An Investigation into Microstructure & Mechanical Behavior of Al Alloy Coatings Deposited by Cold Spray Processing

Ph.D. Dissertation Defense

Reza Rokni

Committee Members:

Dr. Christian Widener
Dr. Grant Crawford
Dr. Michael West
Dr. Phil Ahrenkiel
Dr. Bharat Jasthi

South Dakota School of Mines and Technology

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Outline

- ➢ Introduction
- ➢ Background
- ➢ Motivation for this work
- ➢ Results from this work
  - ✓ Microstructure characterization
  - ✓ Mechanical properties
- ➢ Conclusions

Overview of Cold Spray Technology

Cold gas dynamic spraying or CS is a unique solid-state materials consolidation process that utilizes high velocity particles impinging upon a substrate to build up coatings and/or freestanding structures without the use of combustion fuels.

Main Parameters:

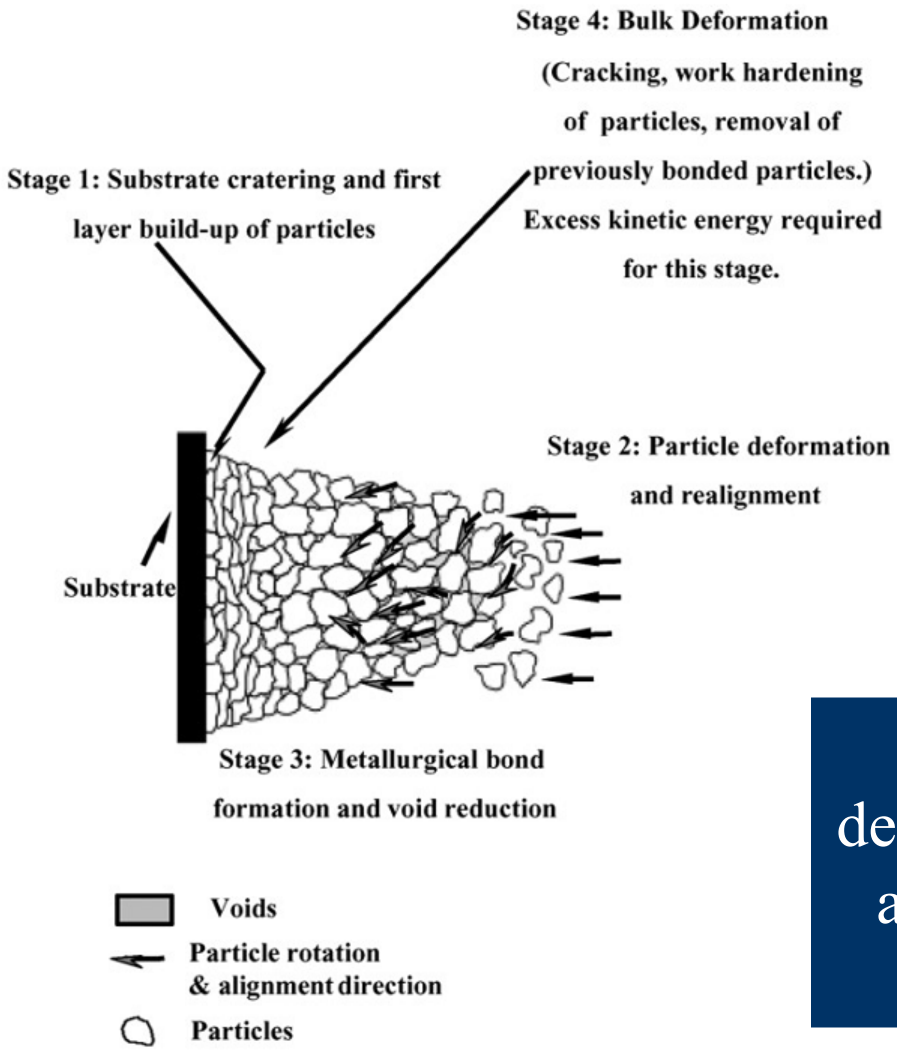
- ✓ Main Gas Pressure
- ✓ Gas Temperature
- ✓ Particle Velocity
- ✓ Particle Size
- ✓ Main Gas Flow Rate
- ✓ Powder Feed Rate

Applications:

- ✓ Dimensional Restoration
- ✓ Wear Resistant Coatings
- ✓ Casting Repair
- ✓ Electronics (metalizing ceramics)
- ✓ Fabrication of Parts
- ✓ Novel Weapon Systems

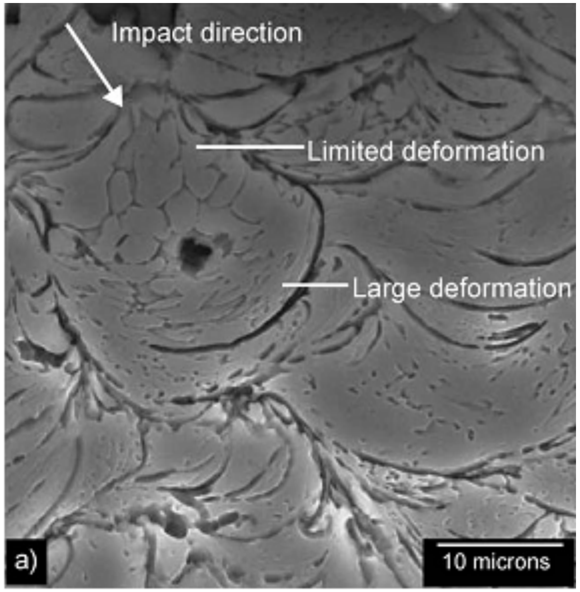
The major problem with cold sprayed coatings is having inferior mechanical properties in compared with bulk materials.

Background



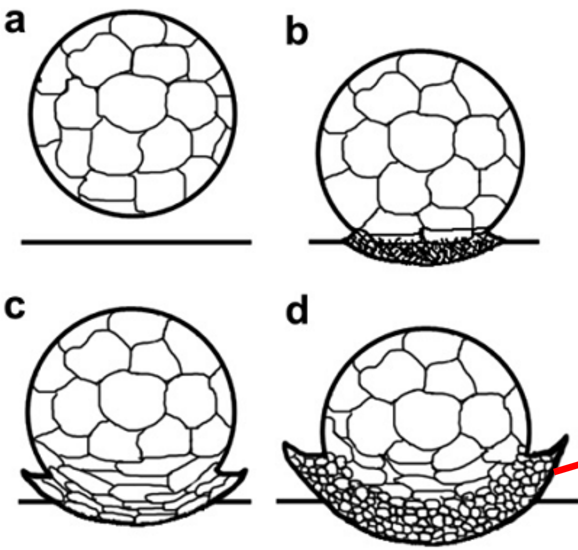
T. Van Steenkiste, J. Smith, R. Teets, Surf. Coat. Technol. 154 (2002) 237-252.

R. Morgan, P. Fox, J. Pattison, C. Sutcliffe, W. O' Neill, Mater. Letters 58 (2004) 1317–1320.

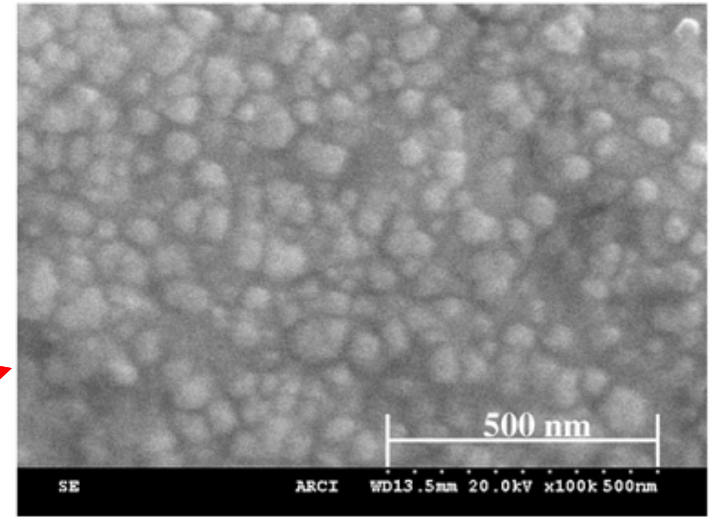


CS imposes high degree of deformation on the powder particles and generates an inhomogeneous microstructure.

Background



K. Kim, M. Watanabe, S. Kuroda, Surf. Coat. Technol. 204 (2010) 2175–2180.



H. Assadi et al. Acta Mater. 51 (2003) 4379–4394.

(b) Generation of a high impact region and the resultant intensive shear stress

(c) Formation of dislocation structures and pancaked grains

(d) Recrystallized grains in some areas

Motivations of this work

Addressing Concerns About:

- ✓ Limited attention has been dedicated to microstructural study of cold spray processed (CSP) alloys.
- ✓ Limited study on characteristics of the microstructure in different areas of the deposit.
- ✓ Lack of information in the literature on formation mechanisms for the microstructural features in deposited material.
- ✓ Lack of enough data on local and bulk mechanical properties.

There is a need for a systematic study relating:
Processing - Microstructure - Mechanical Behavior.

Research Thrusts

Cold Spray Processing

- ✓ Different aluminum alloys, 6061, 5083 and 7075
- ✓ Non-isothermal Annealing
- ✓ Heat treatments

Microstructure Characterization

- ✓ Grain structure (EBSD/BSE)
- ✓ Precipitates (SEM/DSC/EDS)
- ✓ Dislocation density (XRD/TEM)
- ✓ Lattice parameter (XRD)
- ✓ Microstrain (XRD)

Deformation Behavior

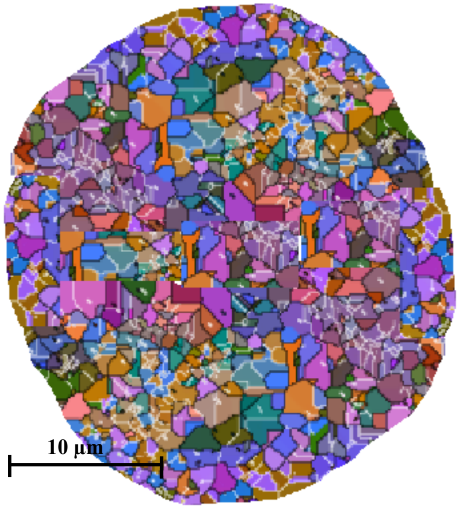
- ✓ Microhardness
- ✓ Nanohardness
- ✓ Microtensile

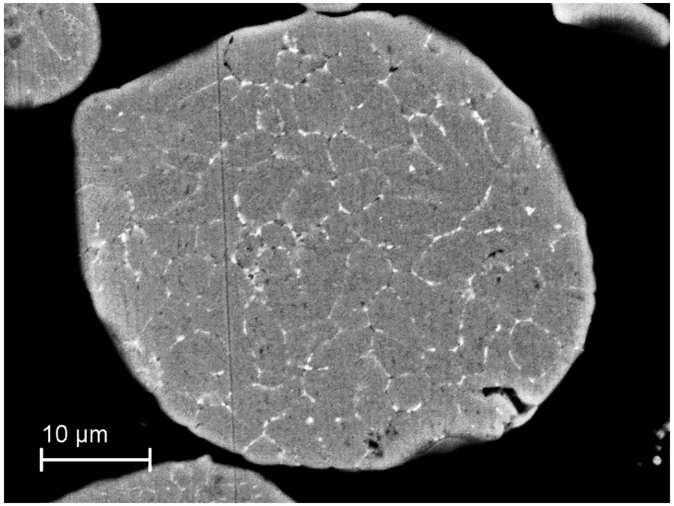
Gas-atomized 6061 Al powder and the CSP deposit

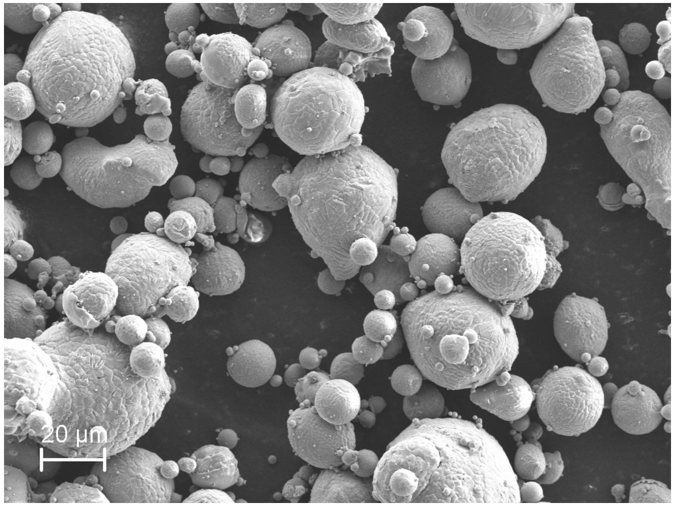
Powder Morphology and Structure

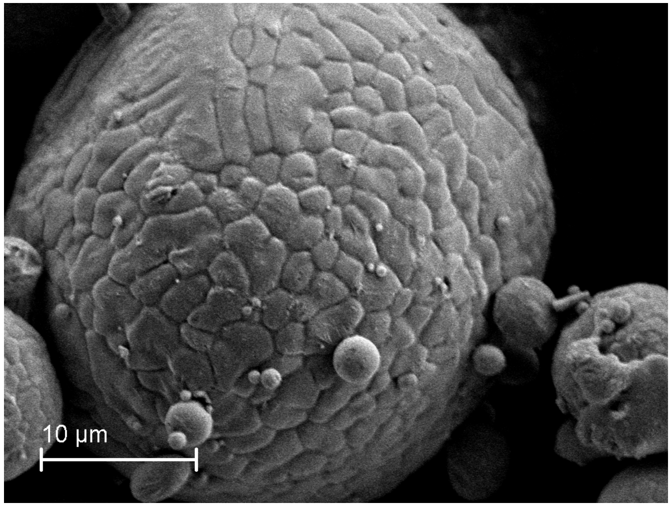
Particle Size Analysis

Size range of 5-50 µm and average size of 38.7 µm



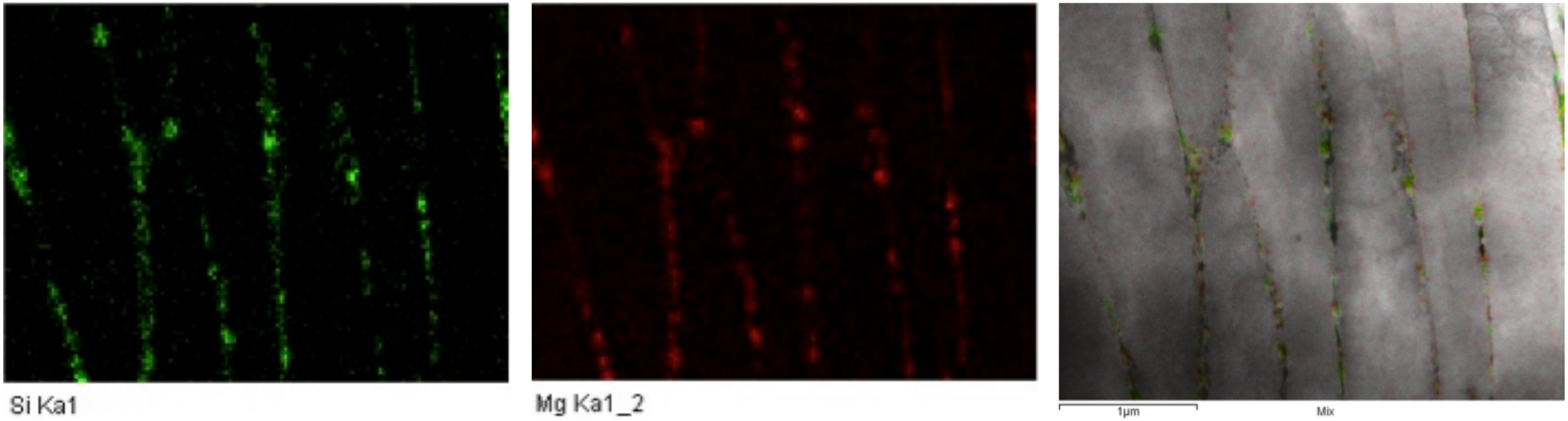






- Powder grains are not relaxed
- **Medium and uniform DD**
- Solute segregation

GB Solute segregation



- More extreme solute segregation at the grain boundaries

Element	Wt% (P. Gbs)	Wt% (P. Matrix)	Wt% (CSP layer Matrix)
Al	88.6	97.4	98.4
Mg	2.2	1.0	0.7
Si	4.5	1.0	0.7
Fe	3.2	-	-
Cu	1.5	0.6	0.2
Total	100	100	100

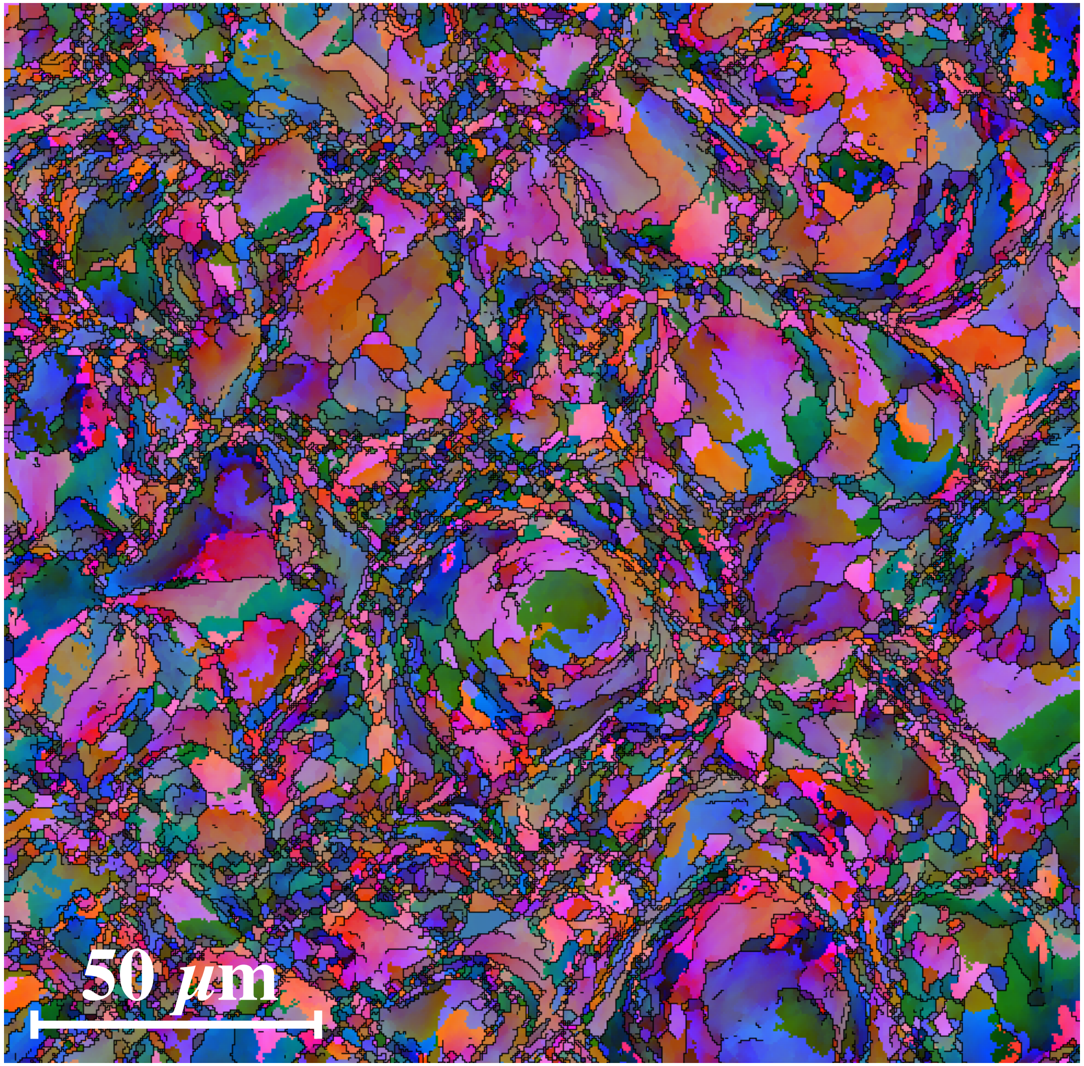
M.R. Rokni, C.A. Widener, V.R. Champagne, Microstructural stability of ultrafine grained cold sprayed 6061 Al alloy, Appl. Surf. Sci. 290 (2014) 482-489.

M.R. Rokni, C.A. Widener, S.P. Ahrenkiel, B.K. Jasthi, V.R. Champagne, Annealing Behavior of 6061 Al Deposited by High Pressure CS, Surface Engineering 30 (2014) 361-368.

