



*Interim Report I*

**5 Inch Thick 2219-T87 Plate Low Ductility Investigation**

– Material analysis on broken tensile samples

EM31 Investigation Team

EM31/NASA-Marshall Space Flight Center

November 22, 2021

# 2219-T87 Plate Low Ductility Investigation Support

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**Investigation planning & coordination:** Po Chen, Ben Rupp

**Metallography & optical microscopy:** Matt Medders, Sam Mcleroy, Po Chen

**SEM/fractography:** Ellen Rabenberg

**DSC:** Ching-Hua Su, Jeff Quick, Po Chen

**Electrical conductivity (EC):** Ching-Hua Su

1. DSC is intended to compare incipient melting temperature and mass % of Al-Cu eutectic phase
2. EC is an indirect way to compare precipitate size and distribution.
  - In general, the material that has larger precipitate size and wider spacing between precipitate particles has higher EC

# Samples Received from EM21 for Low Ductility Investigation

## Broken tensile samples

Specimen ID, Test temperature	Fracture elongation (%)
9492854 720-4, RT	6.67%
9492854 720-2, LN2	7.23%
9492854 720-23, LH2	2.55%
930071 722C-3, RT	1.70%
930071 722C-5, LN2	1.00%
930071 722C-10, LH2	1.15%
930082 723C-2, RT	1.33%
930082 723C-6, LN2	1.25%
930082 723C-10, LH2	1.10%



## 5" plate block

1. One from lot 9492854
2. Two from lot 930071
3. Two from lot 930082

## Broken Tensile Samples

1. Two from lot 930081  
(ductility  $\approx$  0.8% in LN<sub>2</sub>)

**To be investigated**

**Investigation on-going**

# Sample Preparation and Analysis Status for Broken Tensile Samples

Specimen ID	% EL	Priority	Sample preparation		Metallurgical Analysis		DSC/EC Analyses	
			Met samples	DSC/EC samples	Optical microscopy	SEM	DSC	EC
9492854 720-4, RT	6.67%	2	√	√	√			
9492854 720-2, LN2	7.23%	1	√	√	√	√	√	√
9492854 720-23, LH2	2.55%	1	√	√	√			
930071 722C-3, RT	1.70%	2						
930071 722C-5, LN2	1.00%	1	√	√	√	√	√	√
930071 722C-10, LH2	1.15%	2						
930082 723C-2, RT	1.33%	2						
930082 723C-6, LN2	1.25%	1	√	√	√	√	√	√
930082 723C-10, LH2	1.10%	2						

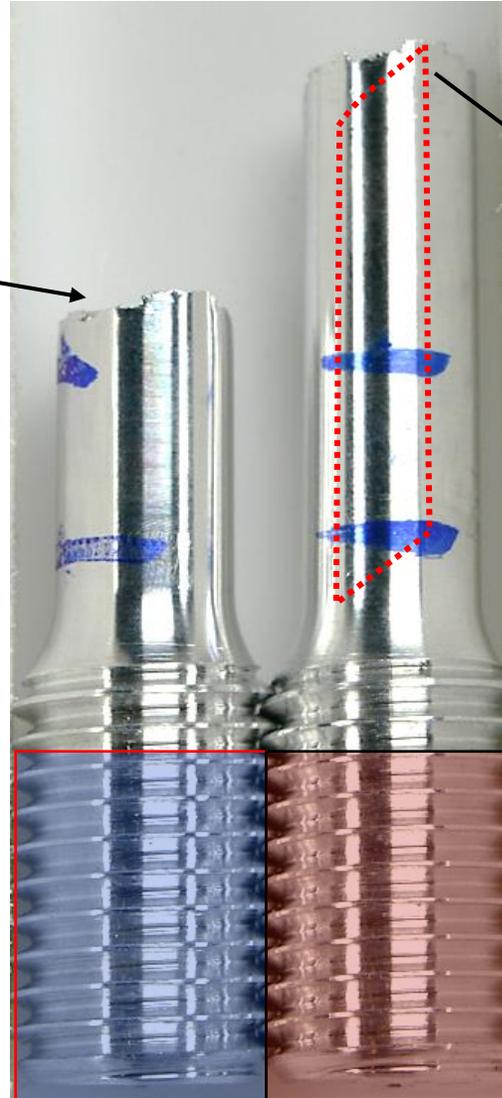
√: denotes completion of sample preparation or metallurgical analysis

1. Divide the broken tensile samples into two groups, priority 1 & 2
2. Focus on 3 LN<sub>2</sub> samples from 3 different lots to determine root cause of low ductility in a timely fashion

# Material Analysis Plan for Thick 2219 Plate Samples



SEM analysis on fracture surface to determine fracture mode, amount and distribution of Cu-rich precipitates

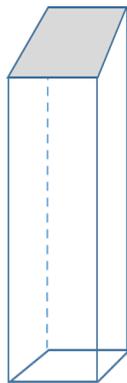


Section sample along rolling direction. Analyze grain structure and GB precipitation near the fracture surface using OM and SEM. The focus is on size & distribution of coarse GB Cu-rich precipitates



DSC sample (4.5 mm d. x 10 mm)

EC sample (2.5 mm x 2.5 mm x 15 mm)



Extract DSC & EC samples from the threaded section



Extract met samples from the threaded section to determine "L" direction



# Key Findings after the Material Analysis on Three Broken Tensile Samples

		Key Findings						
Specimen ID, Test T	Ductility	Grain size	% Rx	Remnant dendrite cells	Fracture mode	Density of coarse Cu-rich $\theta$ phase ( $Al_2Cu$ ) at GB	Incipient melting temperature ( DSC)	EC
9492854 720-2, LN2	7.23%	smaller	higher	fewer	TG	low	$\approx 548$ °C	lower
930071 722C-5, LN2	1.00%	larger	lower	more	IG	high	$\approx 548$ °C	higher
930082 723C-6, LN2	1.25%	larger	lower	more	IG	high	$\approx 548$ °C	higher

Rx: Recrystallization; TG: Transgranular fracture; IG: Intergranular fracture  
 GB: Grain boundary; EC: Electricity conductivity, DSC: Differential Scanning Calorimetry

## **Detrimental effects of coarse Cu-rich particles ( $\theta$ phase - $Al_2Cu$ ):**

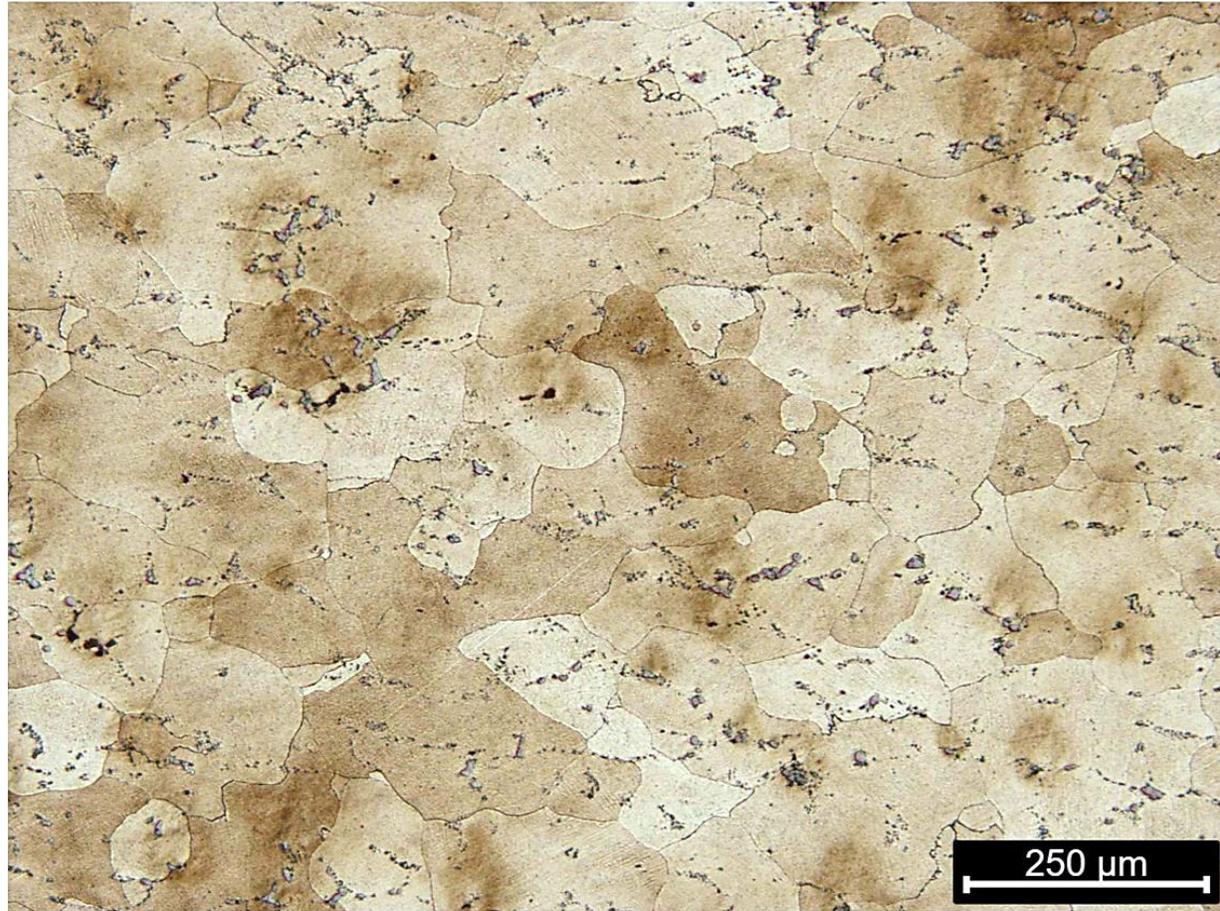
1. They are stress risers and will increase  $K_t$  during tensile testing and promote crack initiation
2. The greater the number of coarse Cu-rich particles, the easier for micro-cracks to connect
3. Many connected micro-cracks lead to larger cracks and a drastic reduction in fracture elongation

