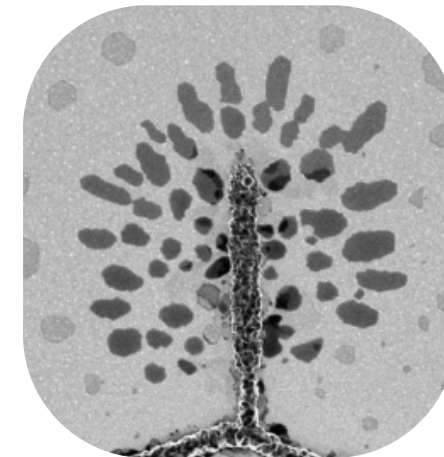
International Collaborators

- ✓ Prof. Malgorzata Lewandowska, Warsaw University of Technology, Poland
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- ✓ Prof. Rafal Abdank-Kozubski, Jagiellonian University, Poland
- ✓ Prof. Matthias Türpe, Mahle GmbH, Germany
- ✓ Prof. George Kaptay, Bay Zoltan Nonprofit Ltd., Hungary
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- ✓ Prof. Norman Zhou, University of Waterloo, Canada
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- ✓ Prof. Daniel Ariosa, Universidad de la República, Uruguay
- ✓ Dr. S. Yoon, University Stuttgart, Germany

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SWISS PHOTONICS



Thank you for your attention!

Follow-up study on Al-Si/AlN NMLs

- The description of underlying mechanisms and microstructural characteristics that govern the phase stability, diffusion and pre-melting behaviour of nano-confined Al and Al-Si alloys in a NML (Barrier: AlN)
- A significant contribution to the understanding of the melting point depression/superheating phenomena and of phase stability of nano-confined alloys



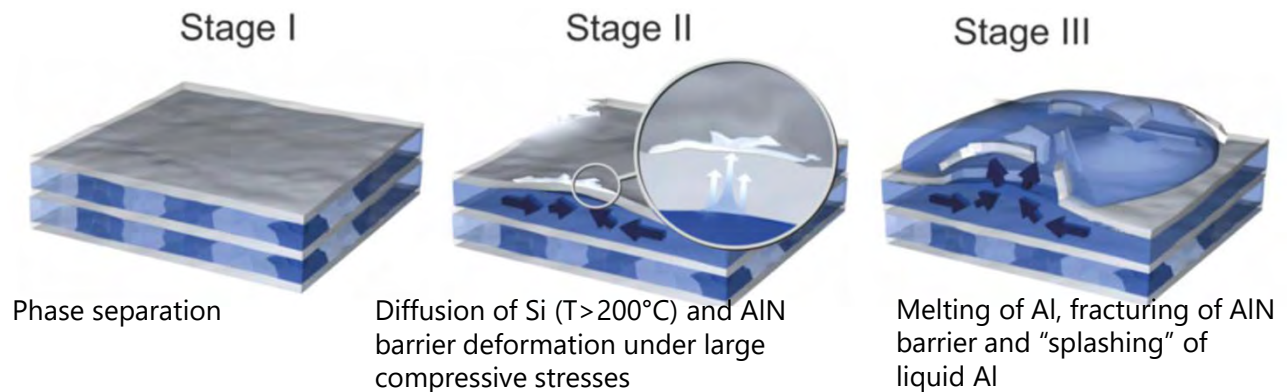
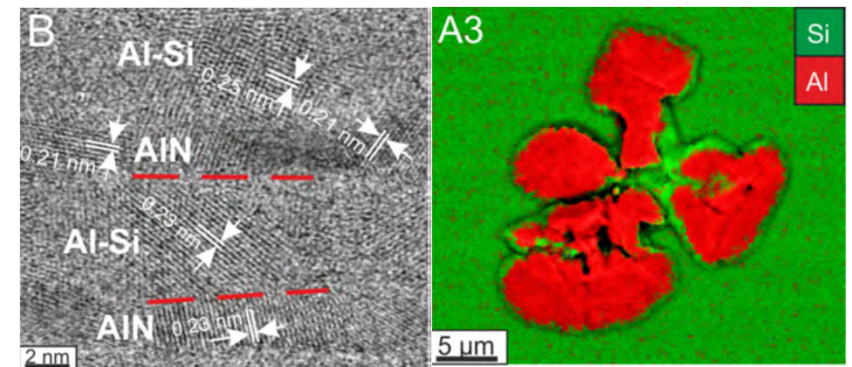
WARSAW UNIVERSITY OF TECHNOLOGY
FACULTY OF MATERIALS SCIENCE AND ENGINEERING

Ph.D. Thesis

Joanna Lipecka, M.Sc.

Phenomenon of melting point depression
in Al-Si/AlN nanomultilayer system and its application
in joining of ultrafine grained aluminium alloys

Supervisor
Professor Malgorzata Lewandowska, Ph.D., D.Sc.



Schema of the microstructural changes of an Al-Si12%at.4nm/AlN3nm NML upon heating