CSP deposition (EBSD)

✓ The microstructure of the deposition is ***inhomogeneous.***

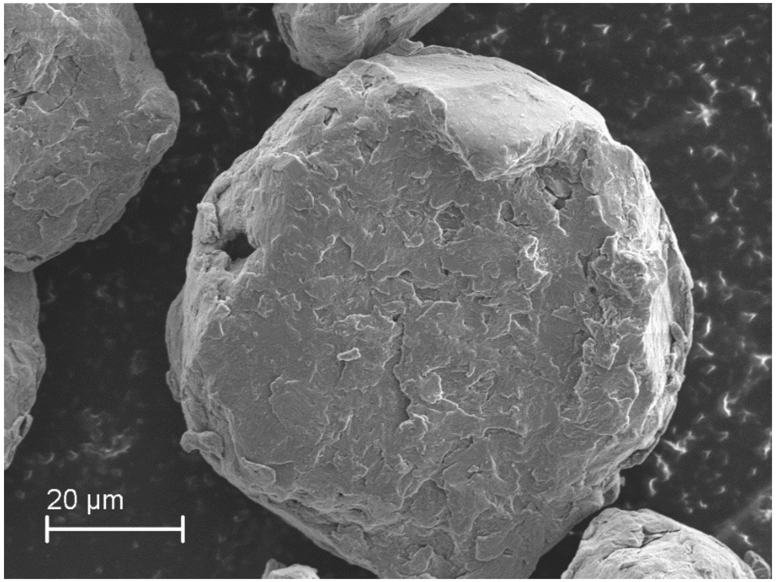
- No preferred texture
- Less deformation around the center
- UFG structure at the particle interfaces

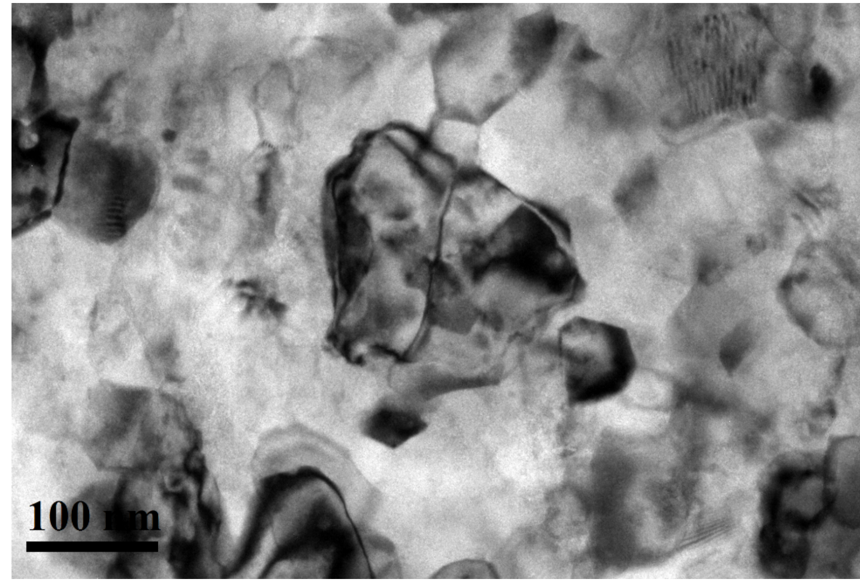


M.R. Rokni, C.A. Widener, V.R. Champagne, Microstructural Evolution of 6061 Al Gas-Atomized Powder and High-Pressure Cold-Sprayed Deposition, Journal of thermal spray technology 23 (2014) 514-524.

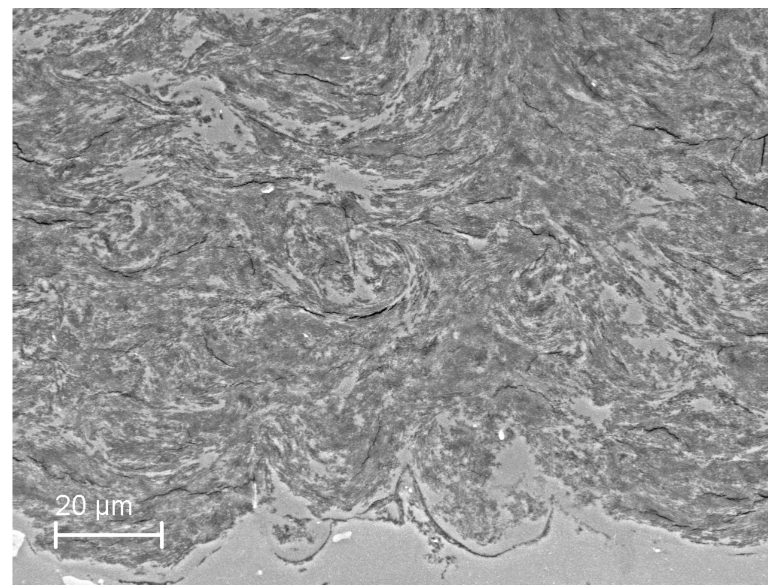
Nanocrystalline 5083 Al powder and the CSP deposit

5083 Nanocrystalline





- ➢ Incoming grain structure is important.
  - ✓ Different microstructural features
  - ✓ Different deformation mechanisms
  - ✓ Different mechanical behavior



M.R. Rokni, C.A. Widener, A.T. Nardi, V.K. Champagne, Nano crystalline high energy milled 5083 Al powder deposited using CS, Applied Surface Science 305 (2014) 797-804.

Key Messages:

- Cold spray can be a promising technique for producing bulk alloy materials.

- The gas-atomized Al alloy powders contain grain boundary solute segregation retained in deposition.

- The microstructure of deposited materials is inhomogeneous and includes distinct microstructural regions.

Gas-atomized 7075 Al powder and CSP deposit

Why Al 7075?

- Aluminum is an abundant resource
- Relatively cheap
- High stiffness/density and strength/density ratios
- Damage tolerant
- Corrosion resistant compared with conventional alloys

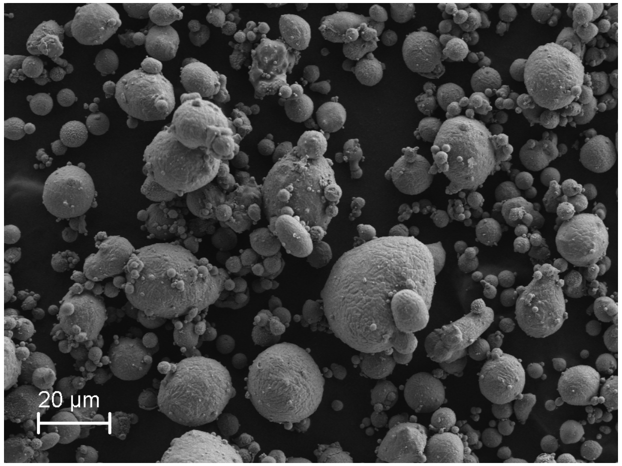
Applications
- Gears and shafts
- Aircraft
- Aerospace and defense applications

Objectives & Experimental Procedure

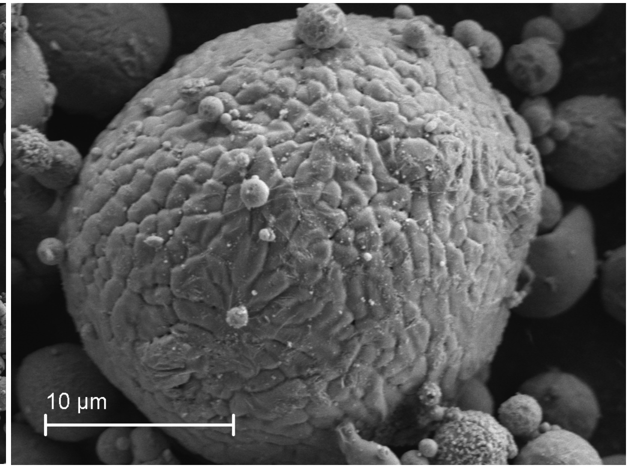
- ✓ 7075 Al coatings were produced using commercially available gas-atomized 7075 Al powder.
- ✓ Microstructural characterization of the as-received powder and the coatings via different microscopy techniques including, TEM, SEM and EBSD.
- ✓ In-situ hot-stage TEM from room temperature to 450ºC.
- ✓ Differential scanning calorimetry (DSC)
- ✓ X-ray diffraction profiles (XRD)
- ✓ Energy-dispersive X-ray spectroscopy (EDS)
- ✓ Microhardness & Nanohardness testing
- ✓ Micro-tensile testing method

➢ The main objective of this work was to study the microstructure and mechanical properties of cold sprayed 7075 coating in as-deposited and heat treated conditions.

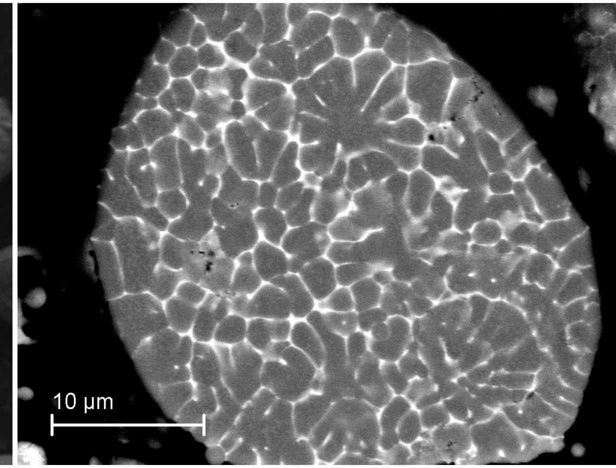
Background – Solute Segregation



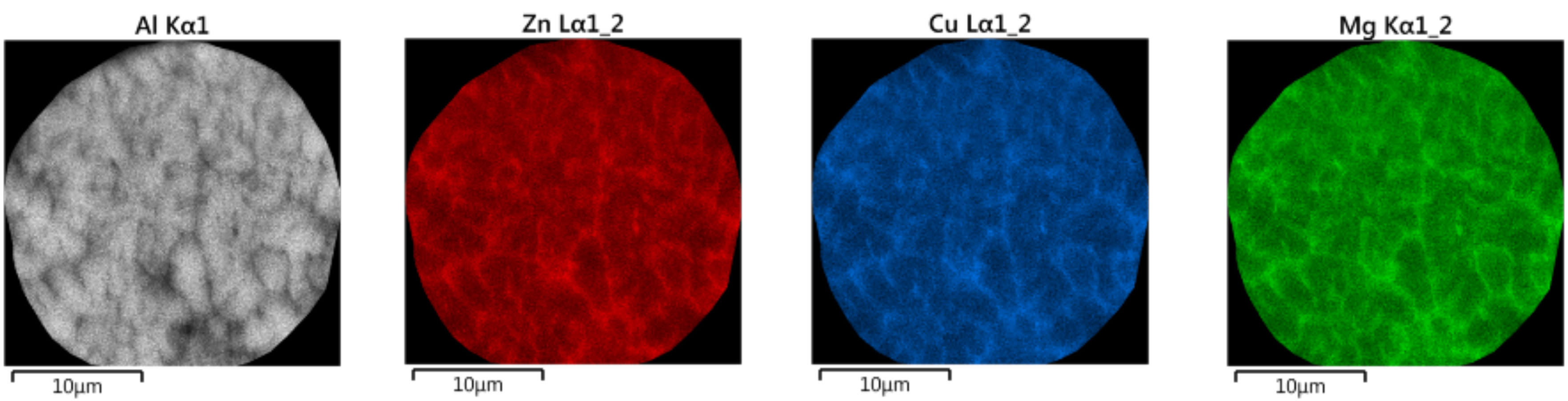
18.6 ± 8.2 µm in size



Surface structure of a ~1-3 µm sized grains

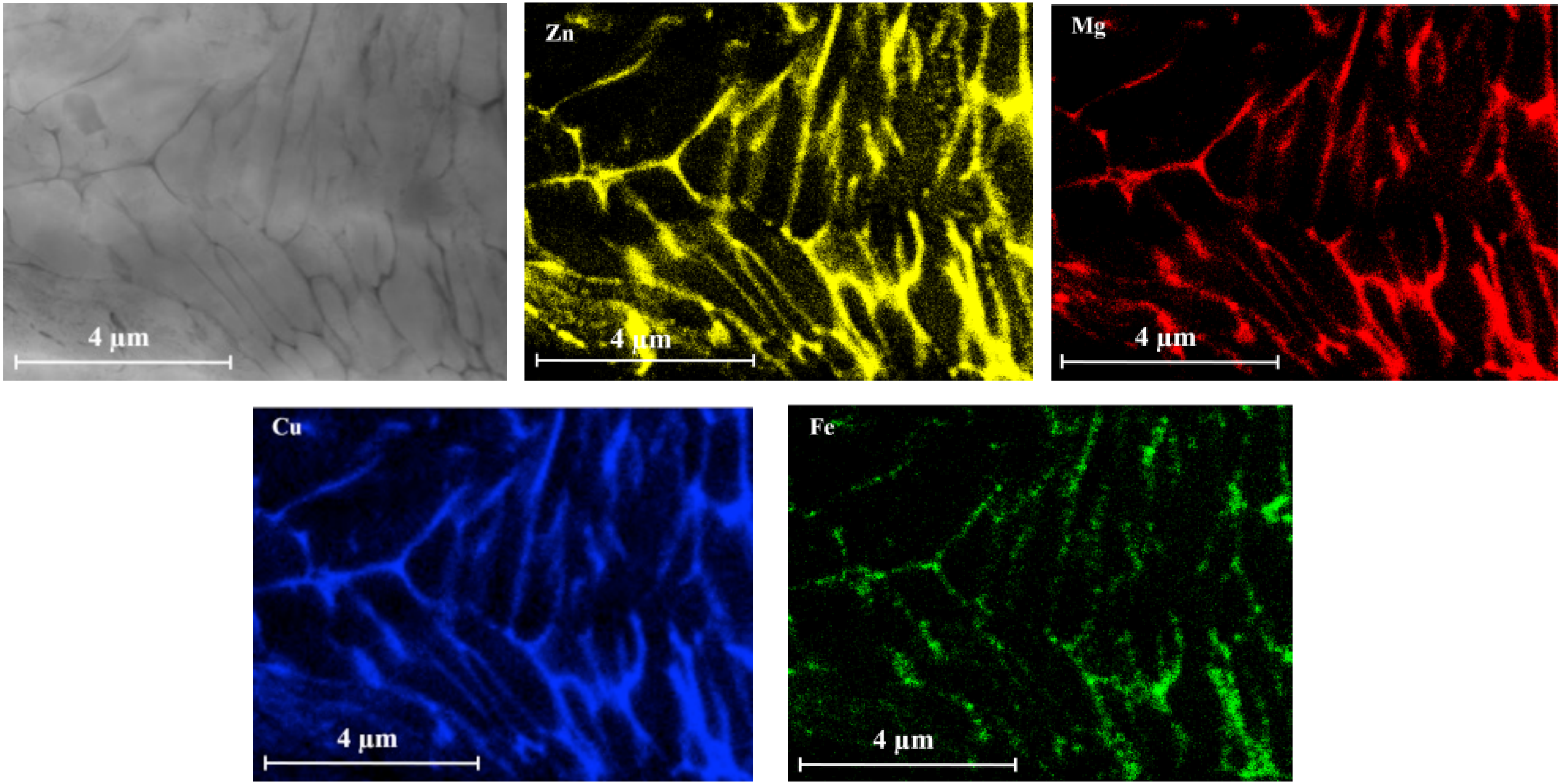


evidence for GB solute segregation



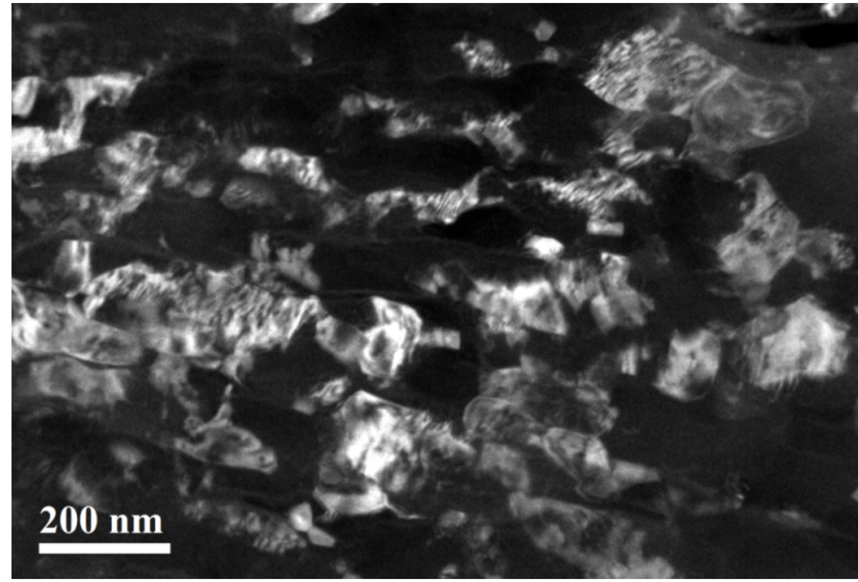
Background – Solute Segregation

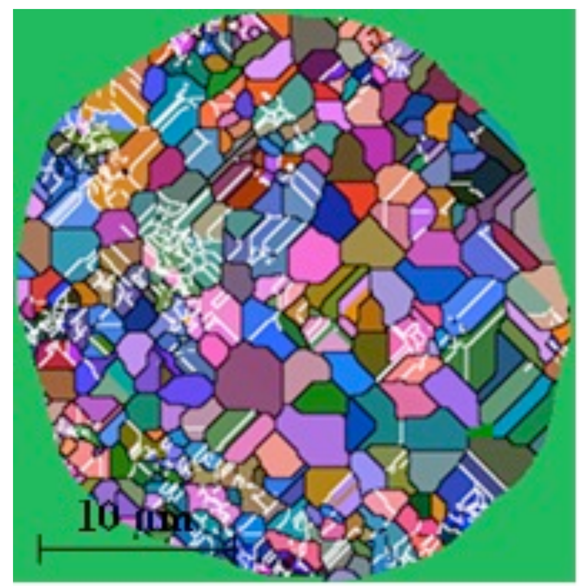
✓ Retention of solute segregation at the grain boundaries (GBs) of the cold sprayed deposition.



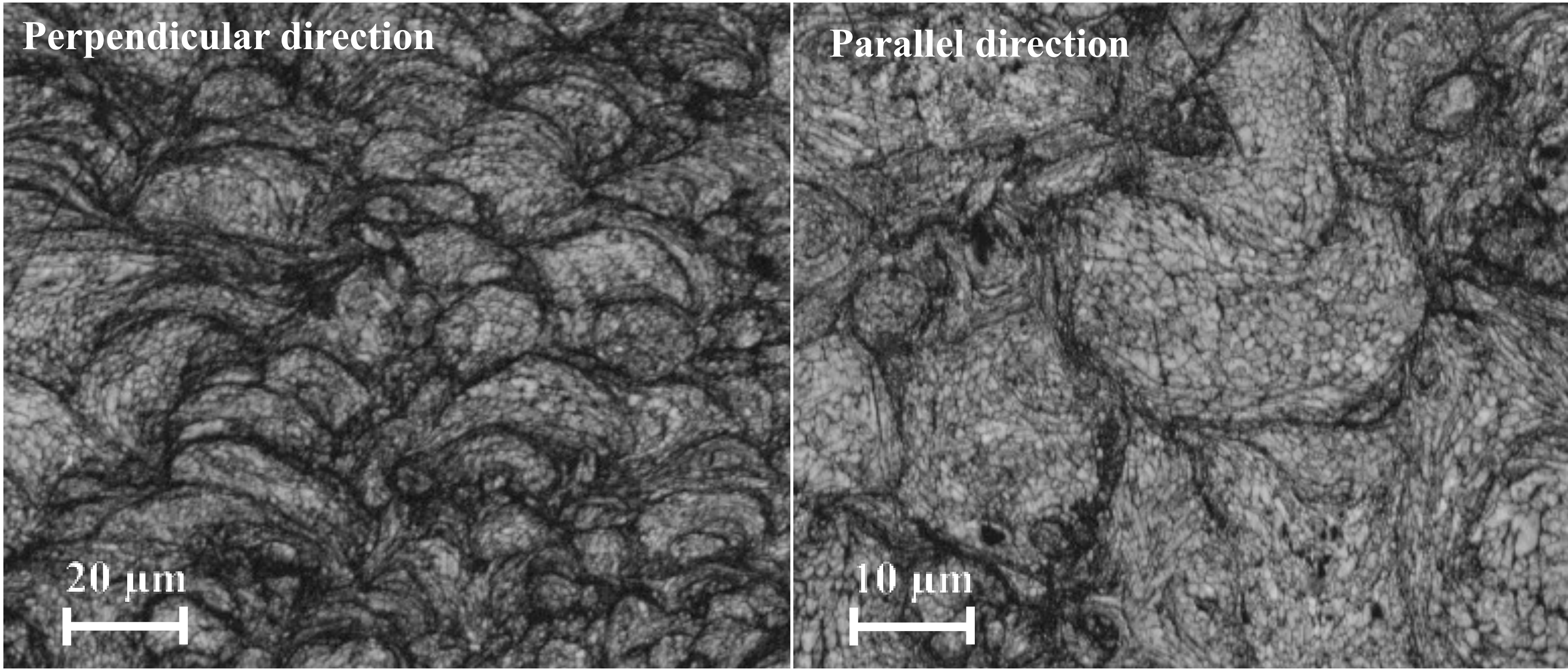
TEM study of the powder

- ✓ Some internal UFG and even nano structures in powder particles
- ✓ Moderate density of dislocations and dislocation substructures
- ✓ Internal HAGBs (black lines) and LAGBs (white lines)