# **Optical Microscopy Results - Charts 8 – 18**

OM lead: Matt Medders

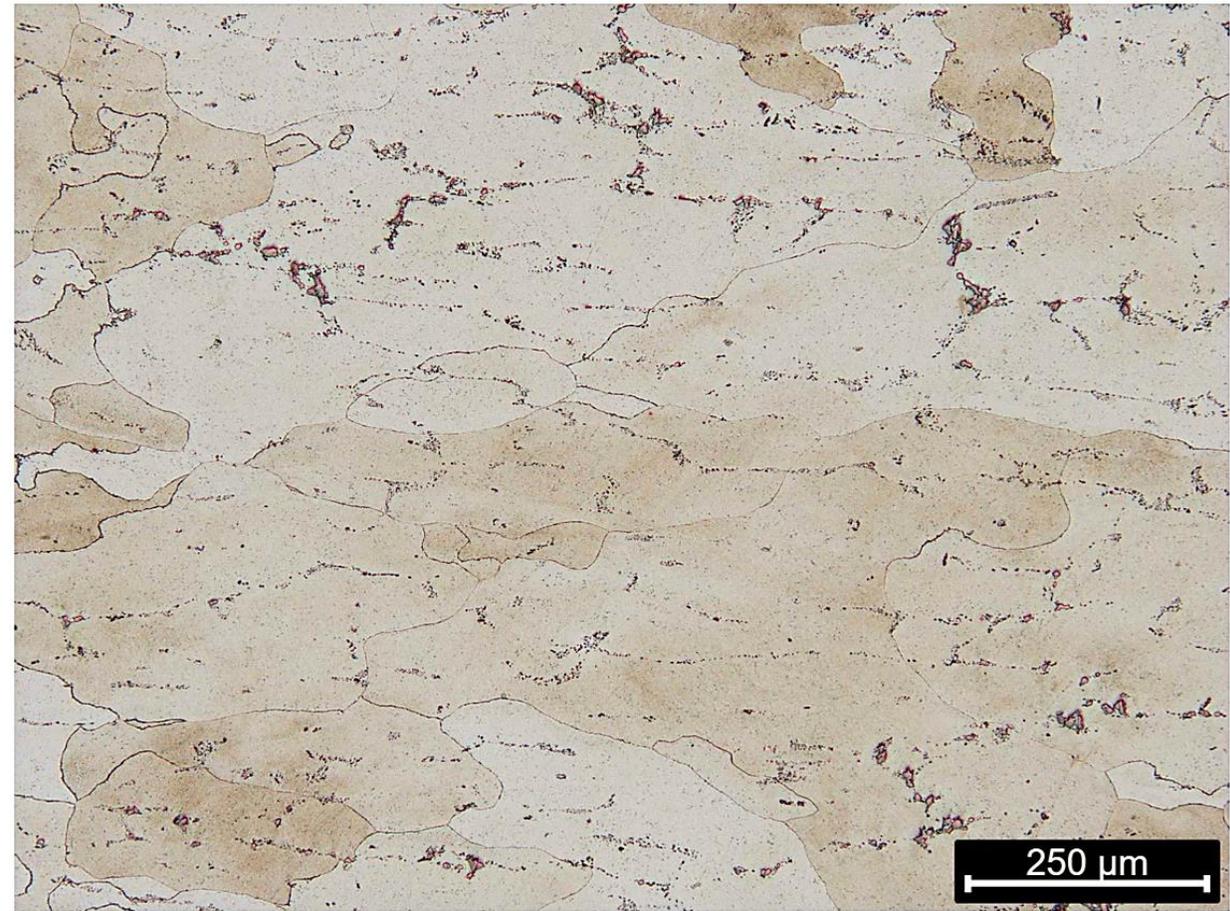
## Grain Structure Comparison – 720-2 vs. 723C-6

Higher Rx, smaller GS, fewer dendritic cells



720-2 LN2 – 7.23%

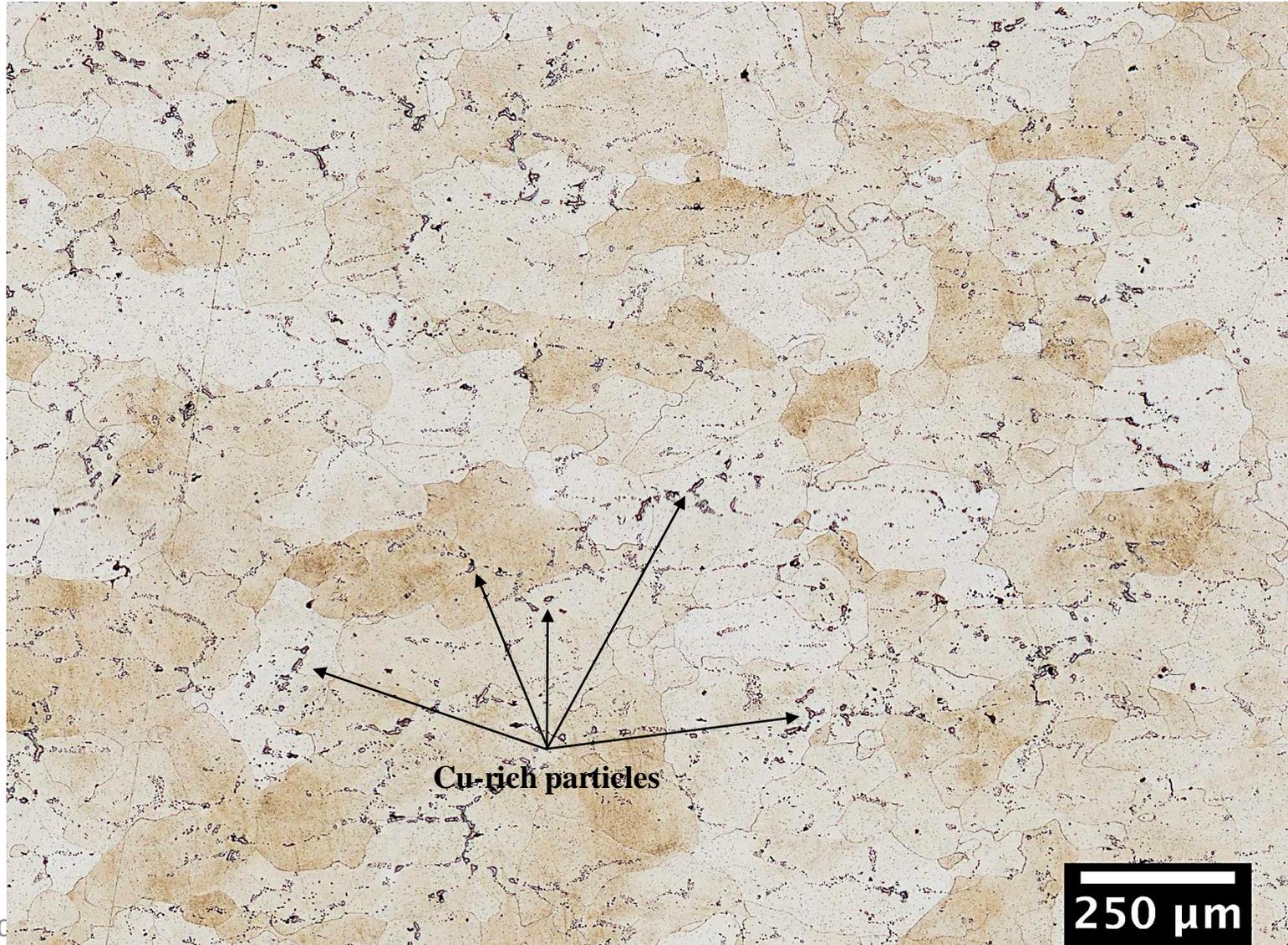
Lower Rx, larger GS, distinct prior dendritic structure