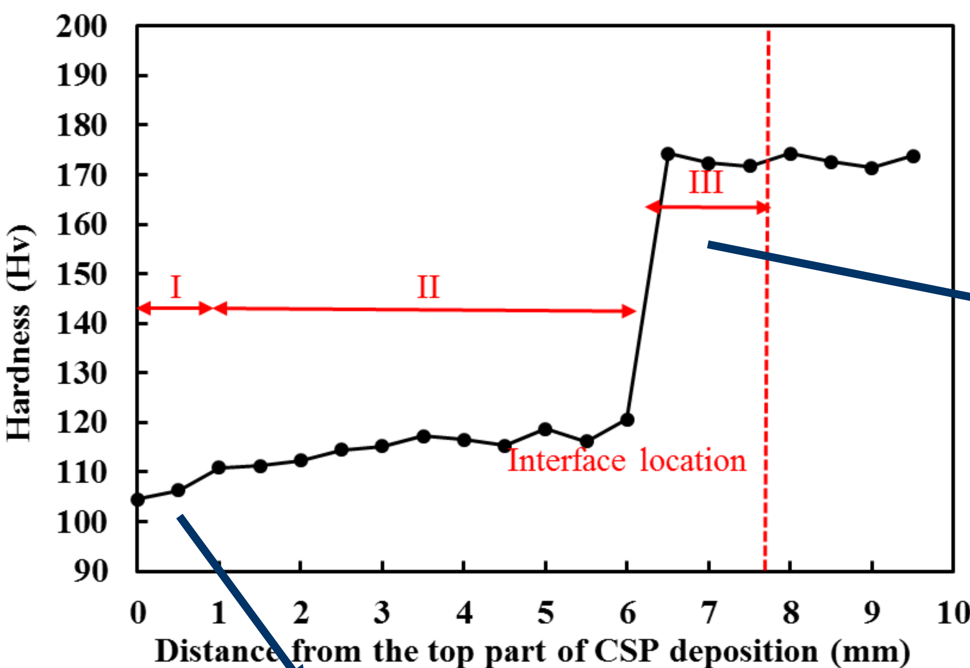


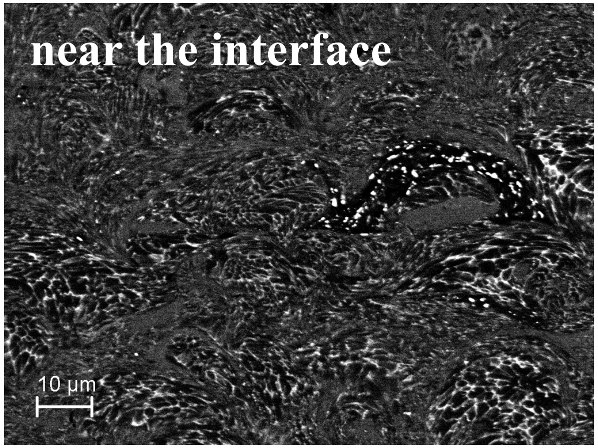
Overall structure of the CSP layer

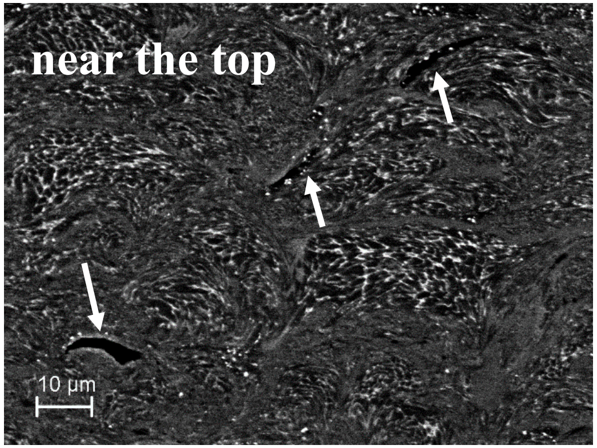


- ✓ Elongation of spherical particles
- ✓ No evidence of voids or porosity

Overall structure of the CSP layer



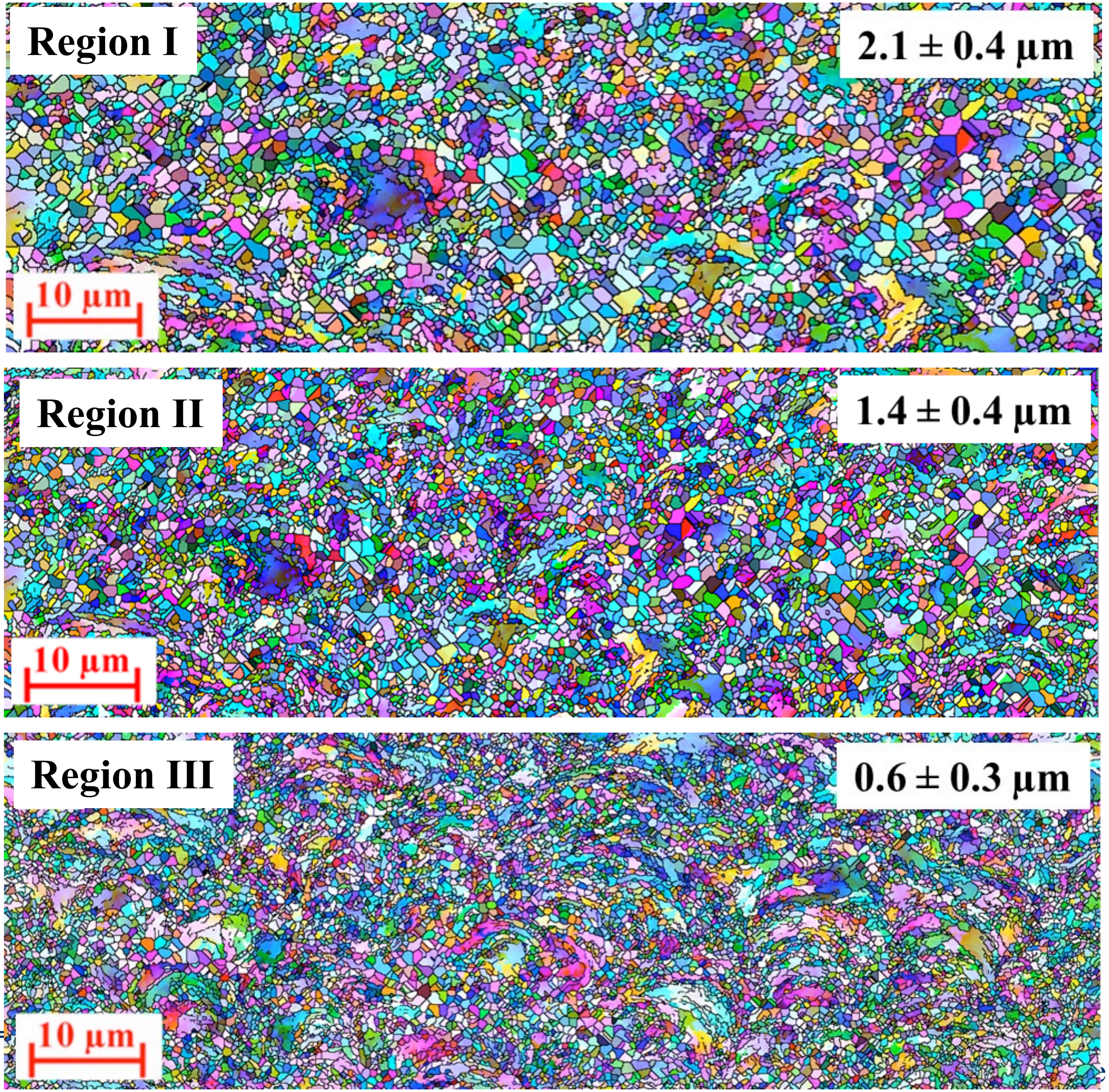




- ✓ Presence of more porosity and void in the top portion of the deposit

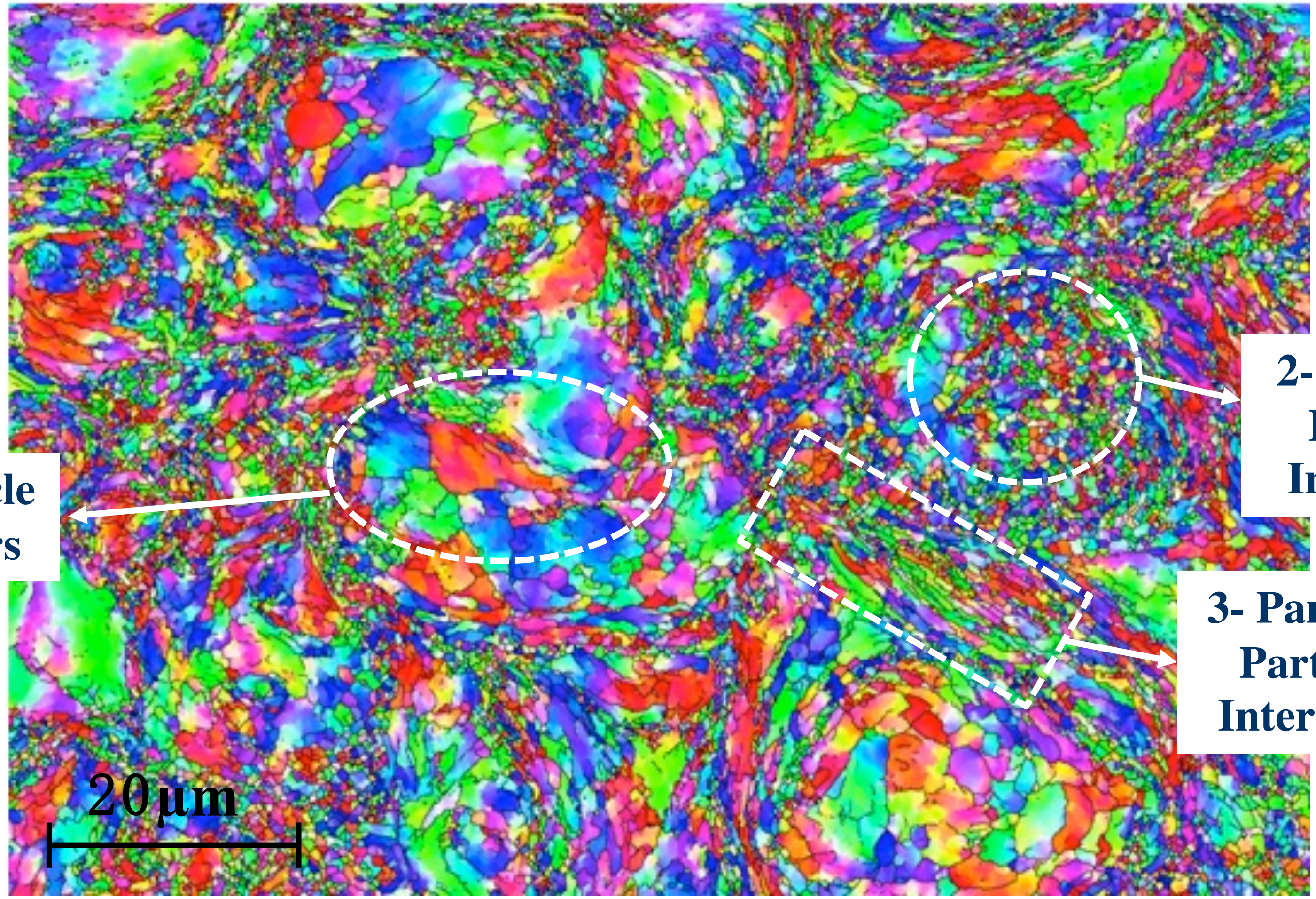
Overall structure of the CSP layer

- ✓ Different grain sizes across the deposition.



EBSD characterization

✓ CS imposes high strains into the particles and generates ***inhomogeneous*** microstructure.



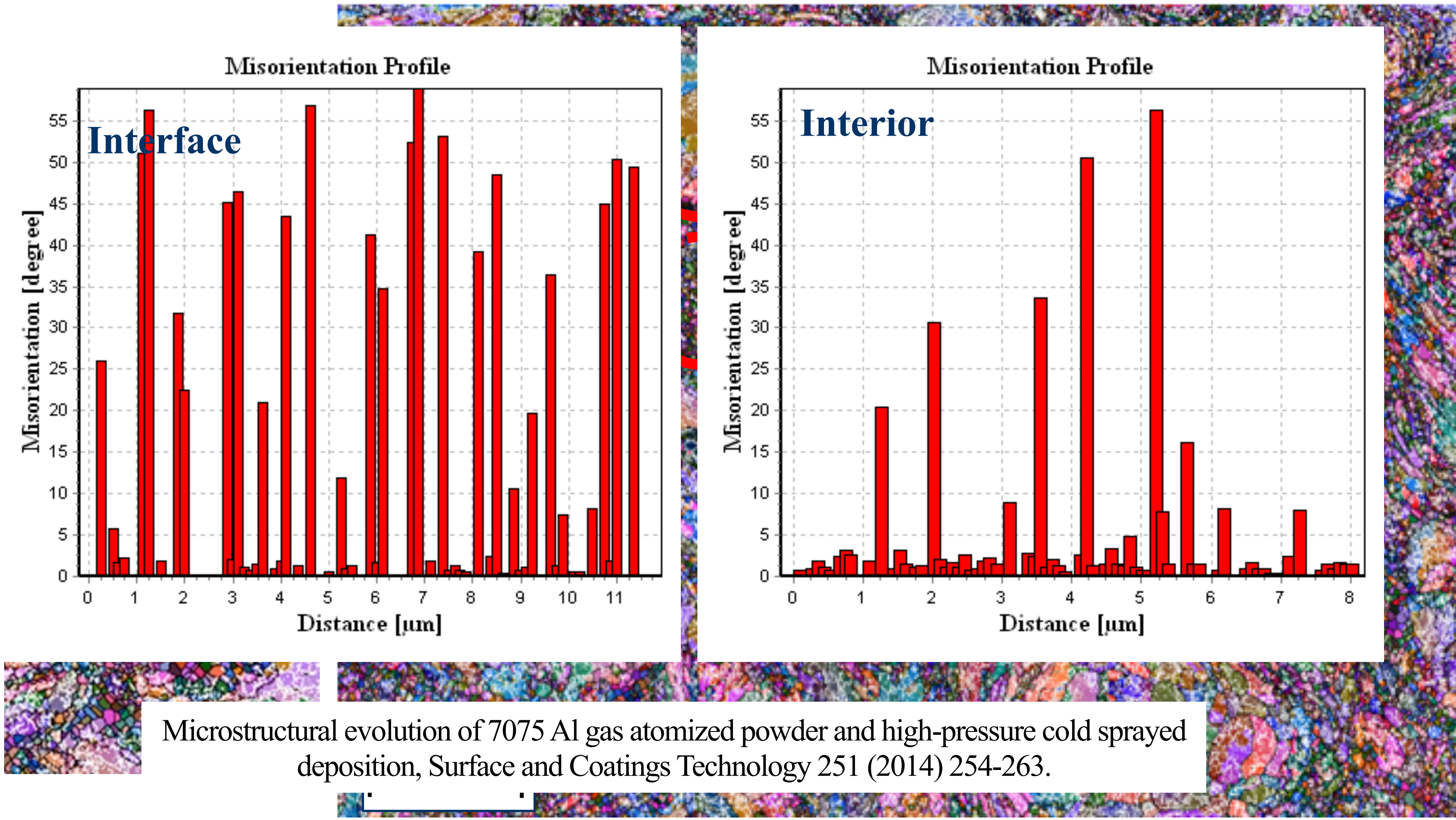
1- Particle Interiors

2- Particle-Particle Interfaces

3- Particle-Particle Interfaces

EBSD characterization

✓A limited number, compared to particle interiors, of LAGBs (dislocations) in these areas.



Microstructural evolution of 7075 Al gas atomized powder and high-pressure cold sprayed deposition, Surface and Coatings Technology 251 (2014) 254-263.

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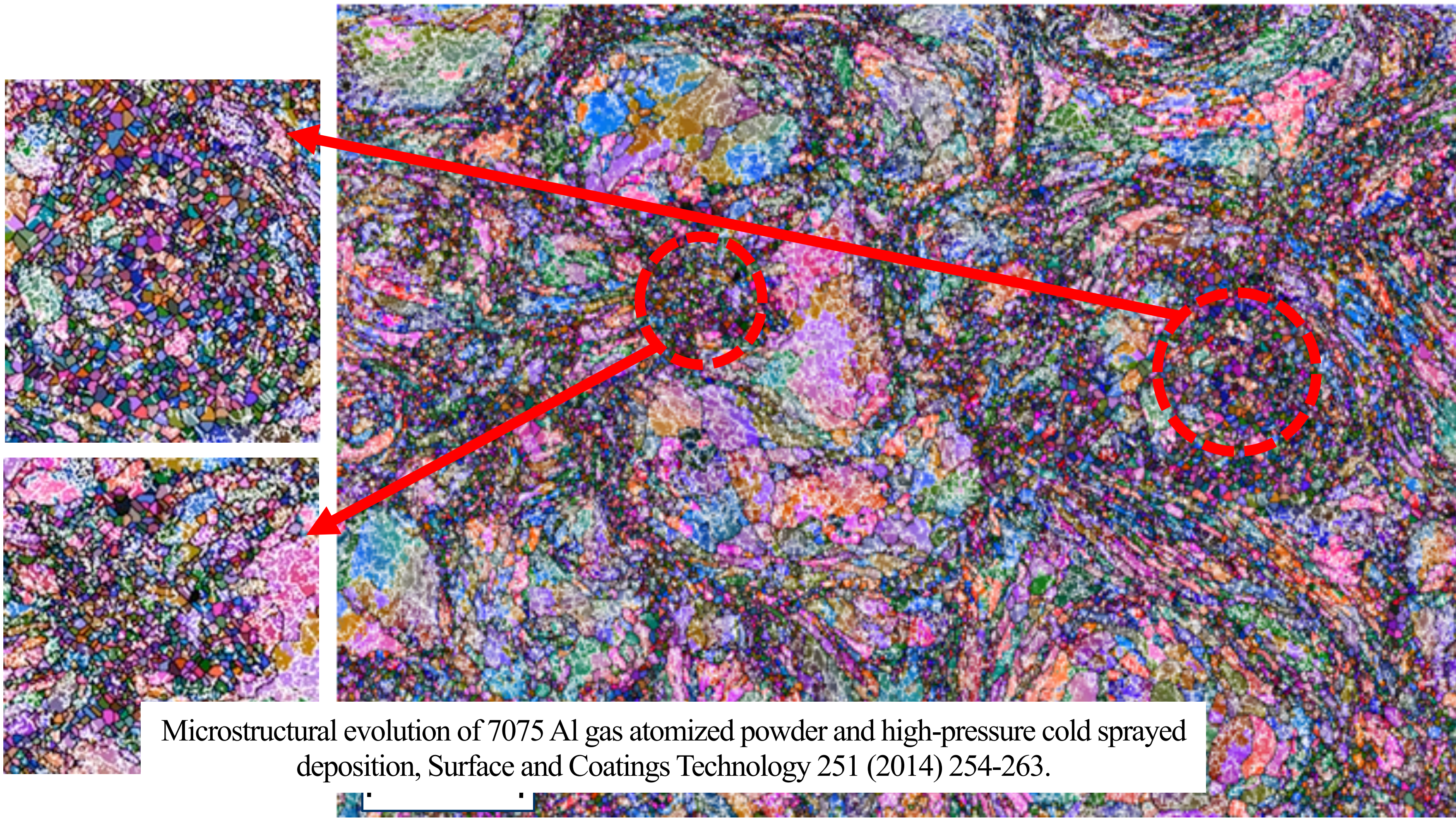

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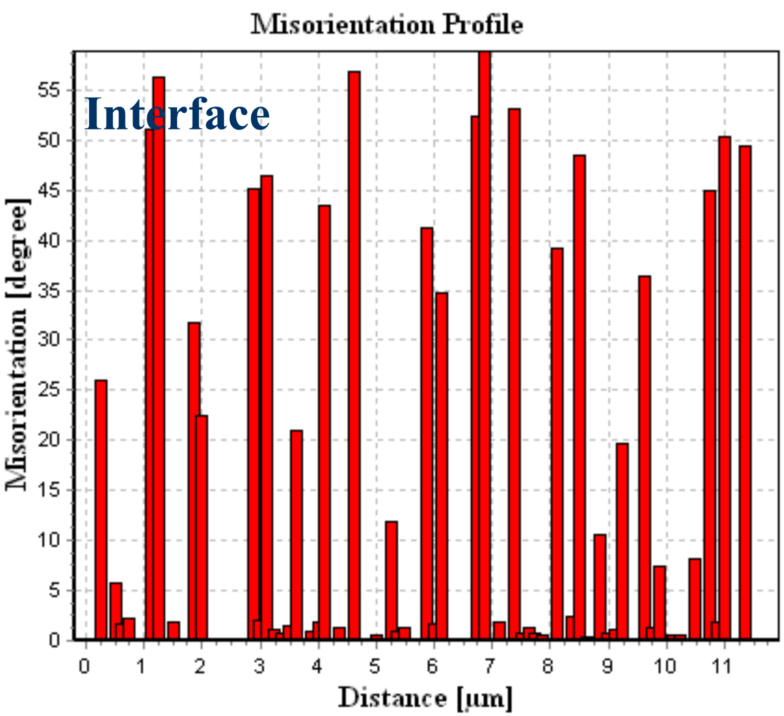
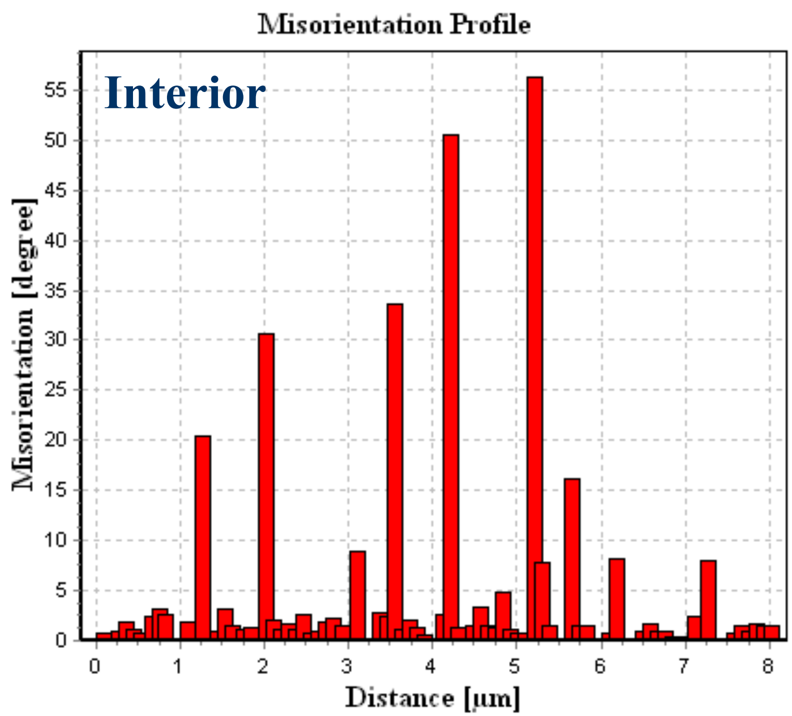
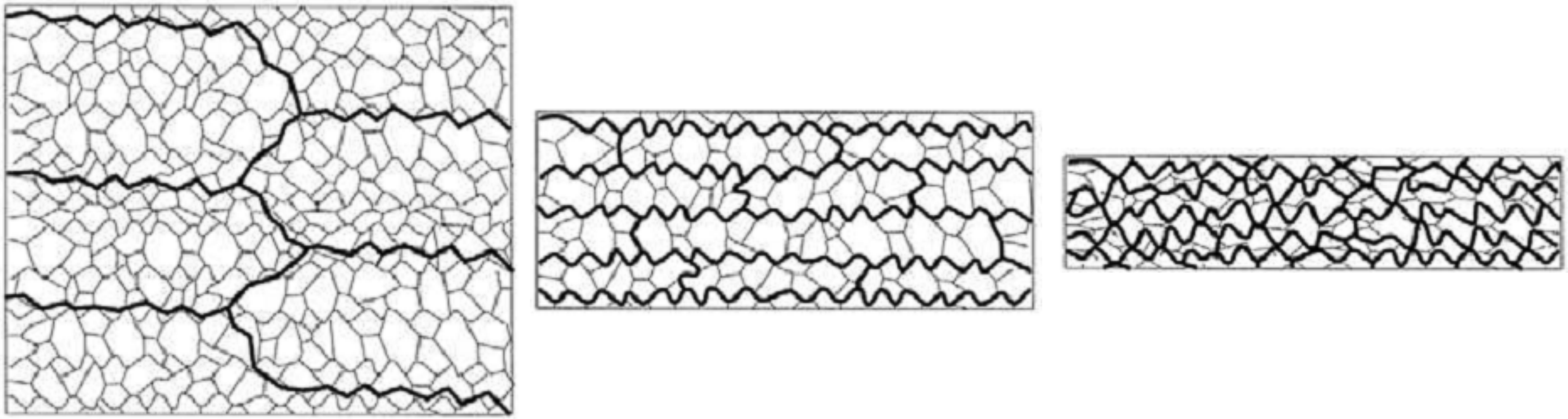




Geometric DRX (GDRX)



1) The serrated HAGBs (thick lines) become closer.

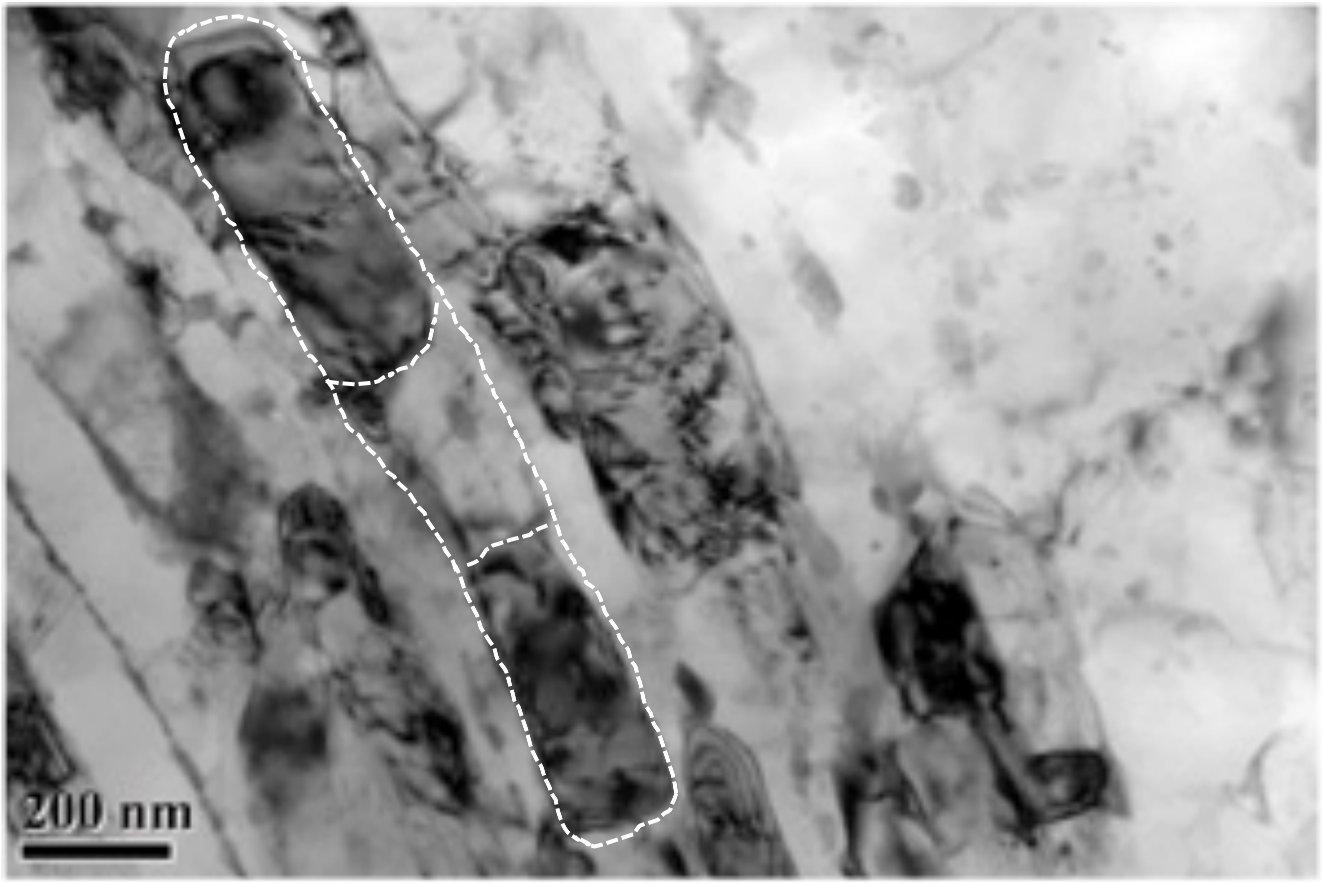
2) The subgrain size remains approximately constant.

3) Eventually the high angle boundaries (HAGBs) impinge.

F.J. Humphreys, M. Hatherly, Recrystallization and Related Annealing Phenomena. Oxford, 2004.

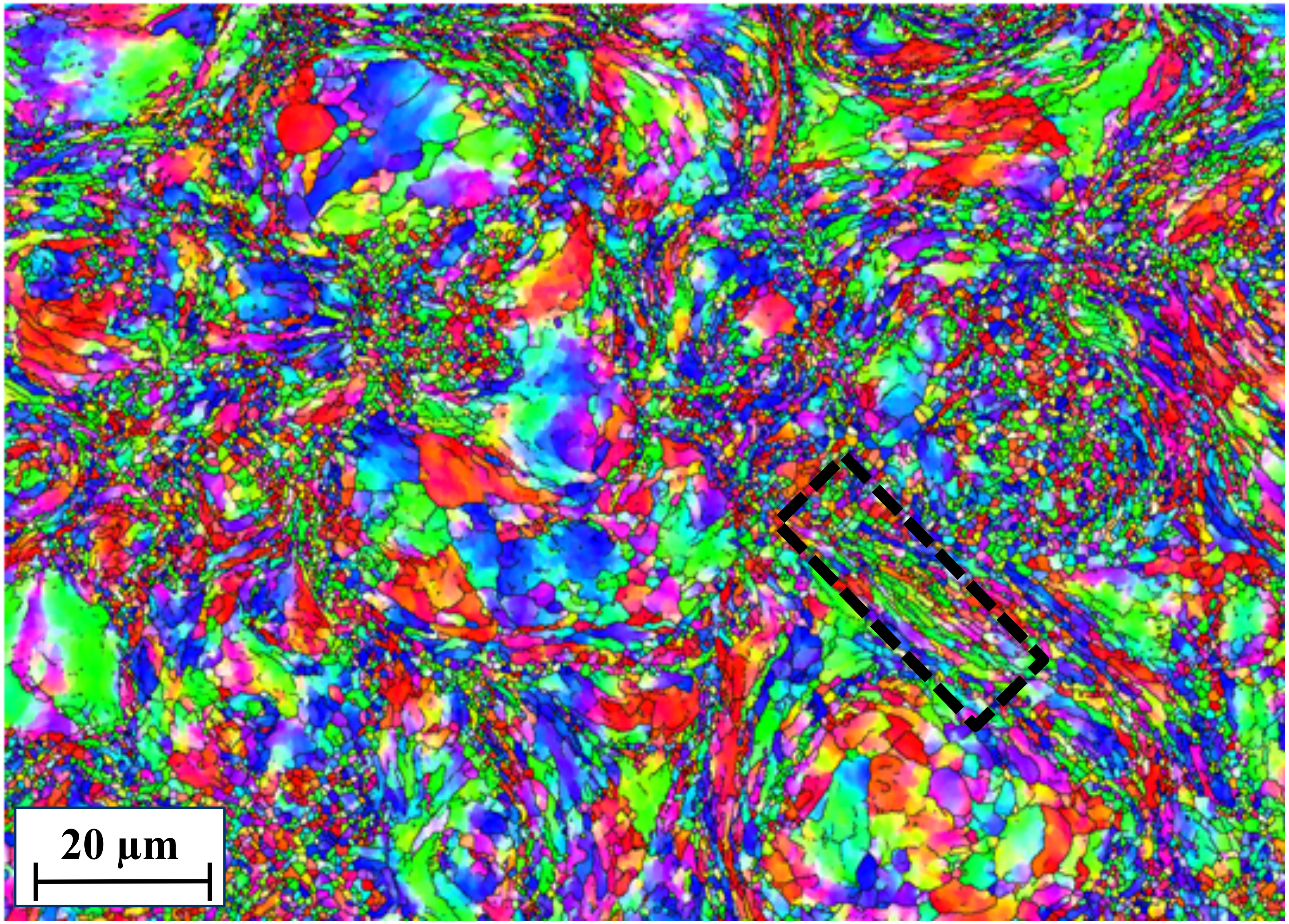
Microstructure of the CSP layer

✓ Some elongated grain structures at particle/particle boundaries



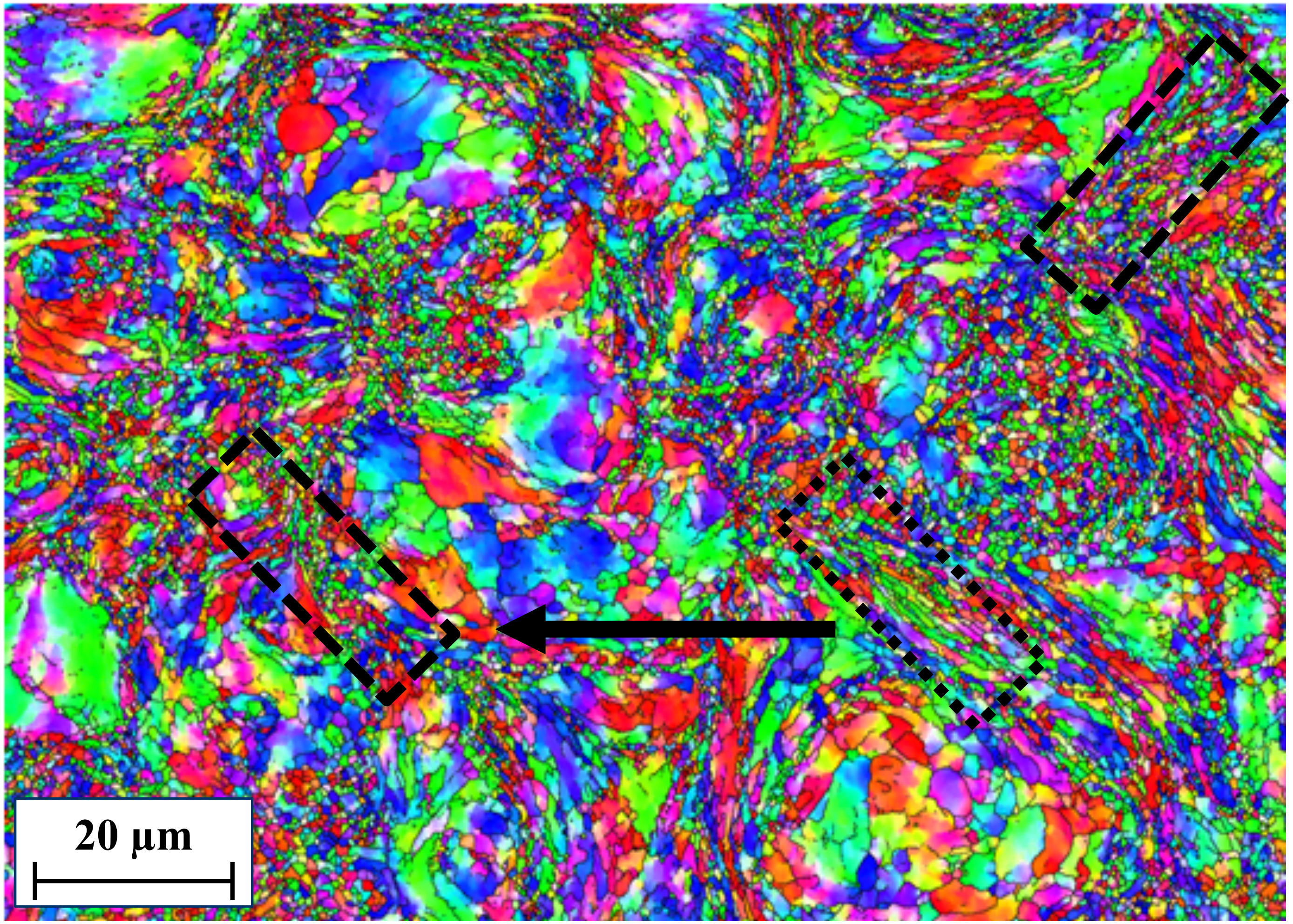
Microstructure of the CSP layer

✓ Subdivided into pancaked subgrains by some LAGBs



Main formation mechanisms

✓ **Geometric DRX (GDRX) is one of the main mechanisms.**



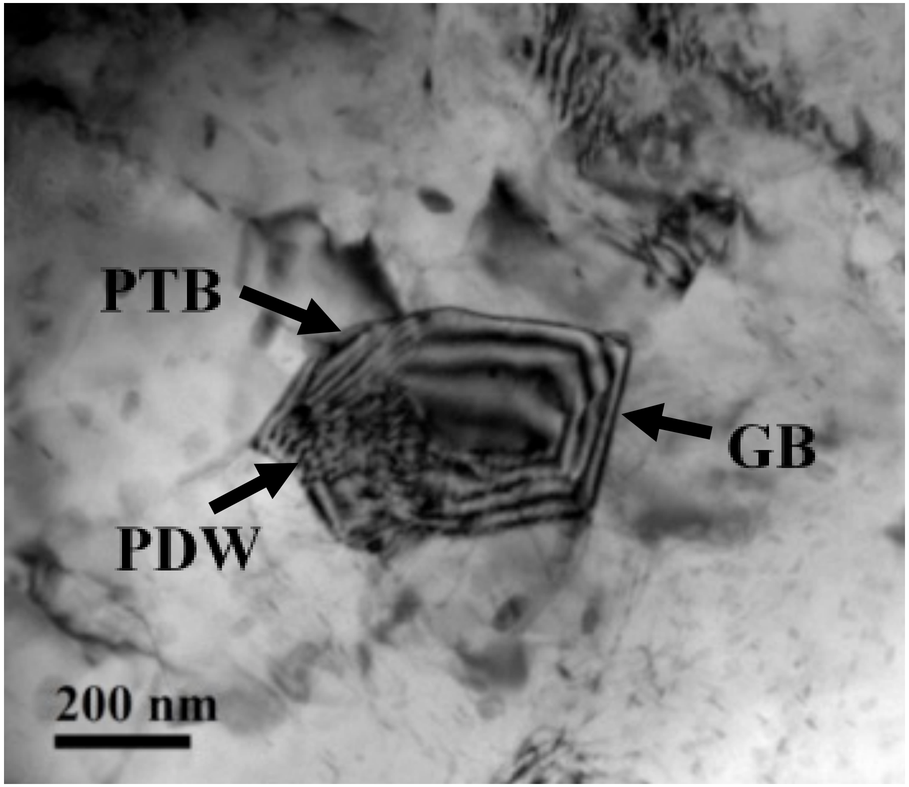
Structure Evolution of GBs

✓ 3 types of boundaries:

1- Polygonized dislocation wall (<1º)

2- Partially transf. boundary (1-5º)

3- Grain boundary (>15º)

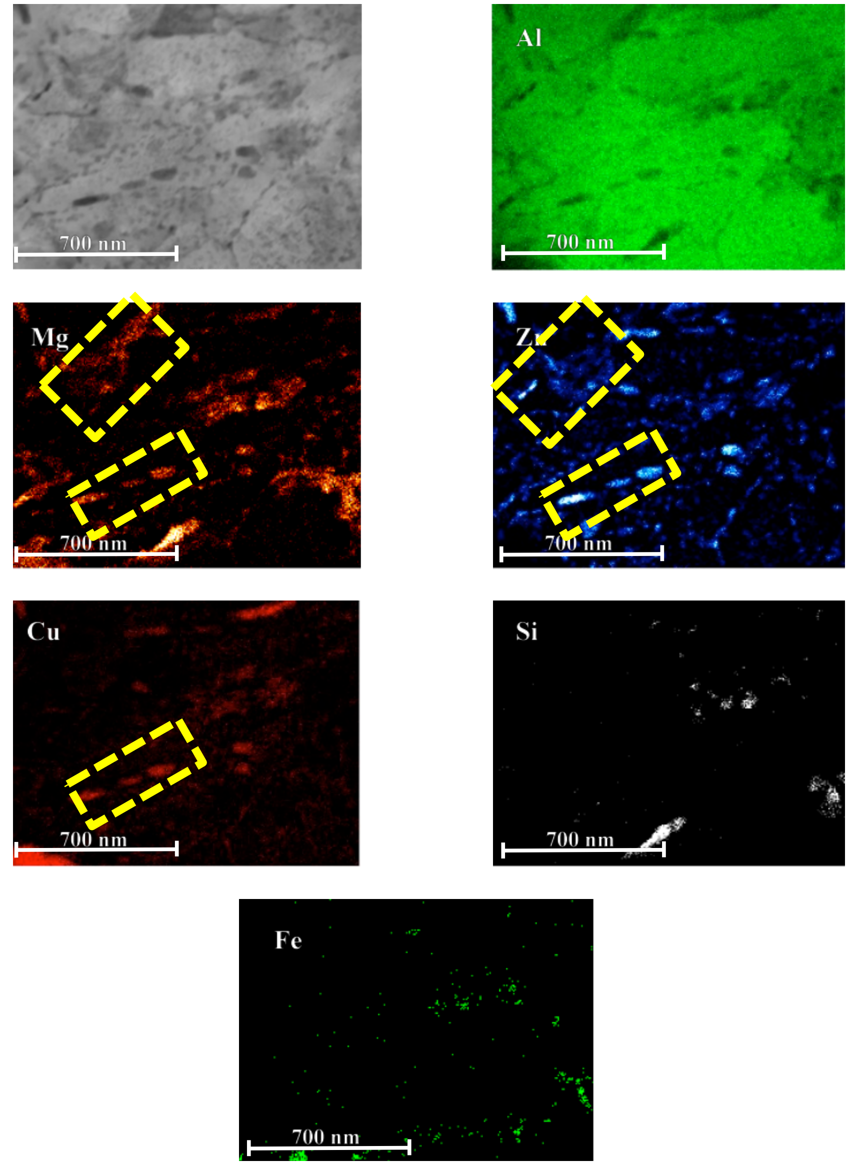


This is evidence of continuous DRX (CDRX). Therefore, a combination CDRX and GDRX is responsible for formation of UFG structures in the microstructure.

Microstructural evolution of 7075 Al gas atomized powder and high-pressure cold sprayed deposition, Surface and Coatings Technology 251 (2014) 254-263.