723C-6 LN2 – 1.25%

# Coarse Cu-Rich Particles Size & Distribution in Sample 720-2

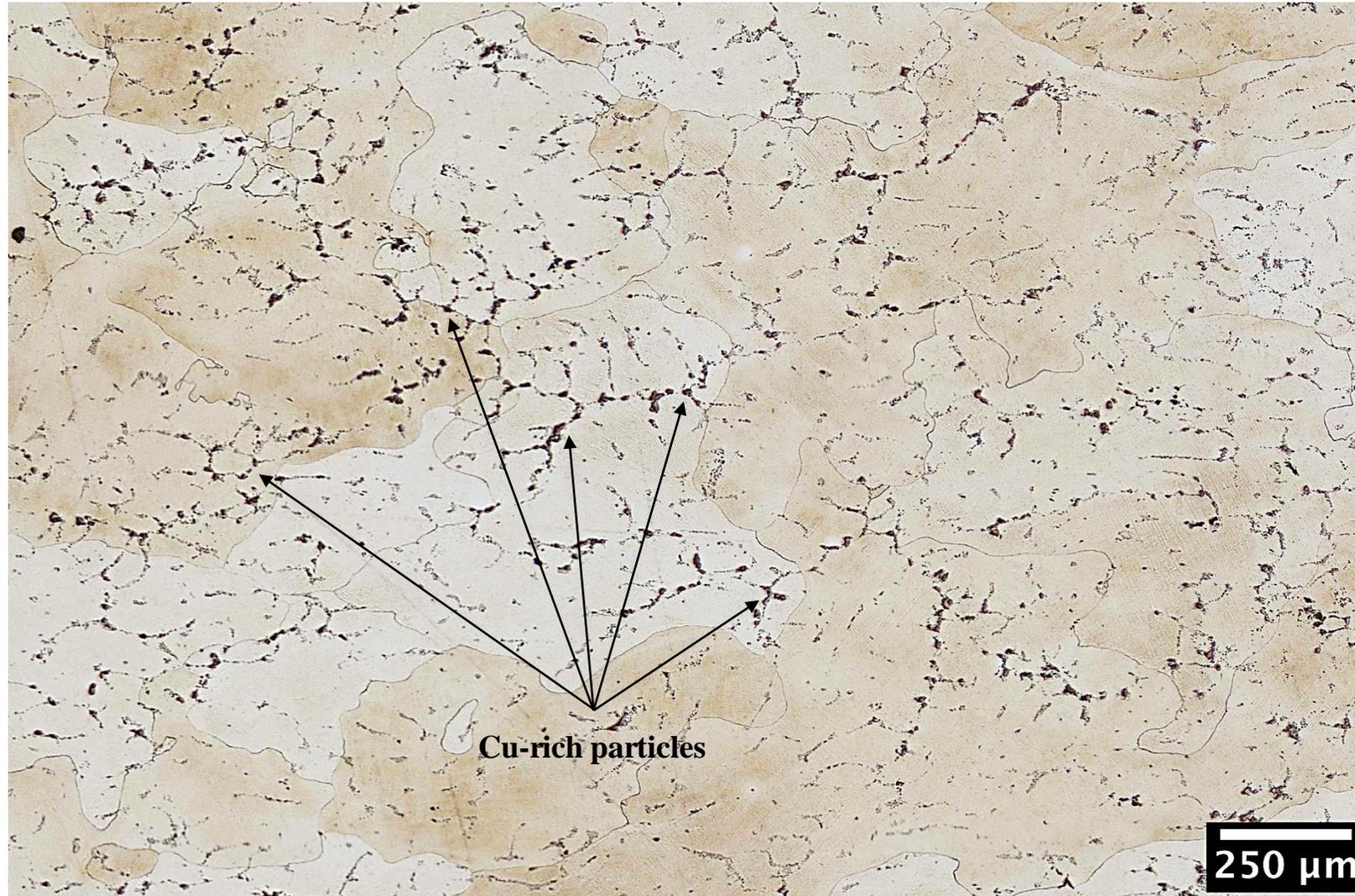
720-2 LN2 – 7.23%



1. The small (pinkish) particles are Cu-rich  $\theta$  phase,  $\text{Al}_2\text{Cu}$
2. More uniform distribution of Cu-rich particles ( $\text{Al}_2\text{Cu}$ - $\theta$  phase)
3. No clear sign of coarse Cu-rich particles at prior dendritic boundaries