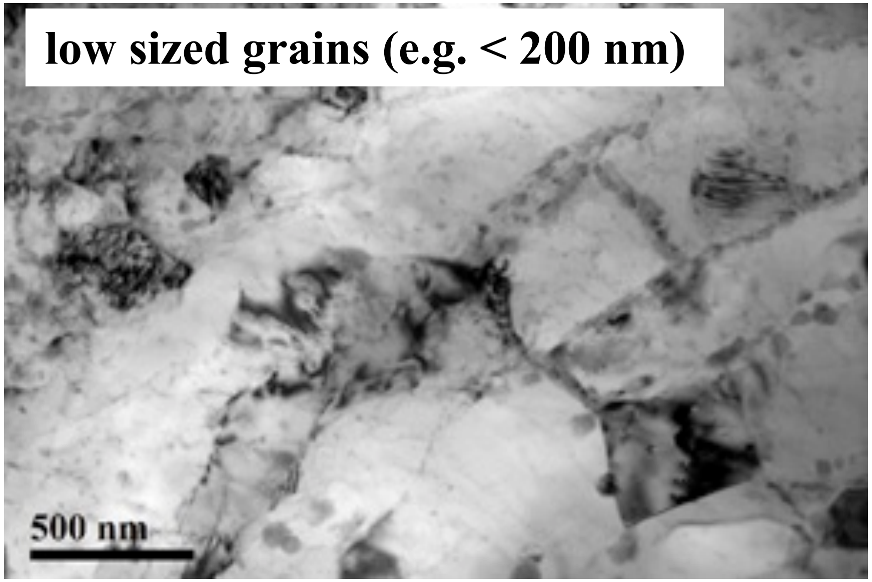
Precipitates in the Deposition

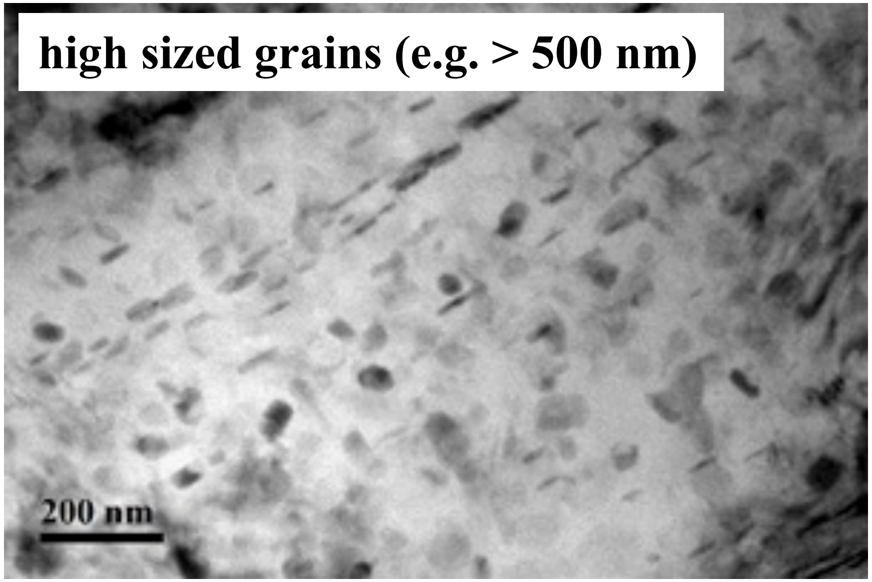
- ✓ A variety of different precipitates composed of various amounts of Al, Zn, Mg, Cu and Si
- ✓ η ($MgZn_2$) and $Mg(Zn,Cu,Al)_2$



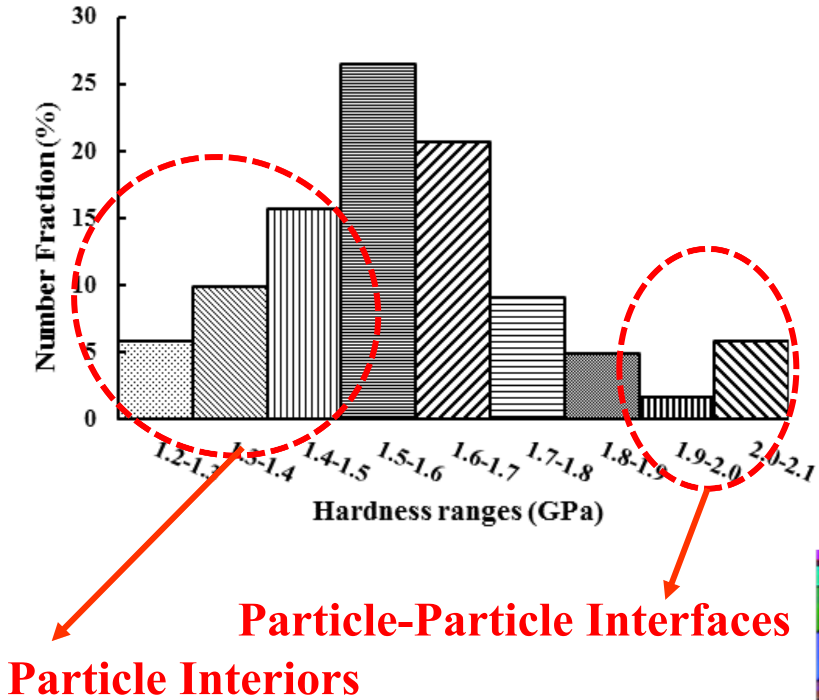
Precipitates in the Deposition

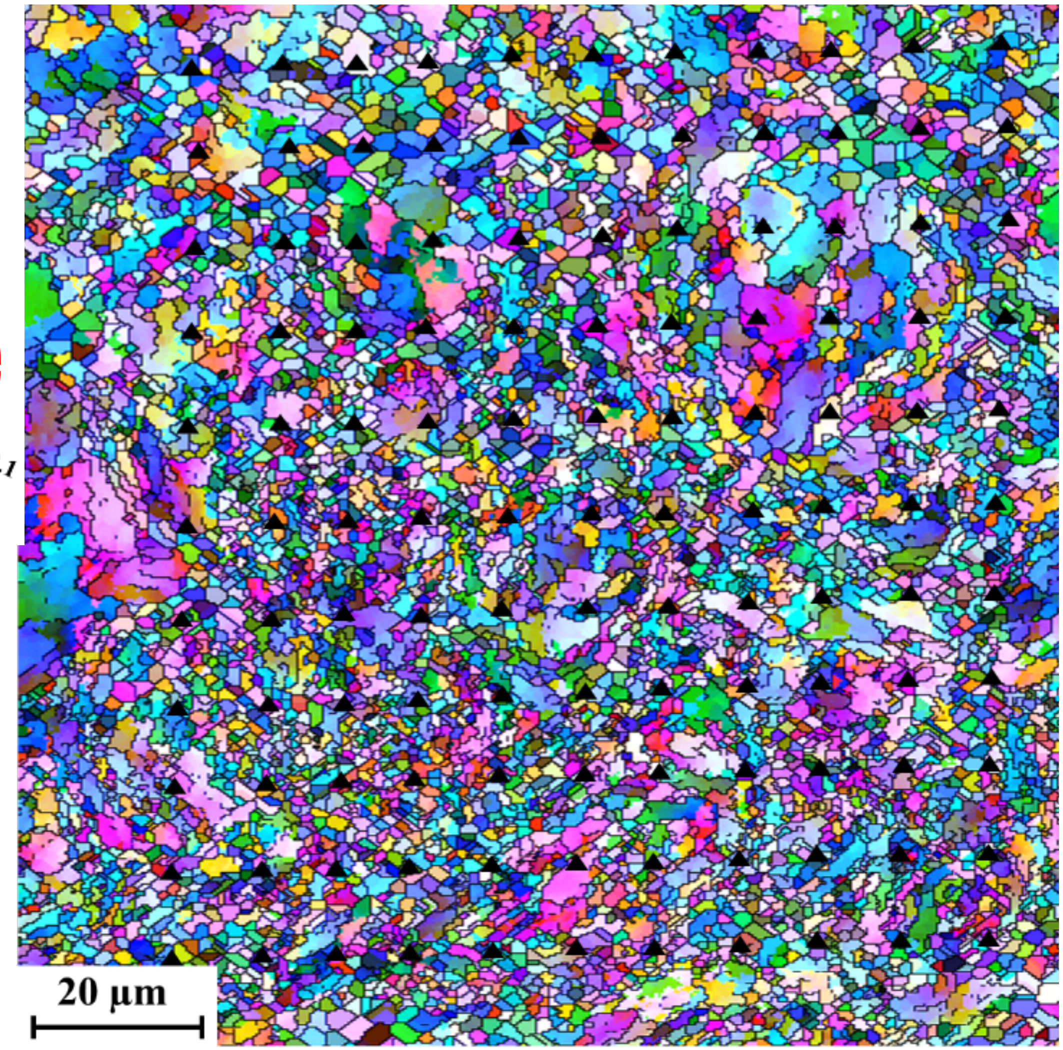
- ✓ Low sized grains: located primarily at grain boundaries
- ✓ High sized grains: distributed throughout the grain interior
- ✓ Due to volume for dislocations interaction and more nucleation sites





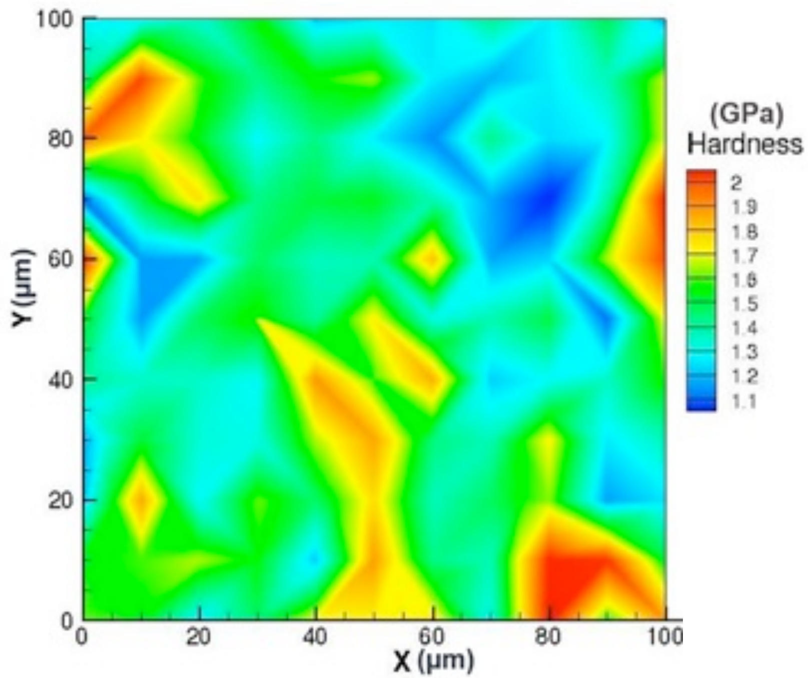
Nanohardness results



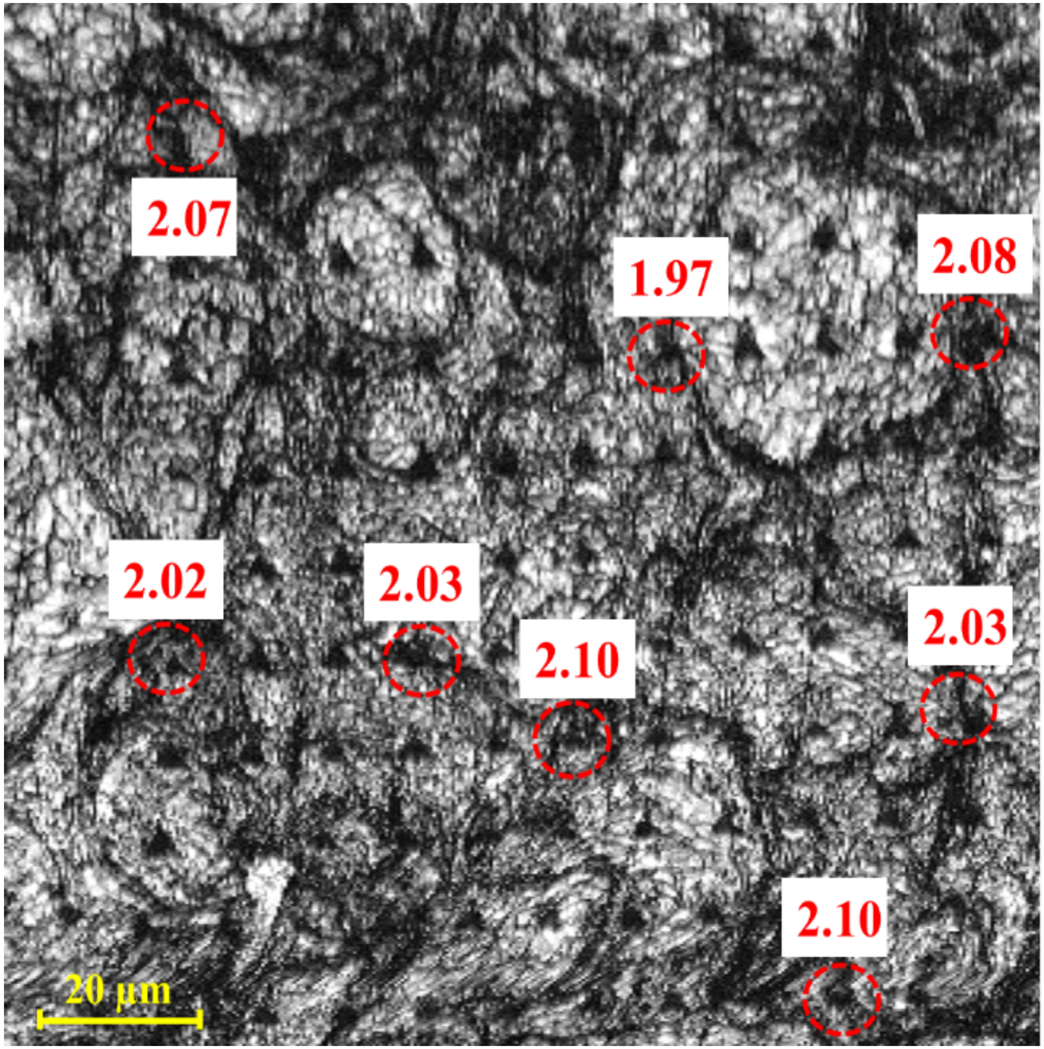


M. R. Rokni, C. A. Widener, G. A. Crawford, M. K. West, An Investigation into Microstructure & Mechanical Properties of cold sprayed 7075 Al deposition, Materials Science & Engineering A 625 (2015) 19-27.

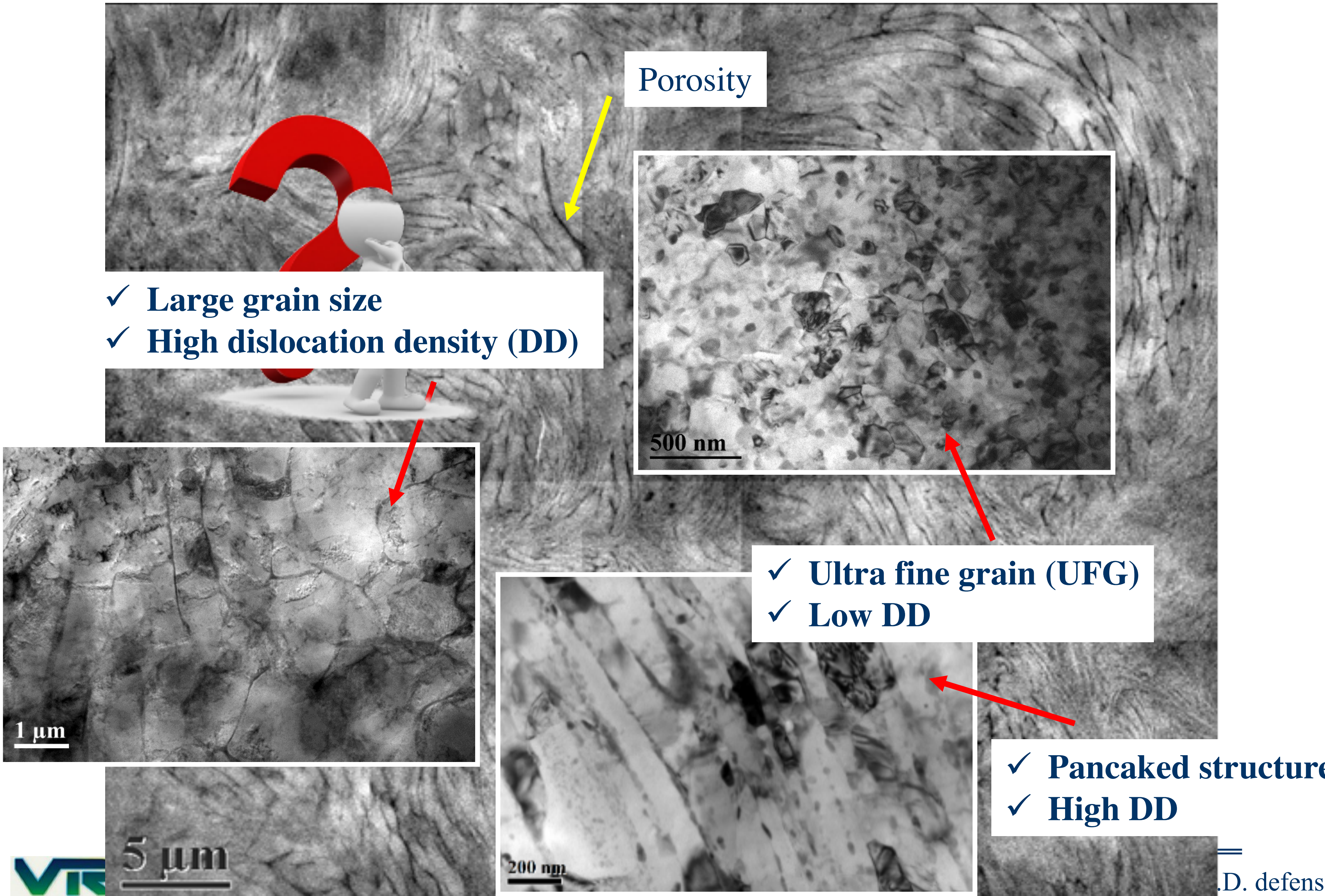
Nanohardness results



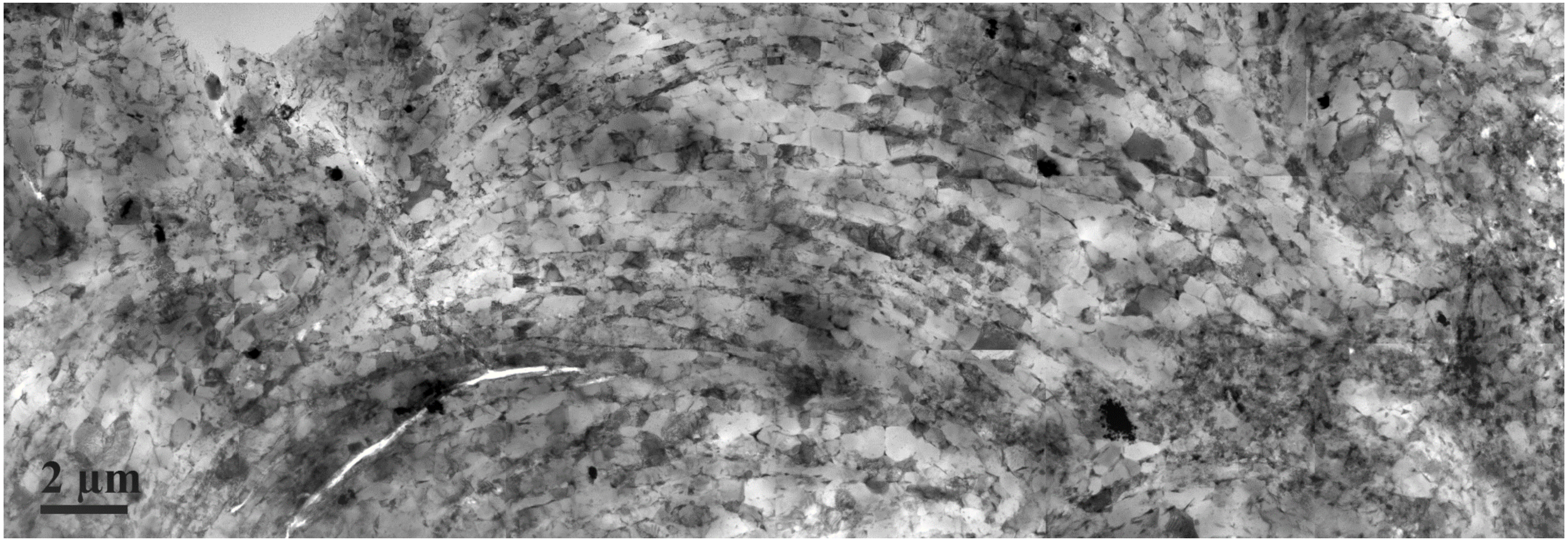
Material	CSP 7075	
Hardness (GPa)	particle interior	particle interfaces
	1.53±0.30	2.01±0.09



Details of Microstructure (TEM)