# Coarse Cu-Rich Particles Size & Distribution in Sample 722C-5

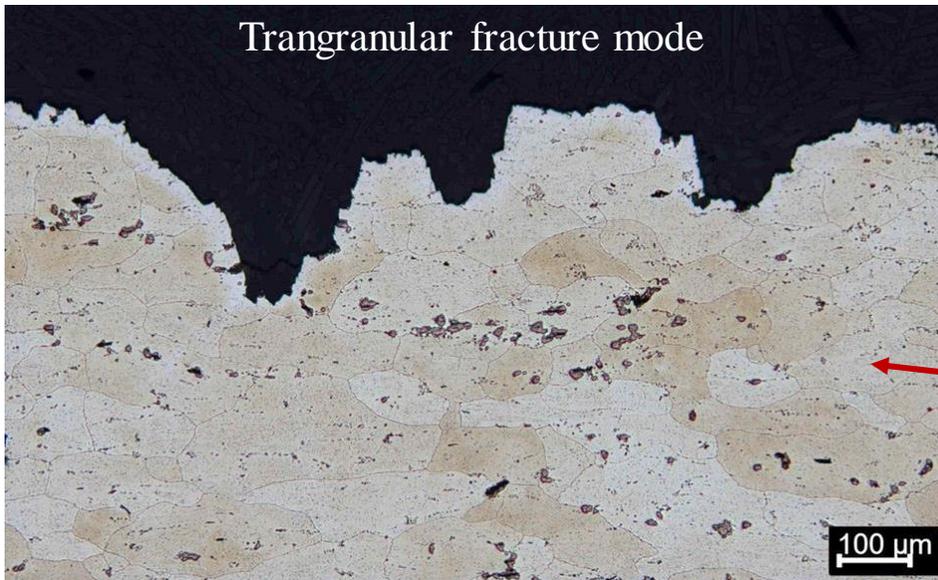
722C-5 LN2 – 1.0%



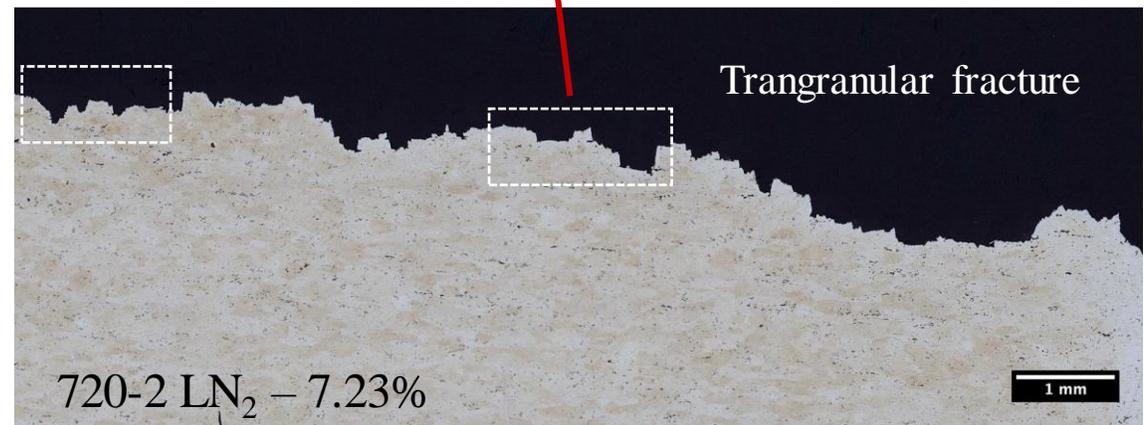
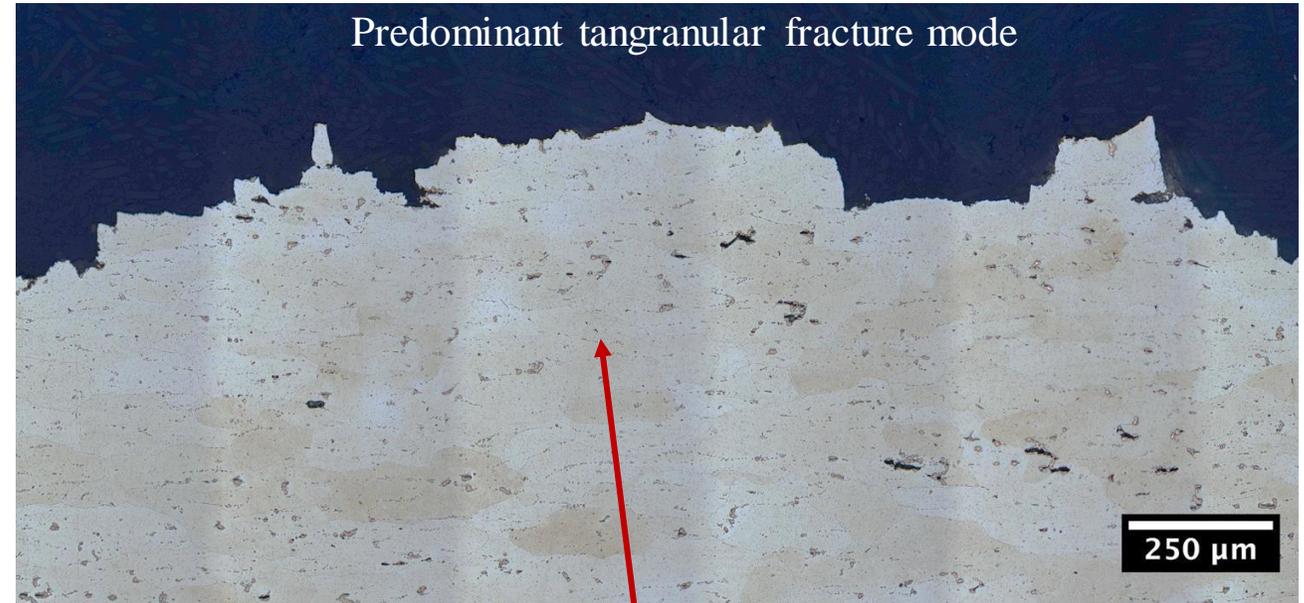
1. The small (pinkish) particles are Cu-rich  $\theta$  phase,  $\text{Al}_2\text{Cu}$
2. Non-uniform distribution of Cu-rich particles ( $\text{Al}_2\text{Cu}-\theta$  phase)
3. More coarse Cu-rich particles are present along the prior dendritic boundaries