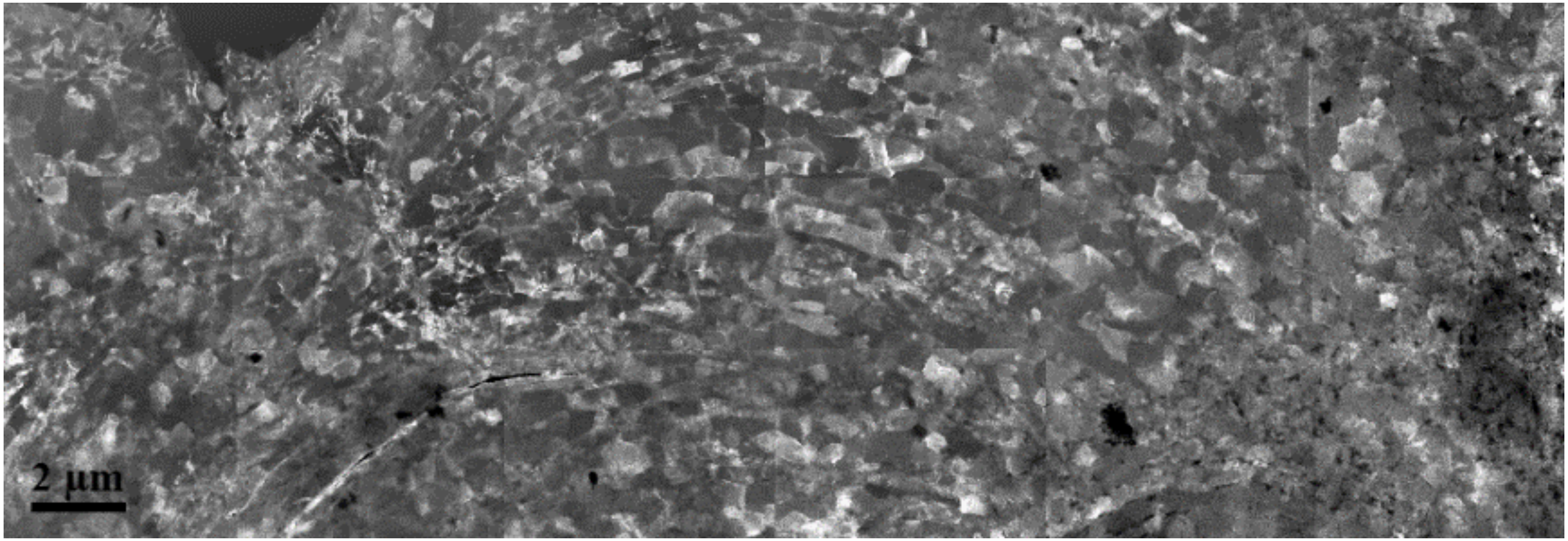
Final microstructure After Annealing



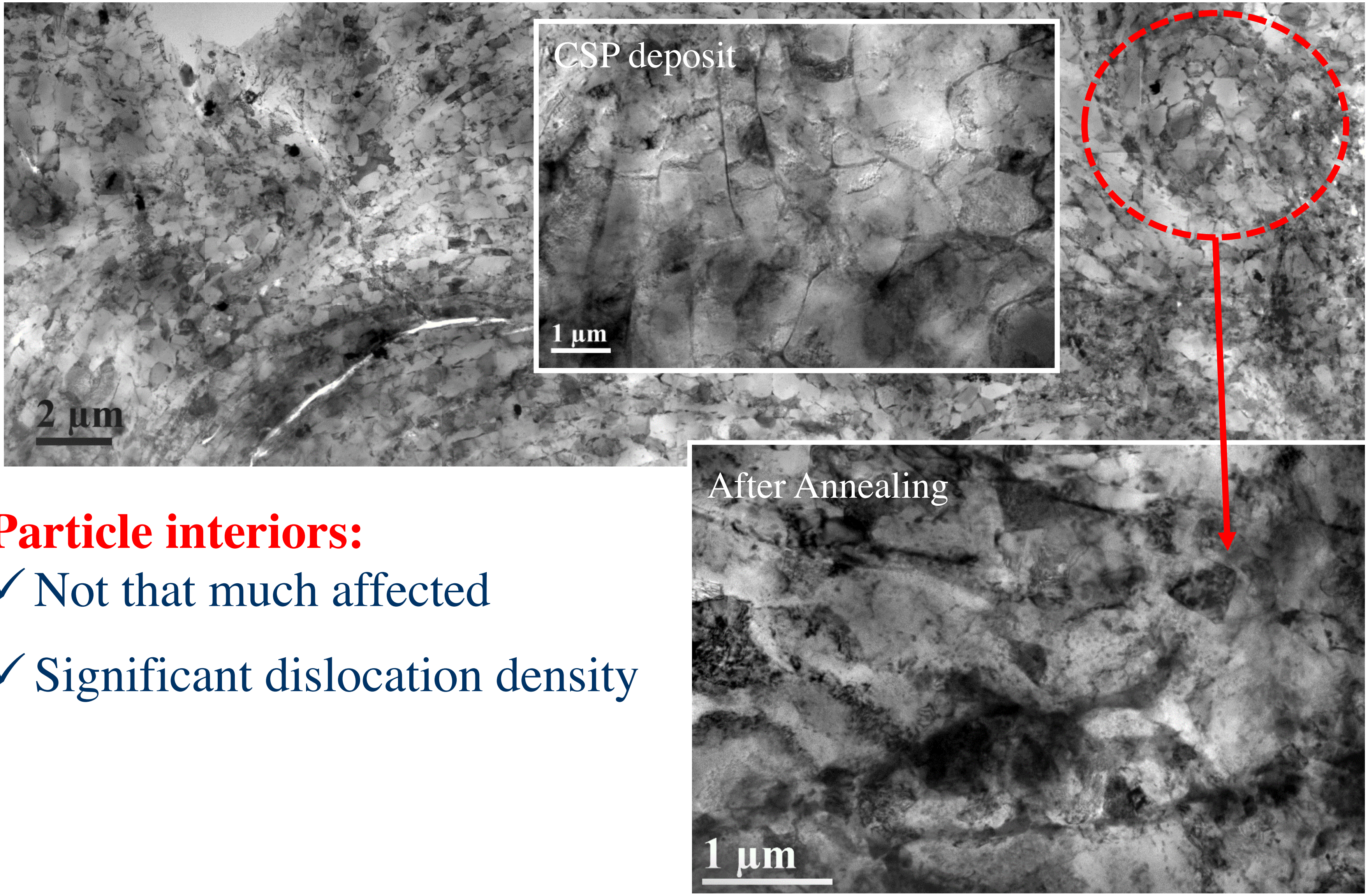
✓ Equiaxed grains with well-defined, straight GBs, unlike the inhomogeneous microstructure observed in the as-deposited coating.

Final microstructure After Annealing

- ✓ Many of the grains are free of dislocations
- ✓ Some grains still contain a medium dislocation density
- ✓ Residual stress relief through the annealing process.



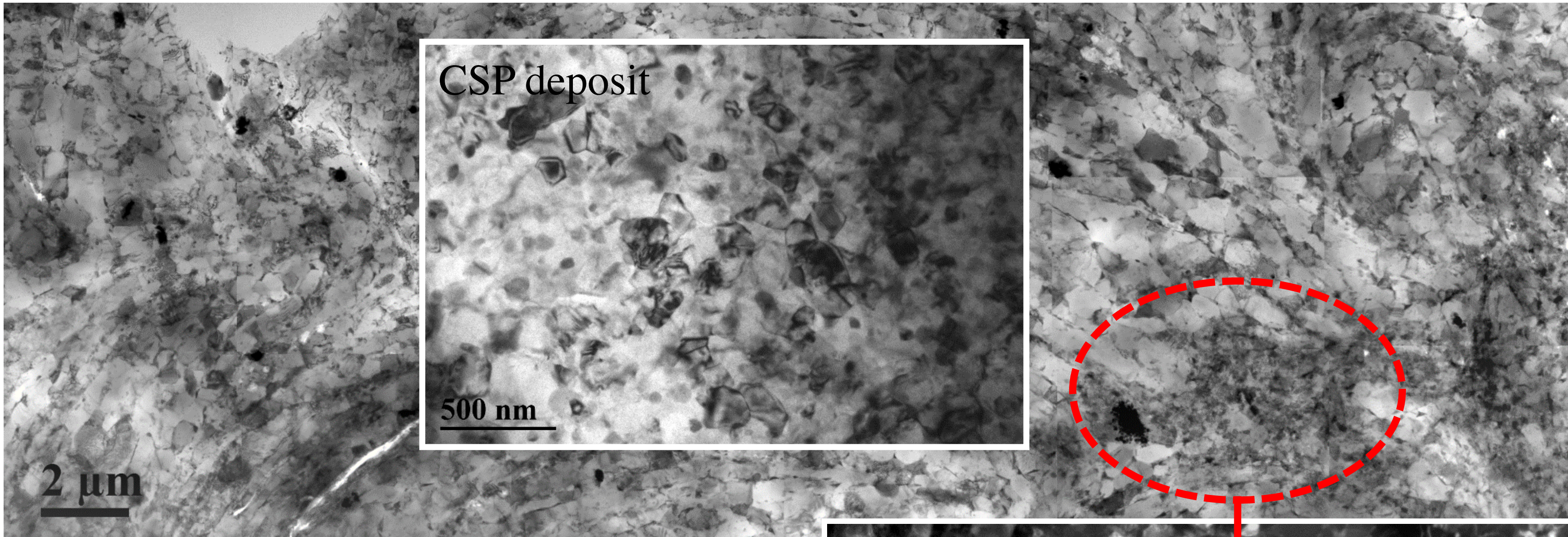
Final microstructure (Particle Interiors)

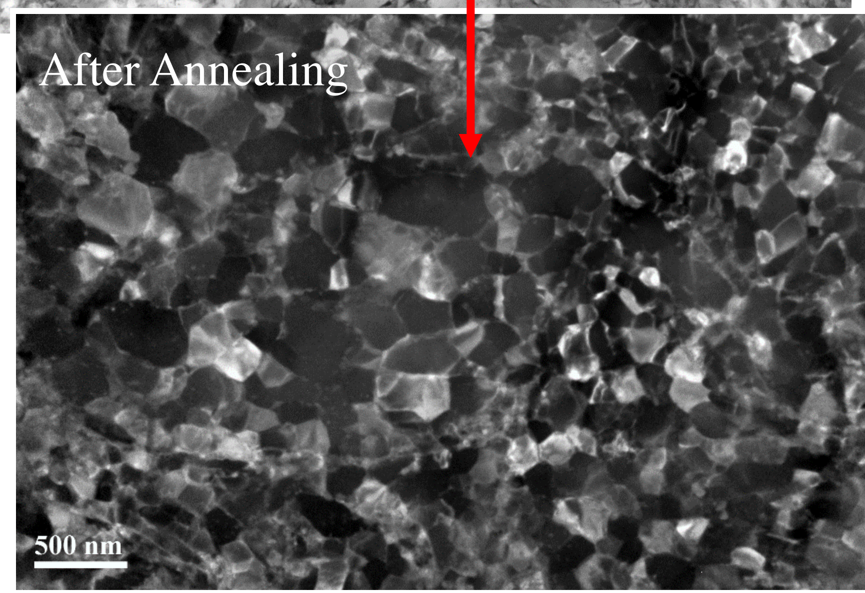


Particle interiors:

- ✓ Not that much affected
- ✓ Significant dislocation density

Final microstructure (UFG structure)

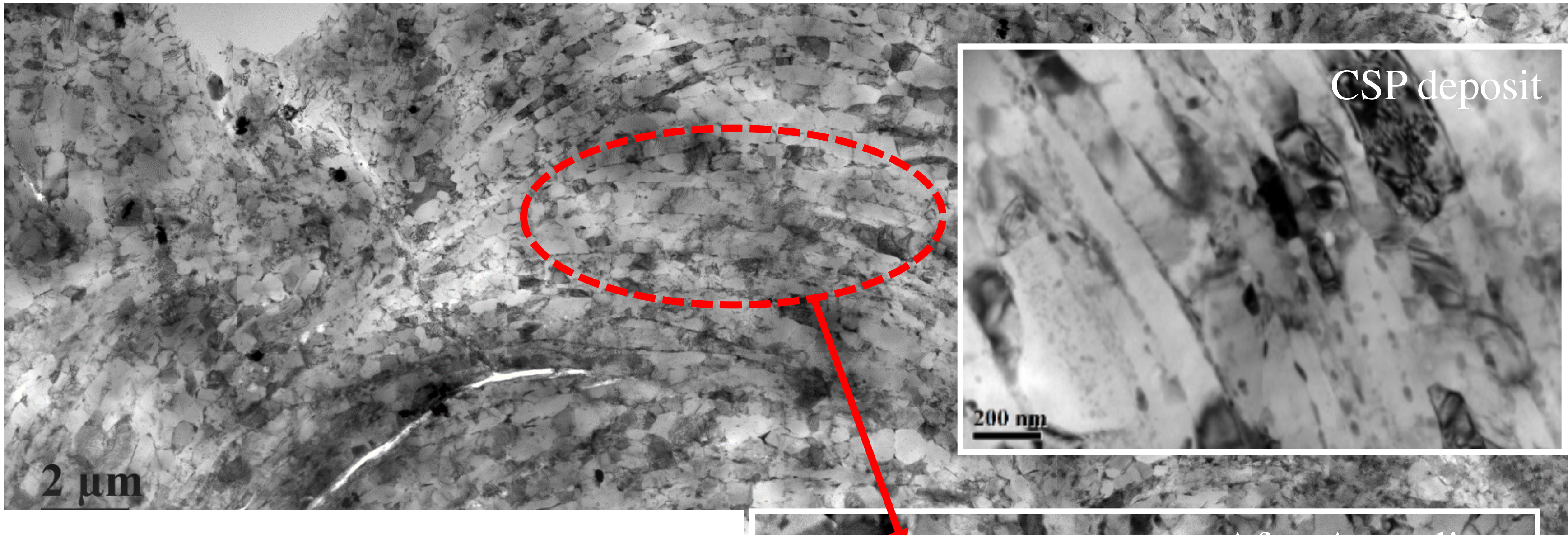




UFG structures:

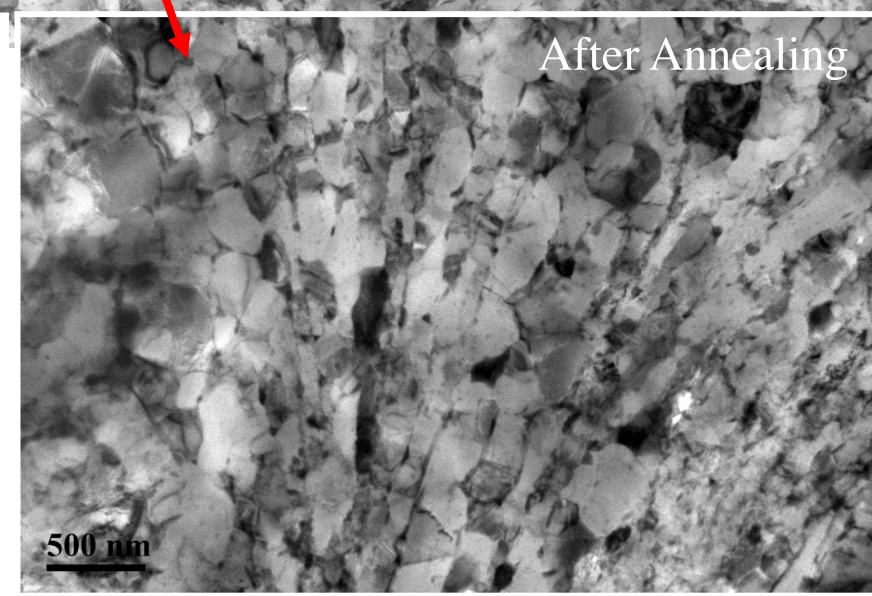
- ✓ Relatively unaffected by the annealing process.
- ✓ Some reduction in overall dislocation density, improved GB definition.

Final Microstructure (Pancaked Str.)

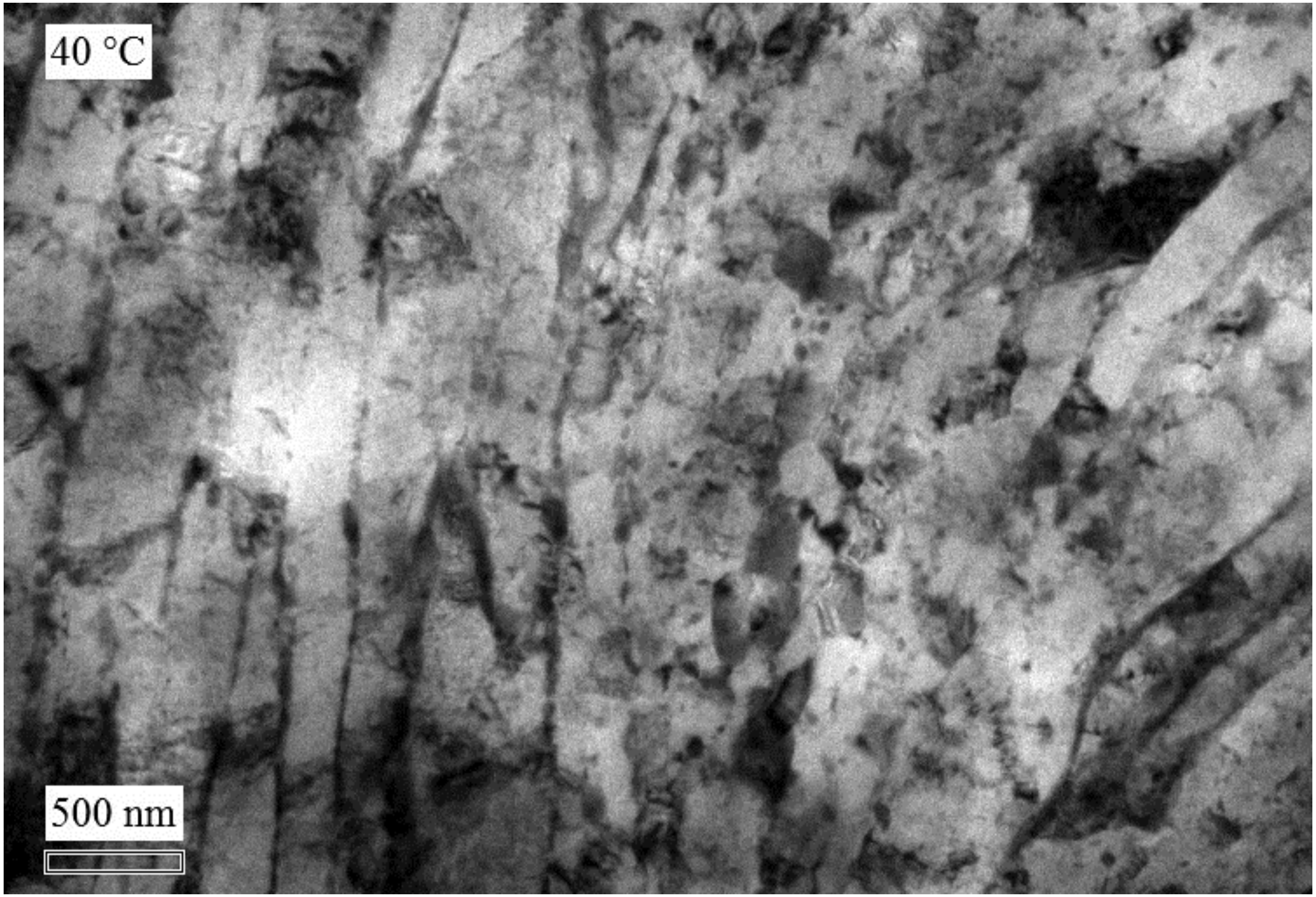


Pancaked structures:

- ✓ Significant recrystallization during the annealing
- ✓ Developing an UFG structure as opposed to pancaked structures

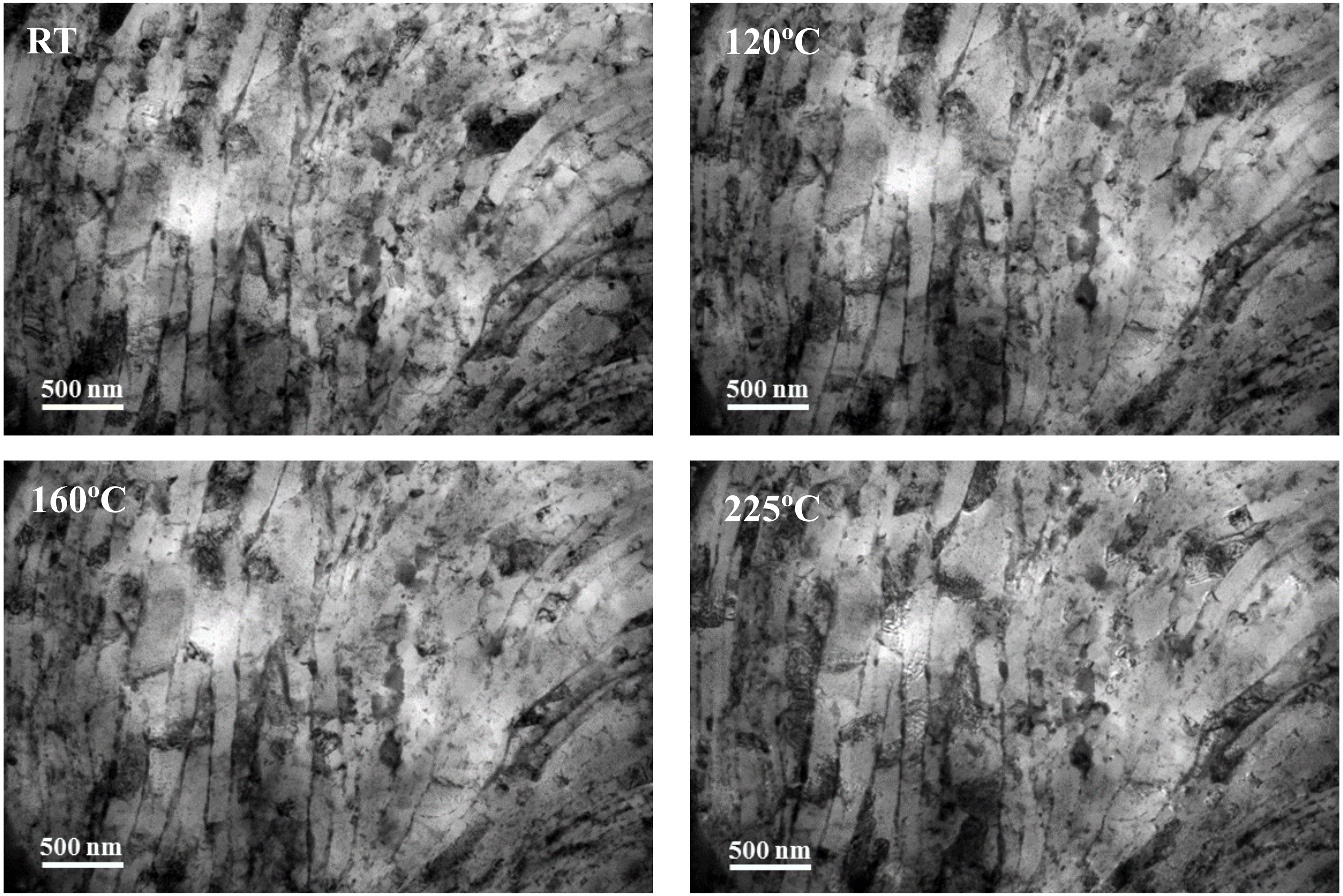


Non-isothermal In-situ TEM Heating



1) Dislocation movement
2) Annihilation of dislocations and substructures
3) Dislocation free microstructure/ RX
4) precipitation and Growth of precipitates

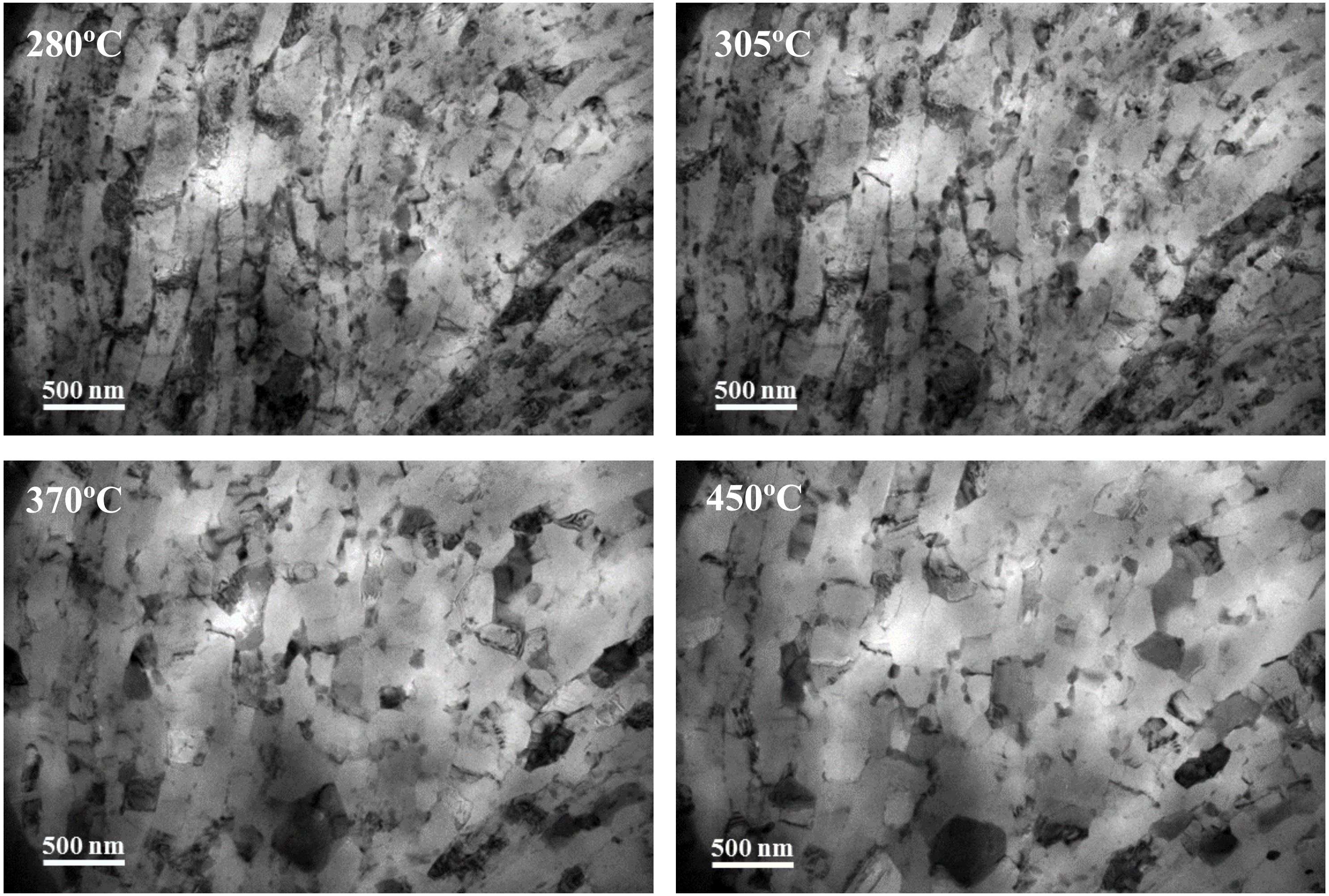
Microstructure During Annealing



From RT to 225˚C:

- ✓**Dislocation movement**
- ✓**Retention of pancaked structure.**

Microstructure During Annealing



At 280˚C: collapse of lamellar structure

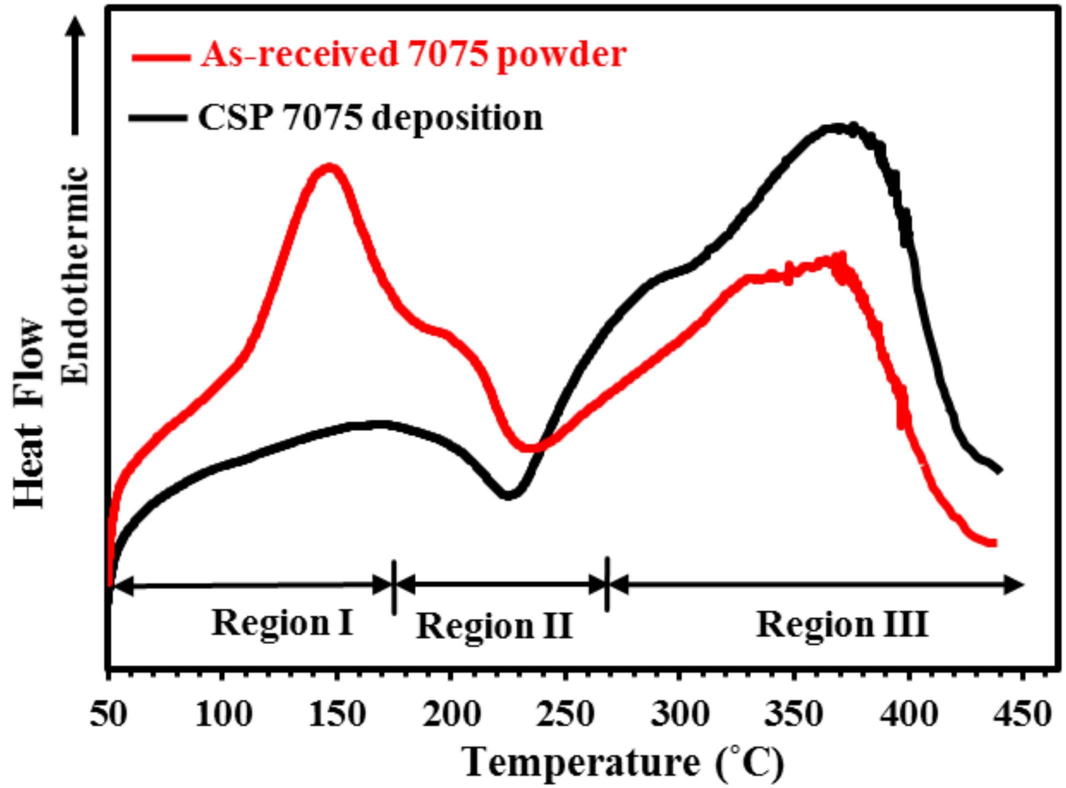
At 370˚C: some evidence of recrystallization

At 450˚C: *Limited grain growth because of grain boundary precipitation*

Differential Scanning Calorimetry

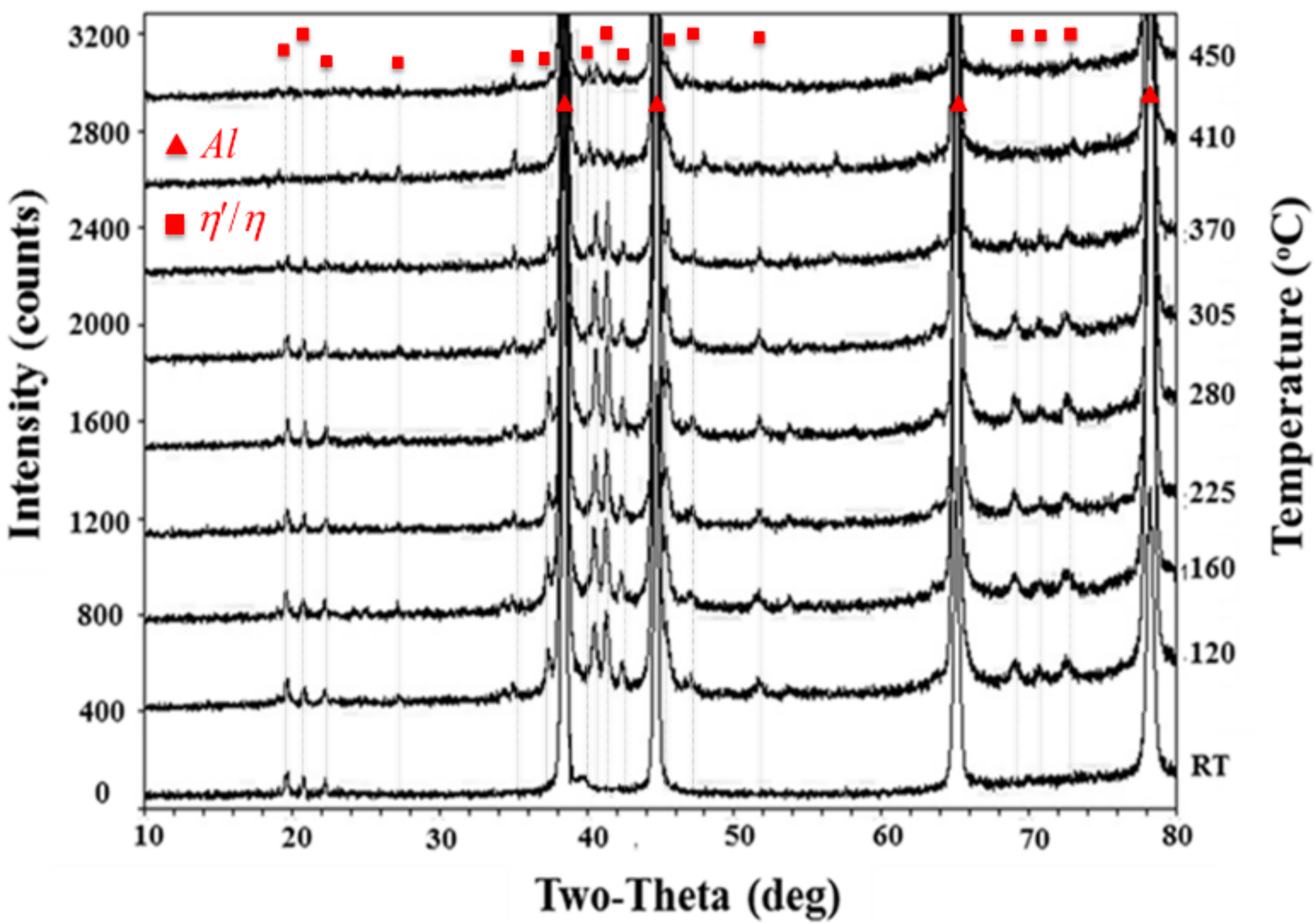
- ✓ An endothermic peak at region I ➔ Dissolution of G-P zones
- ✓ Exothermic peak at region II ➔ Formation of η'/η
- ✓ Endothermic peak at region III ➔ Dissolution of all phases

✓ No strengthening precipitates after about 370ºC in the microstructure

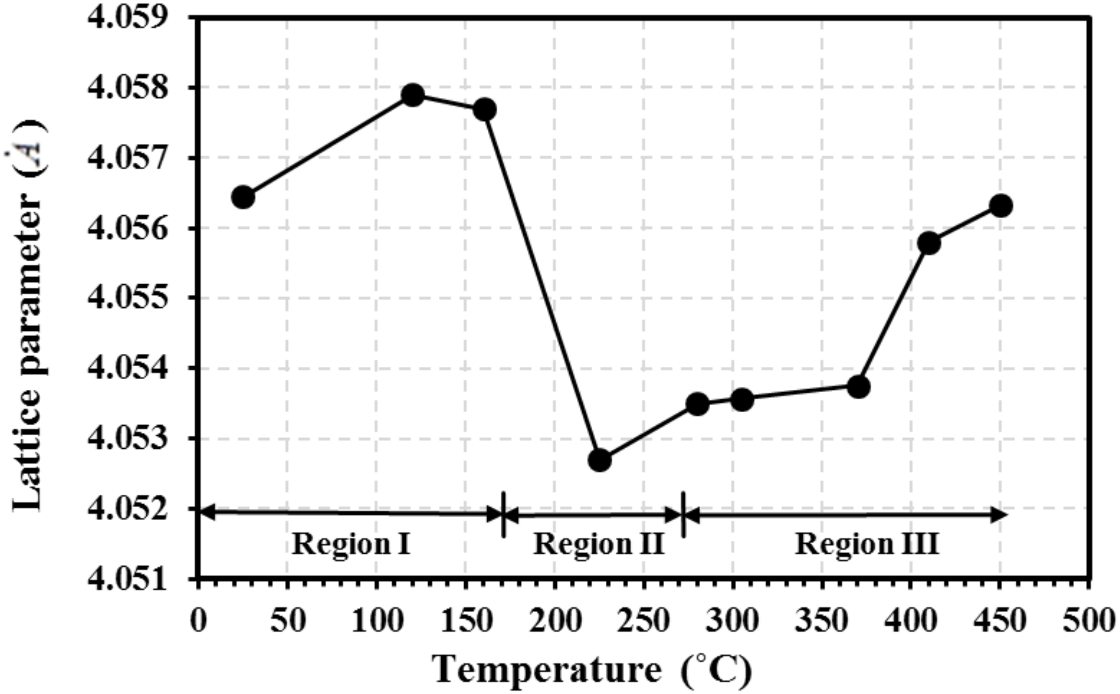


M.R. Rokni, C.A. Widener, G.A. Crawford, Microstructure and Mechanical Properties of cold sprayed 7075 Deposition during Non-isothermal Annealing, submitted to Acta Materialia.

X-ray Diffraction Profile



Microstructure Analysis

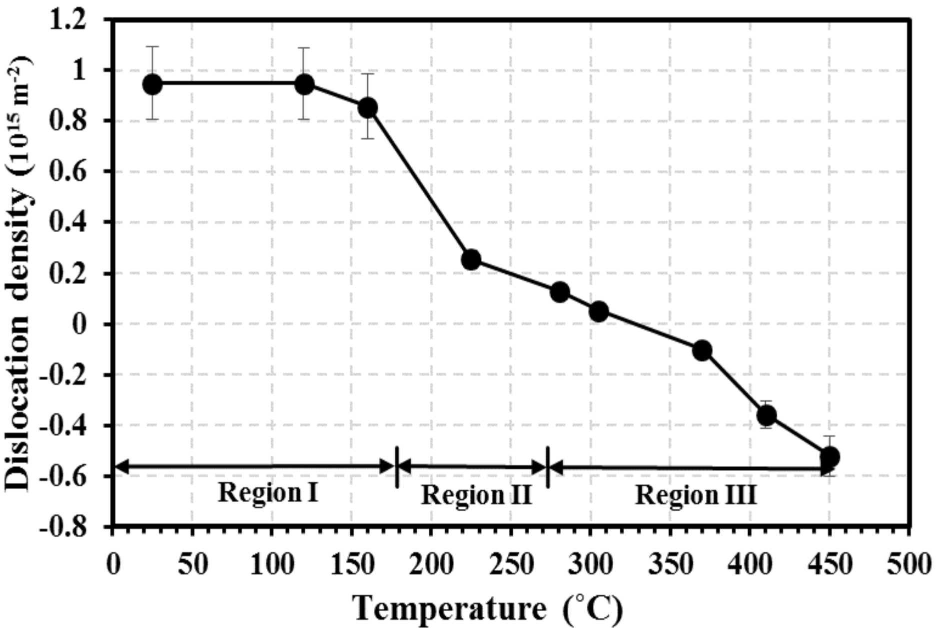


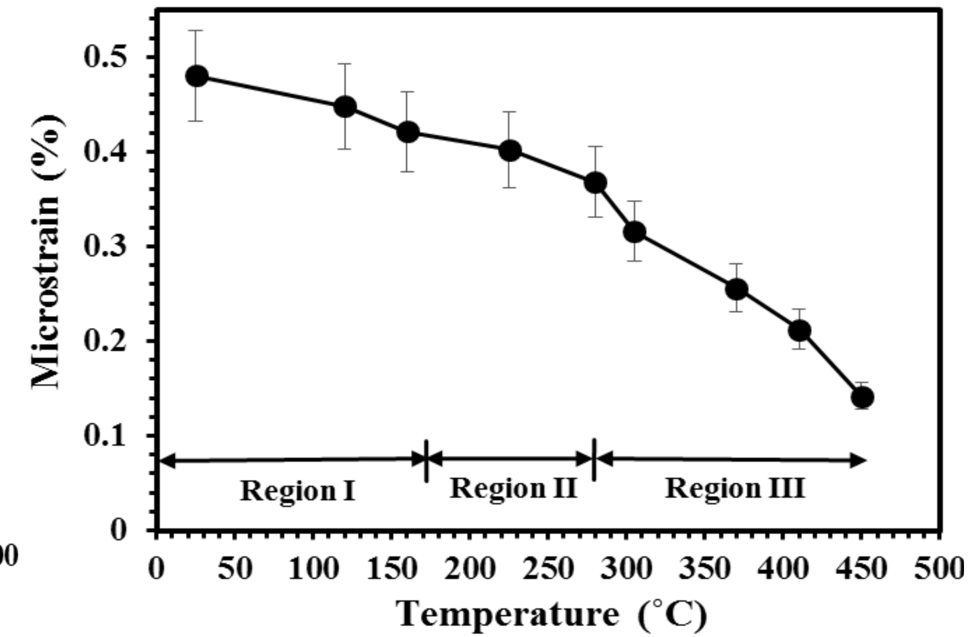
(1) A noticeable increase in LP between due to the dissolution of G-P zones

(2) significant decrease in LP due to the formation of η'/η precipitates

(3) An increase in LP due to dissolution of all phases in the matrix.