# Cross-Sectional View of Fracture Surface - 720-2 LN<sub>2</sub> – 7.23%

1. Predominant transgranular fracture mode
2. No sign of continuous cracking along grain boundaries

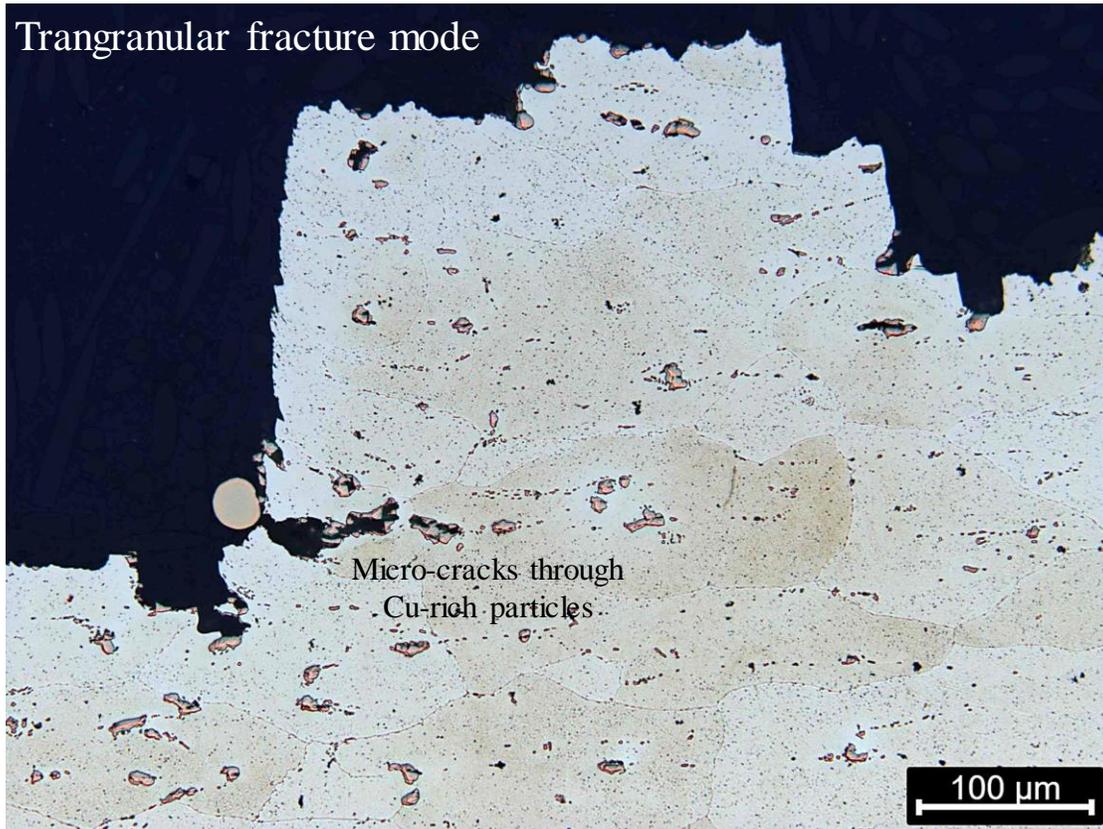


No sign of brittle fracture

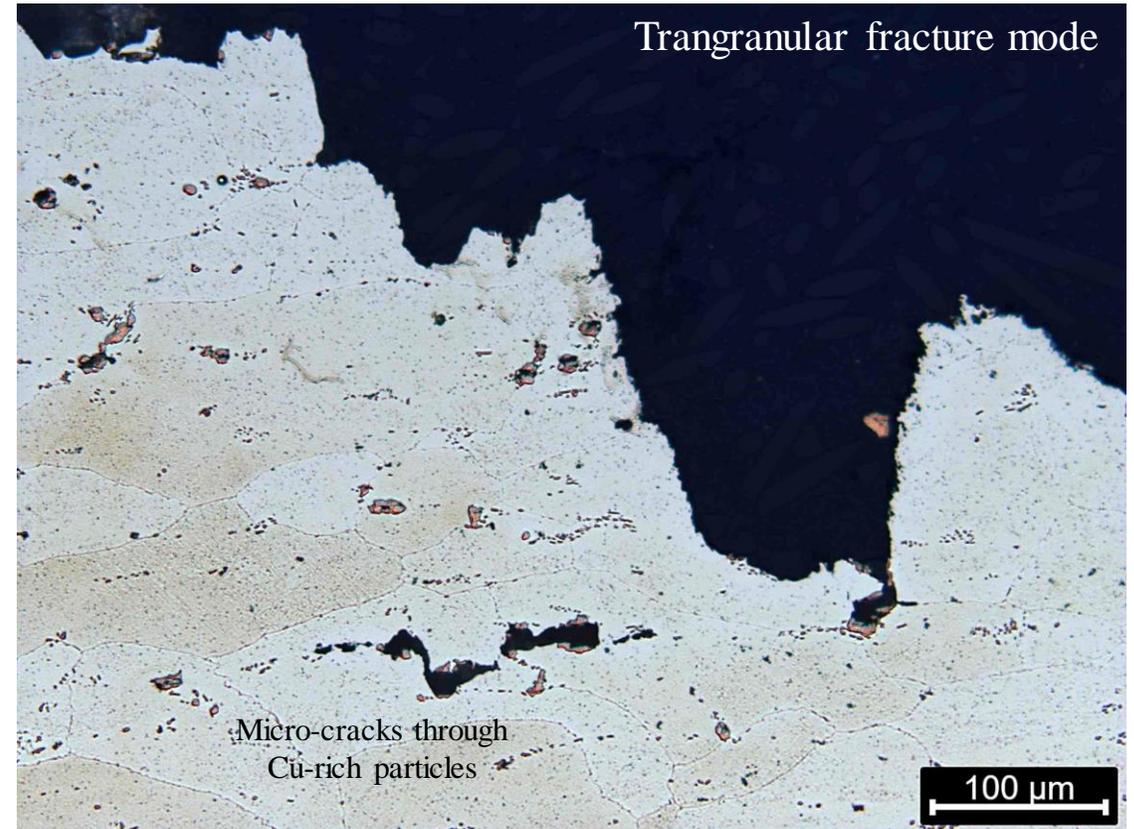


# Close-up View of Fracture Surface - 720-2 LN<sub>2</sub> – 7.23%

720-2 LN<sub>2</sub> – 7.23%



Signs of ductile fracture

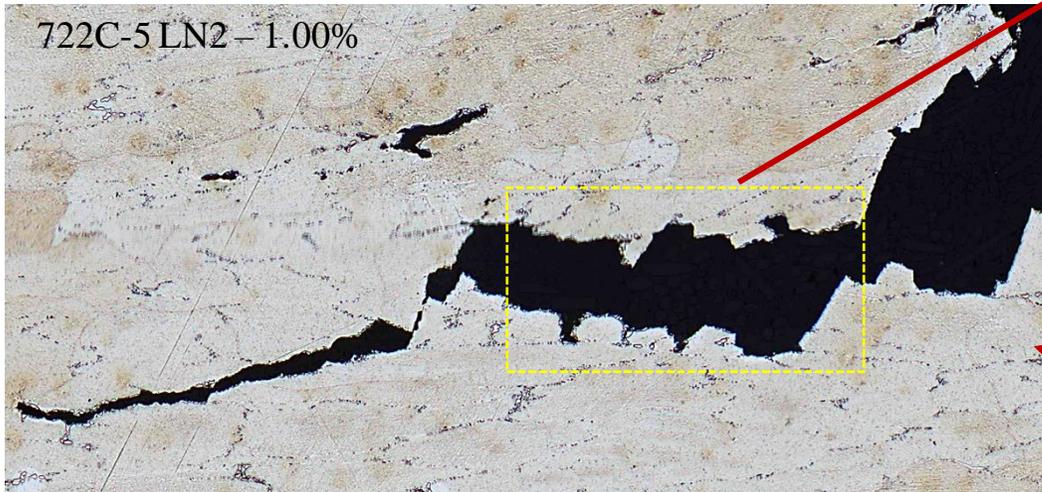


Signs of ductile fracture

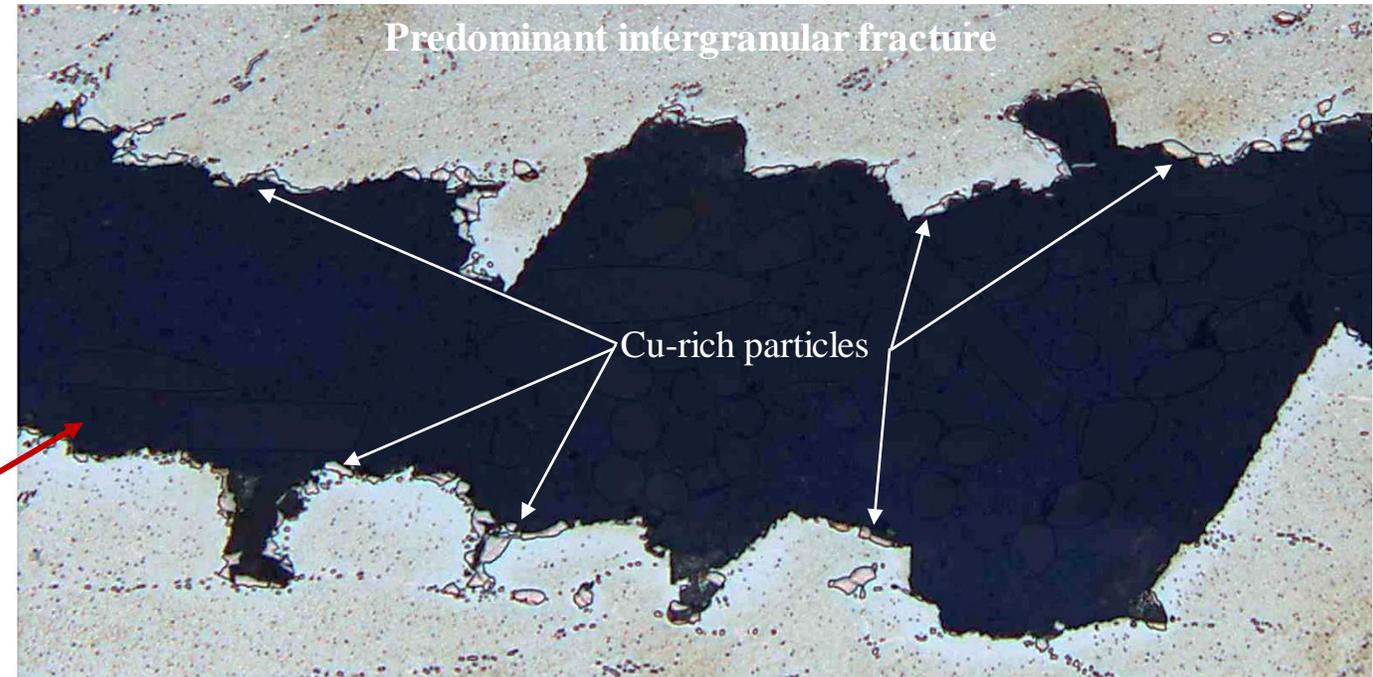
1. Predominant transgranular fracture mode, No sign of continuous cracking along grain boundaries
2. Some isolated micro-cracks, but micro-cracks do not connect through broken Cu-rich particles

# Cross-Sectional View of Fracture Surface - 722C-5 LN<sub>2</sub> – 1.0%

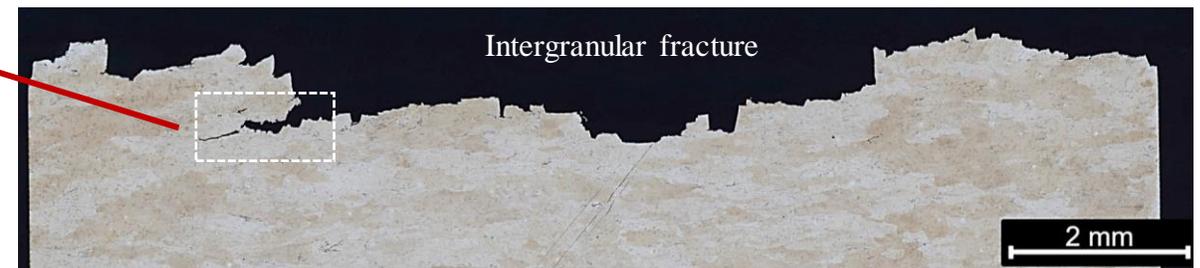
1. Predominant Intergranular fracture mode
2. Clear signs of continuous cracking on Cu-rich particles along grain boundaries



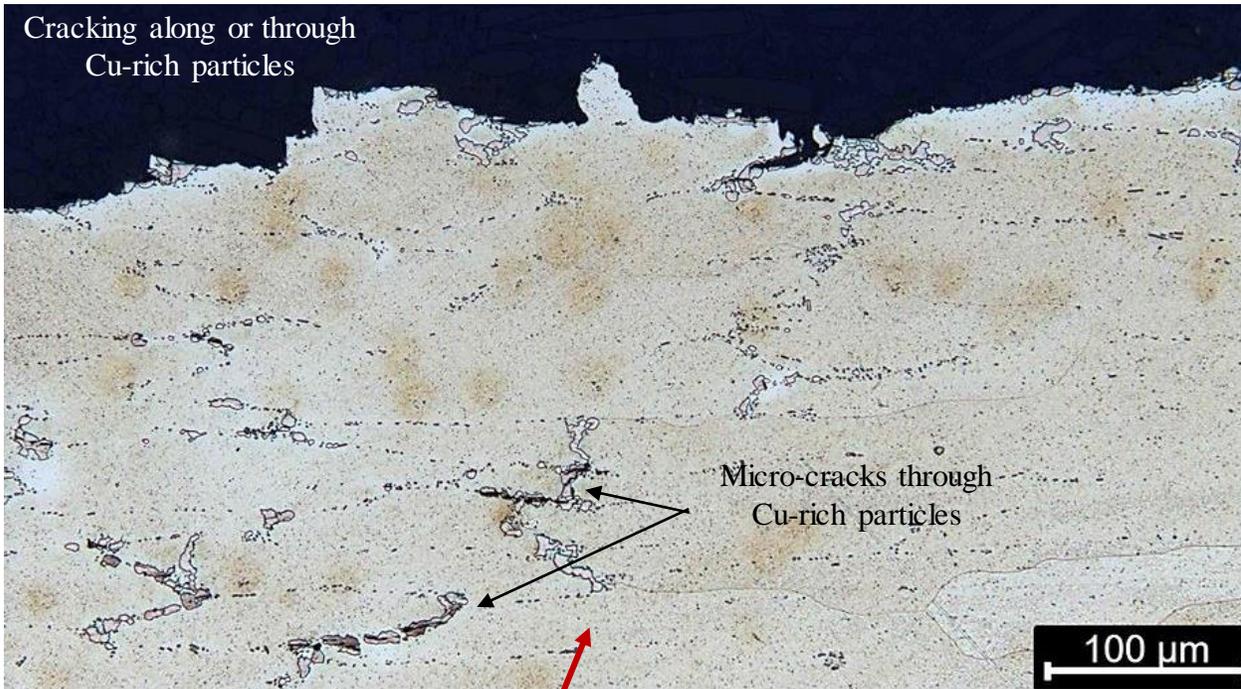
Signs of brittle fracture



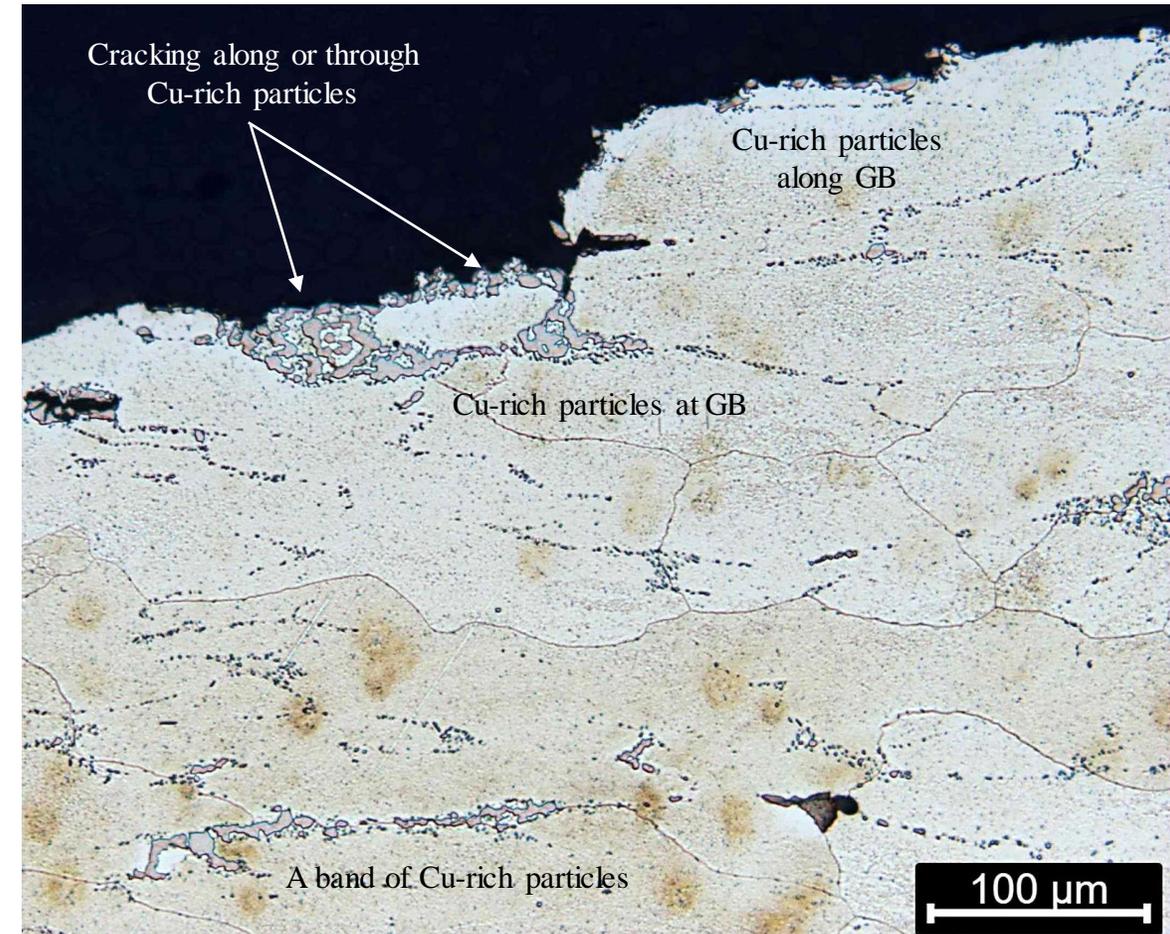
Fracture path along Cu-rich particle boundary



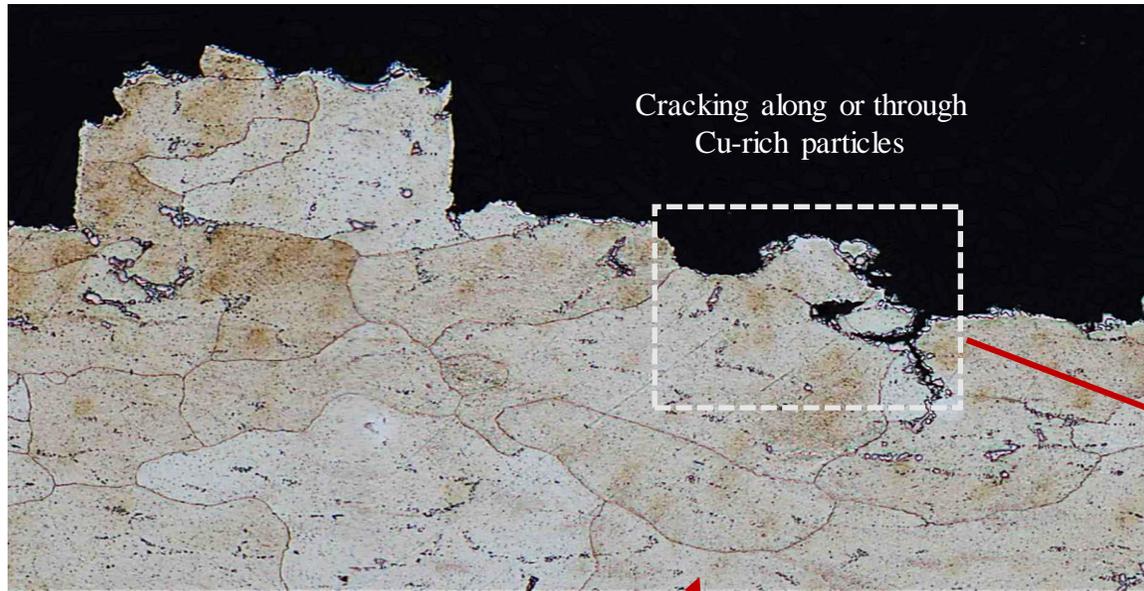
# Cross-Sectional View of Fracture Surface - 722C-5 LN<sub>2</sub> – 1.0%