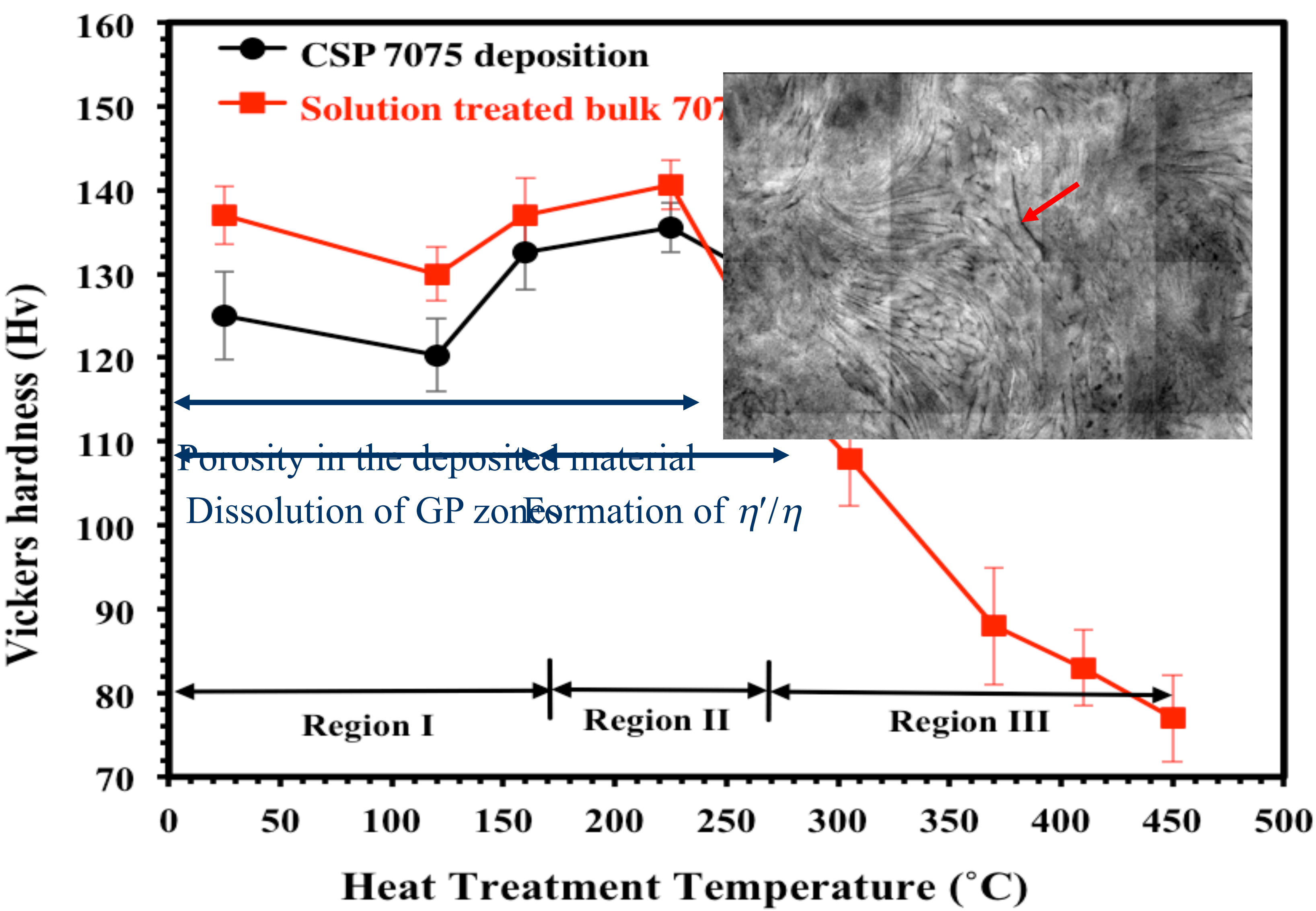
Microstructure Analysis



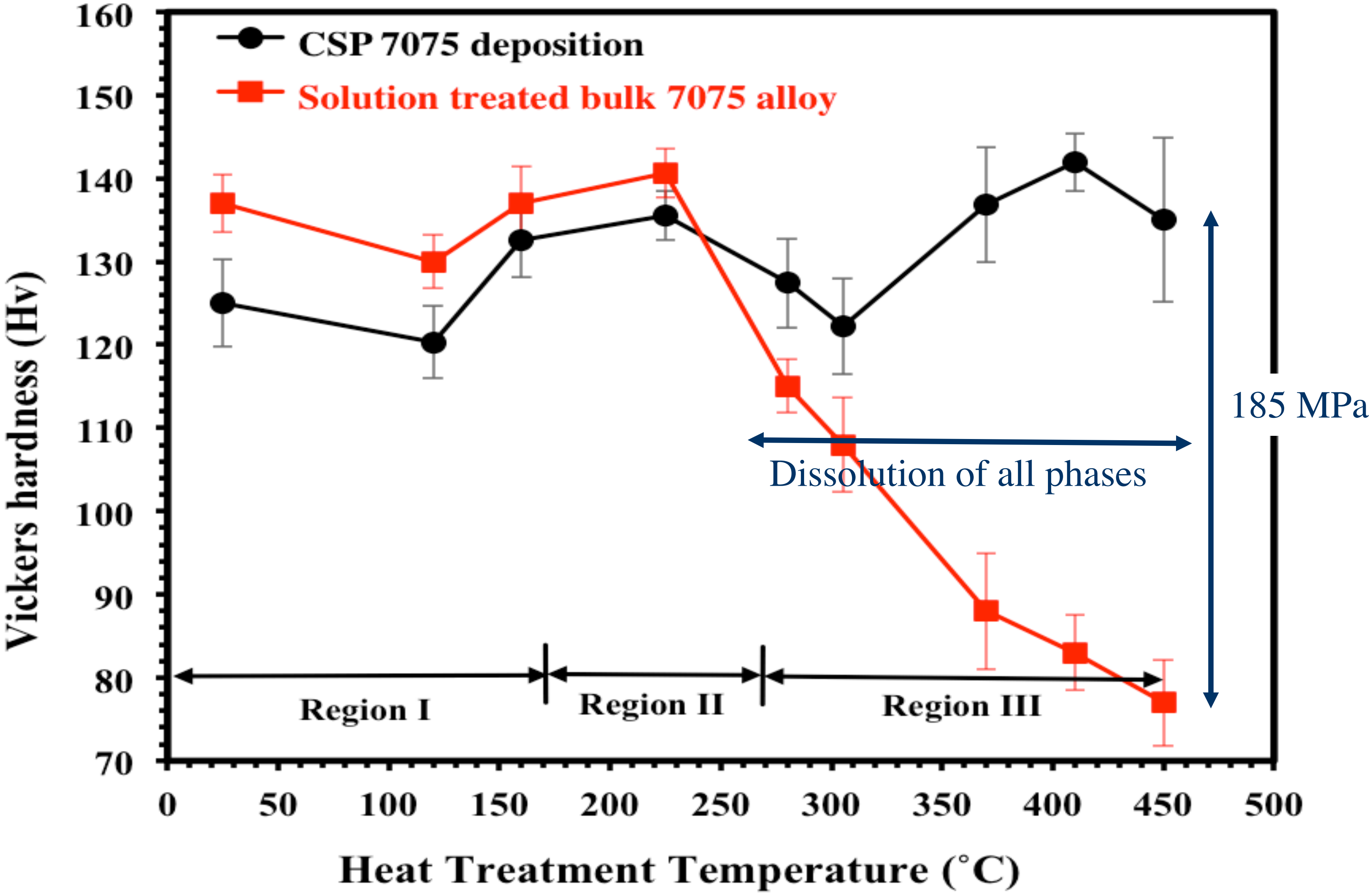


- ✓ Continual decrease in the DD and microstrain by temperature
  - ➢ due to more driving force by annealing
- ✓ Dislocation rearrangement in low temperatures
- ✓ More mobility of dislocations towards higher temperatures

Mechanical Behavior



Mechanical Behavior



Mechanical Behavior

There are four strengthening mechanisms that may play a role in the microhardness evolution of this material during non-isothermal annealing:

- ✓ Grain boundary strengthening
- ✓ Solid solution strengthening
- ✓ Precipitate strengthening
- ✓ Strain hardening.

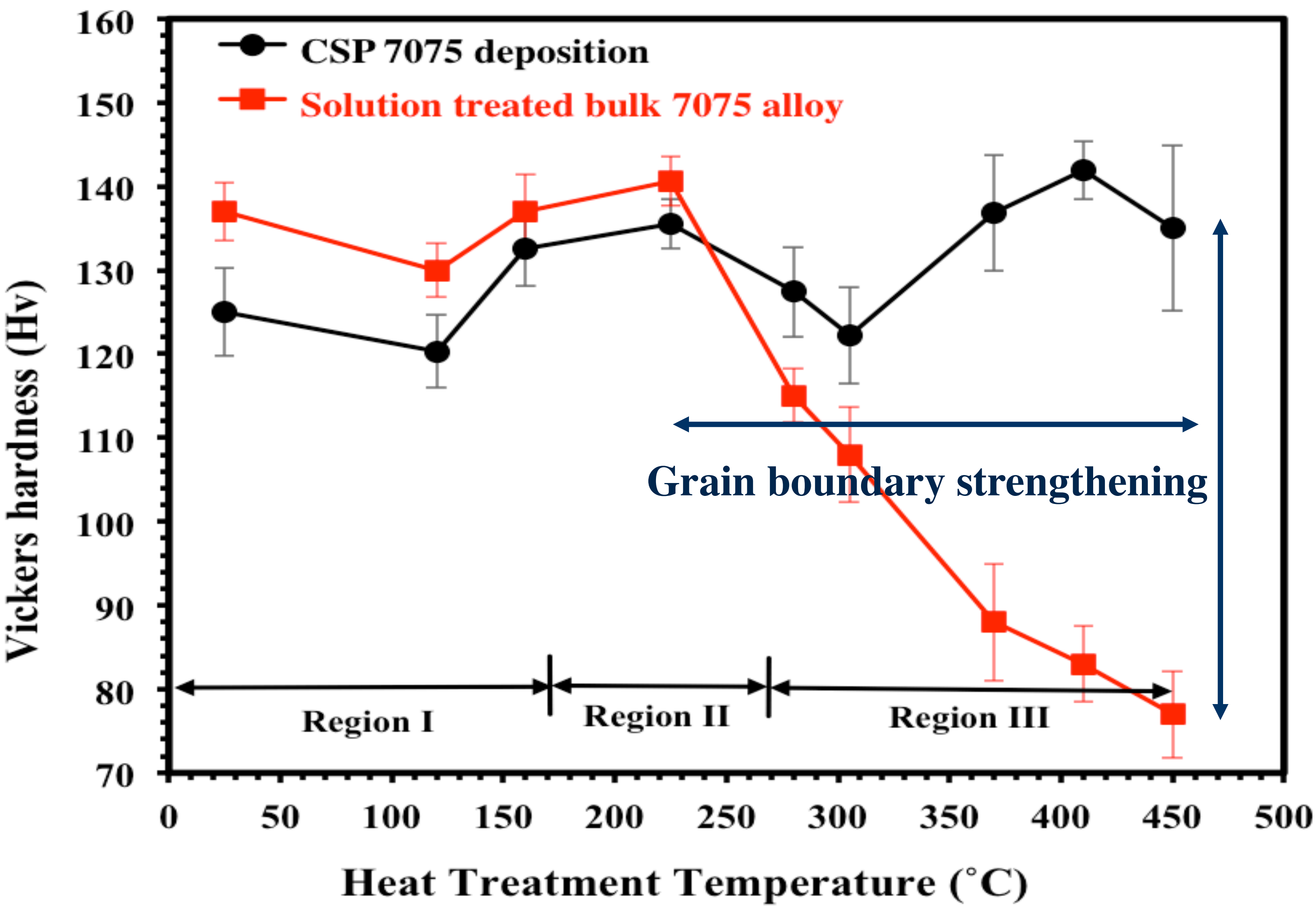
Hall–Petch equation

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}}$$

CSP 7075 deposition **~500 nm**

Solution treated bulk 7075 alloy **100.6 ± 23.2 μm** → **160 MPa**

Mechanical Behavior



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Effect of Heat Treatment

✓ Micro-tensile samples were heat treated to different commercial HTs of 7075 Al alloy:

1- As-deposited (AD)

2- T6

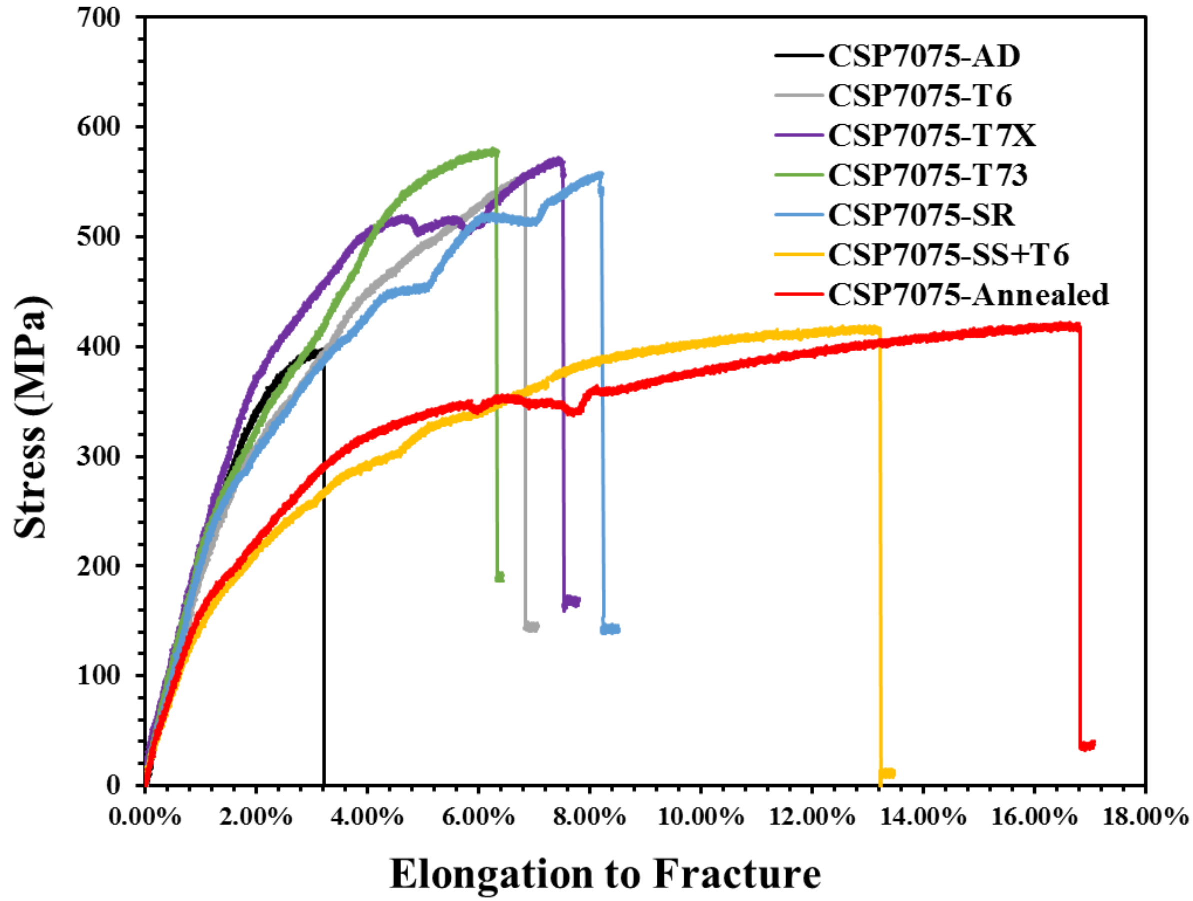
3- 7X

4- T73

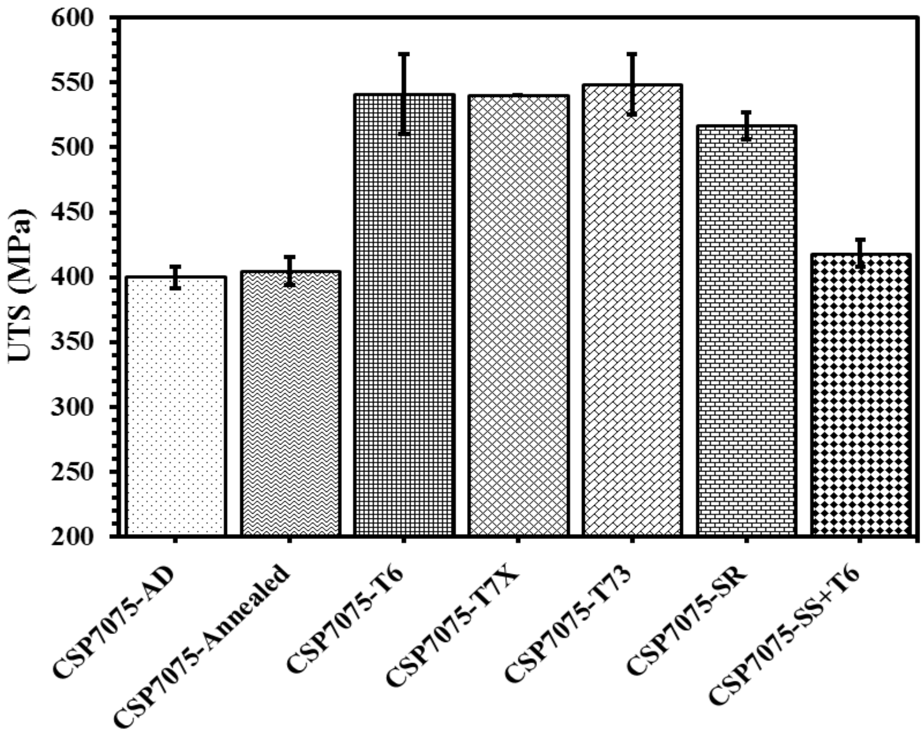
5- Stress relief (SR)

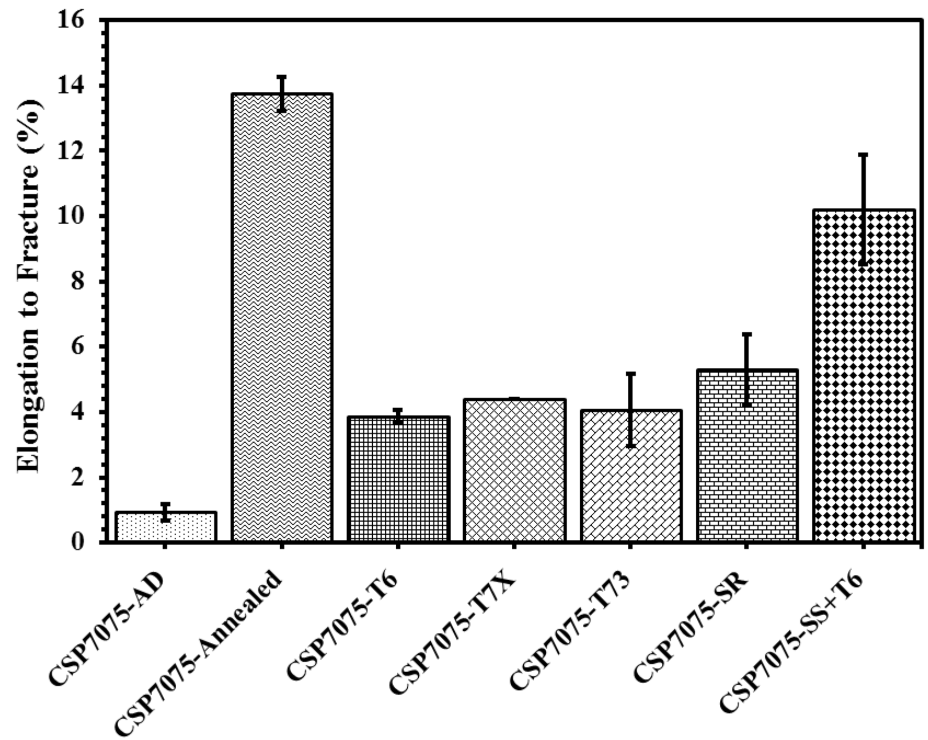
6- Annealing

7- Solution treating + T6 (SS+T6)



Mechanical Properties of HTed Samples





✓ The lowest UTS and ductility for the AD condition.

Due to significant imposed cold plastic deformation

Largely decreased strain-hardening ability

Conclusion/Summary

1. This research detailed the microstructure evolution and mechanical properties of CSP 7075 Al deposition.
2. Significant solute element segregation was observed at grain boundaries of particles.
3. HPCS resulted in the formation of a high quality deposition with limited porosity and inter-particle voids.
4. The deposition was characterized by two distinct regions: particle interfaces (UFG & pancaked) and particle interiors.
5. The formation of the UFG structure was attributed to a combination of CDRX and GDRX.

Conclusion/Summary

6. Microstructural stability of the distinct regions was evaluated during non-isothermal annealing from RT to 450ºC.
7. The pancaked regions experience recrystallization and are converted to UFG structures during the annealing.
8. The microhardness stays high even towards the end of annealing due to grain boundary strengthening mechanism.
9. The mechanical properties of the deposition can be substantially improved with low and high temperature heat treatments
10. This happens due to precipitation of strengthening phases and metallurgical bonding.

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- My family of brothers and sisters here in Rapid City
- My mother, father, brother, and sisters

Contribution to Science

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2. **M.R. Rokni**, C.A. Widener, V.R. Champagne, Microstructural Evolution of 6061 Al Gas-Atomized Powder and High-Pressure Cold-Sprayed Deposition, J thermal spray technology 23 (2014) 514-524.
3. **M.R. Rokni**, C.A. Widener, S.P. Ahrenkiel, B.K. Jasthi, V.R. Champagne, Annealing Behavior of 6061 Al Deposited by High Pressure CS, Surface Engineering 30 ,(2014) 361-368.
4. **M.R. Rokni**, C.A. Widener, V.R. Champagne, Microstructural stability of ultrafine grained cold sprayed 6061 Al alloy, Applied Surface Science 290 (2014) 482-489.
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6. **M.R. Rokni**, C.A. Widener, G.A. Crawford, Microstructural evolution of 7075 Al gas atomized powder and high-pressure cold sprayed deposition, Surf. Coat. Technol. 251 (2014) 254-263.
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8. **M. R. Rokni**, C. A. Widener, G. A. Crawford, M. K. West, An Investigation into Microstructure & Mechanical Properties of cold sprayed 7075 Al deposition, Mater. Sci. Eng. A 625 (2015) 19-27.
9. **M.R. Rokni**, C.A. Widener, G.A. Crawford, Microstructure and Mechanical Properties of cold sprayed 7075 Deposition during Non-isothermal Annealing, submitted to Acta Materialia.
10. **M.R. Rokni**, C.A. Widener, G.A. Crawford, The effects of post CS heat treatments on microstructure & Mechanical properties of 7075 Al depositions, Under preparation.

Success is achieved and maintained by those who develop a burning desire for their high goals and then keep on trying with positive mental attitude.

"Success through a positive mental attitude"

By Napoleon Hill

Thank you for your attention!

Contact:

Dr. Christian Widener
Director of Advance Materials Processing Center (AMP)
South Dakota School of Mines and Technology
Ph. 605-394 6924
Email: christian.widener@sdsmt.edu

Reza Rokni, Ph.D. Candidate
Advance Materials Processing Center (AMP)
South Dakota School of Mines and Technology
Ph. 605-877 6902
Email: reza.rokni@mines.sdsmt.edu

Army Research Laboratories

SOUTH DAKOTA SCHOOL OF MINES & TECHNOLOGY