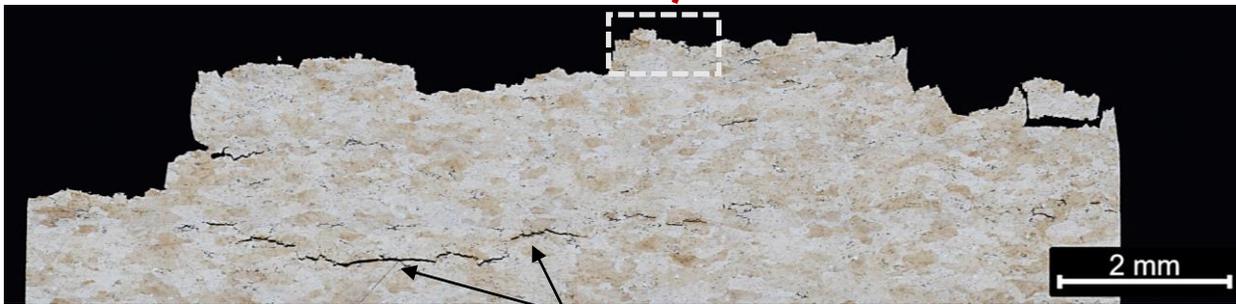
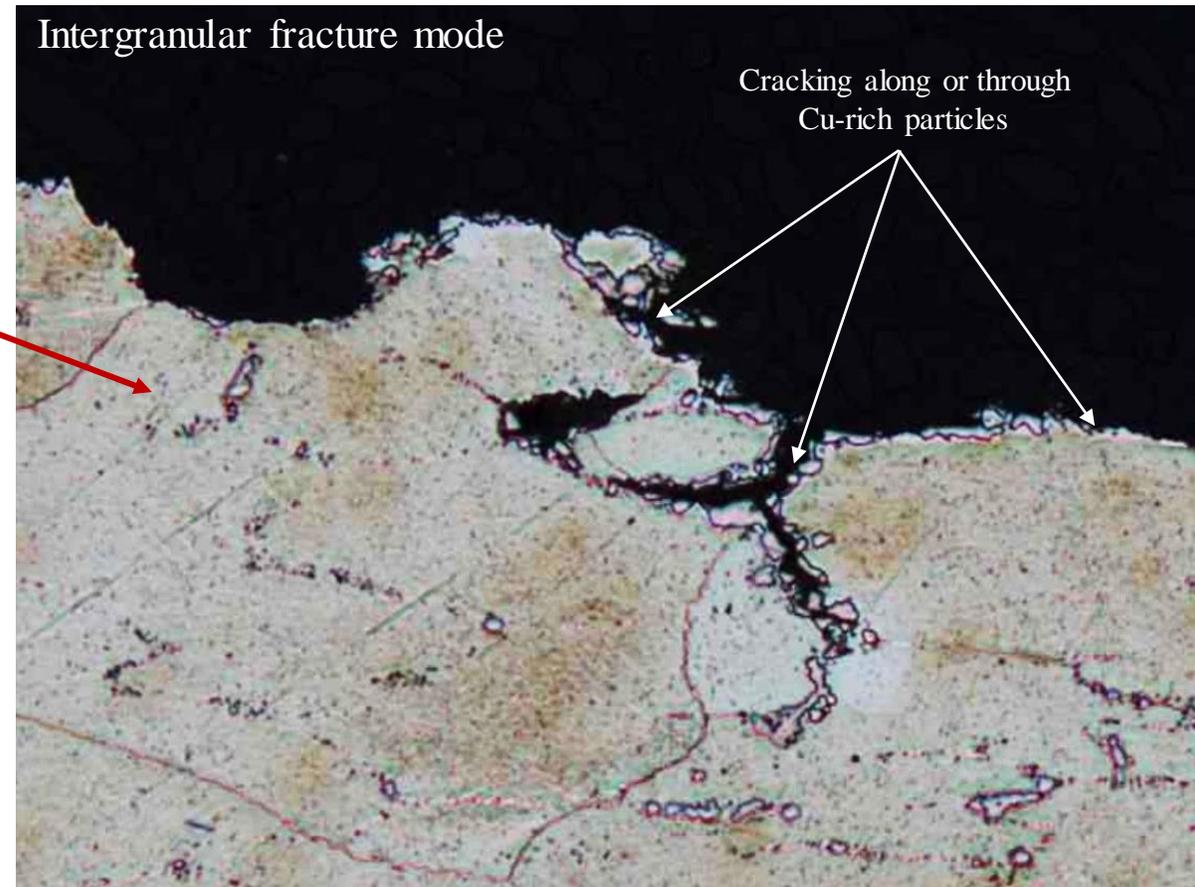
## Signs of brittle fracture



# Cross-Sectional View of Fracture Surface - 723C-6 LN<sub>2</sub> – 1.25%



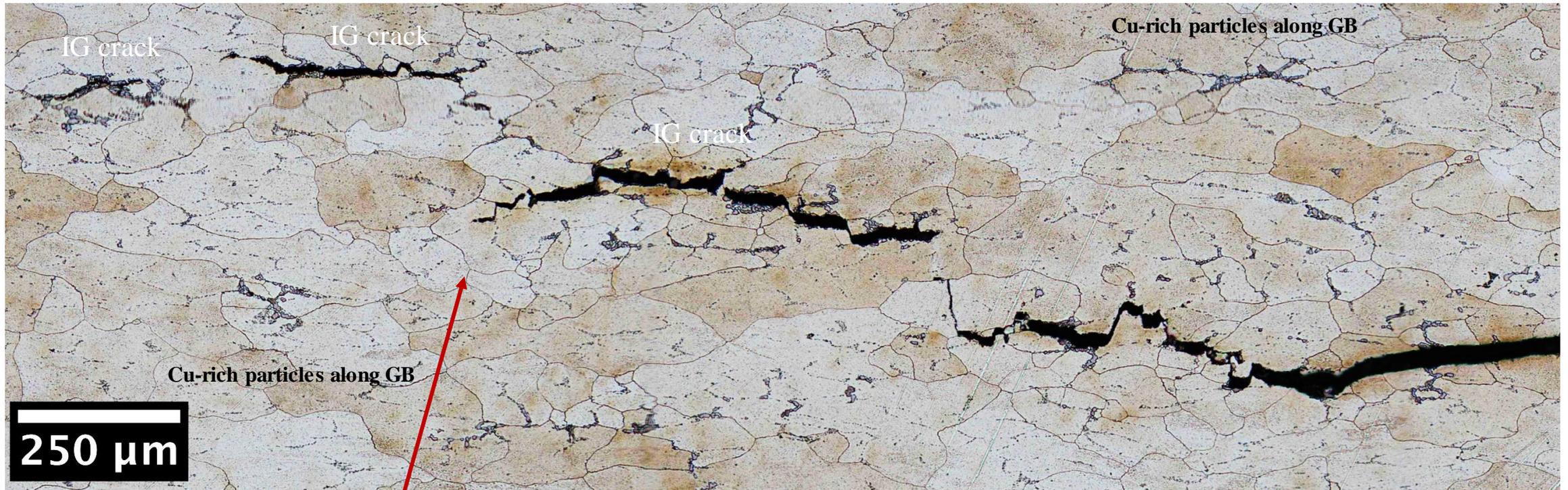
Signs of brittle fracture



Long subsurface cracks  $\perp$   
to the tensile direction

# Cross-up View of Subsurface Cracks - 723C-6 LN<sub>2</sub> - 1.25%

Predominant intergranular fracture mode



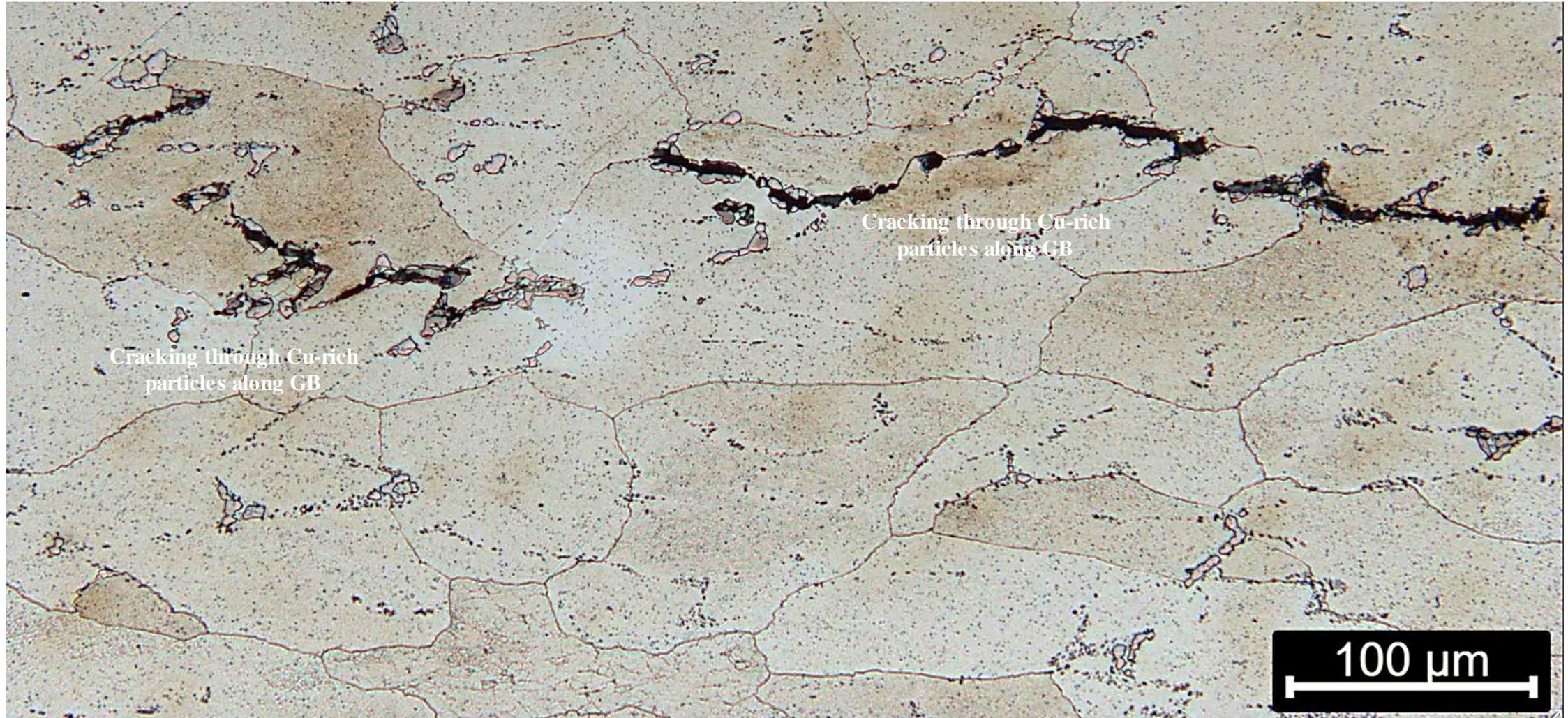
Cracking through GB and Cu-rich particles



Long subsurface cracks  $\perp$  to the tensile direction

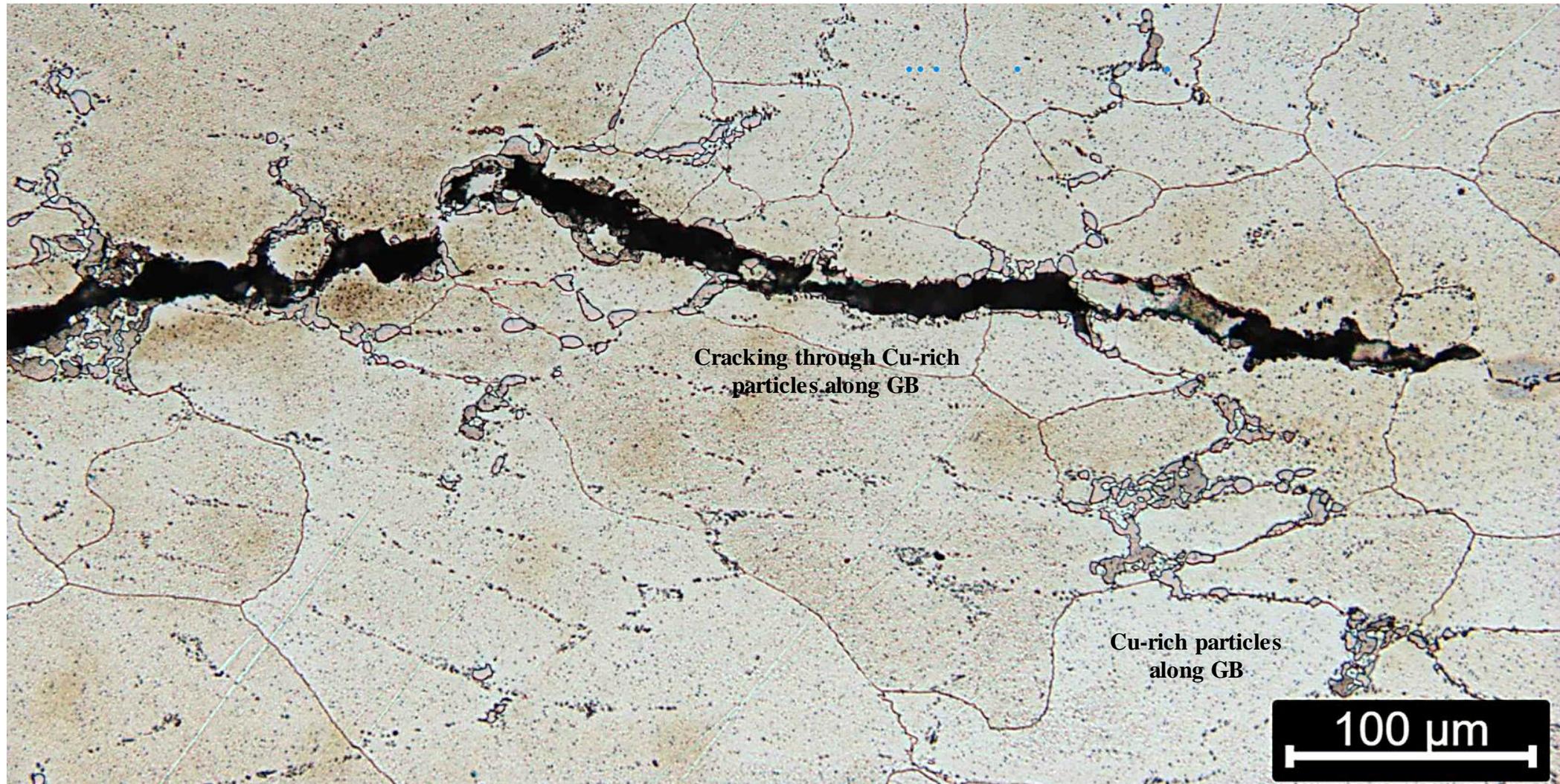
# Cross-up View of Subsurface Cracks - 723C-6 LN<sub>2</sub> – 1.25%

Predominant intergranular fracture mode



# Cross-up View of Subsurface Cracks - 723C-6 LN<sub>2</sub> – 1.25%

Predominant intergranular fracture mode



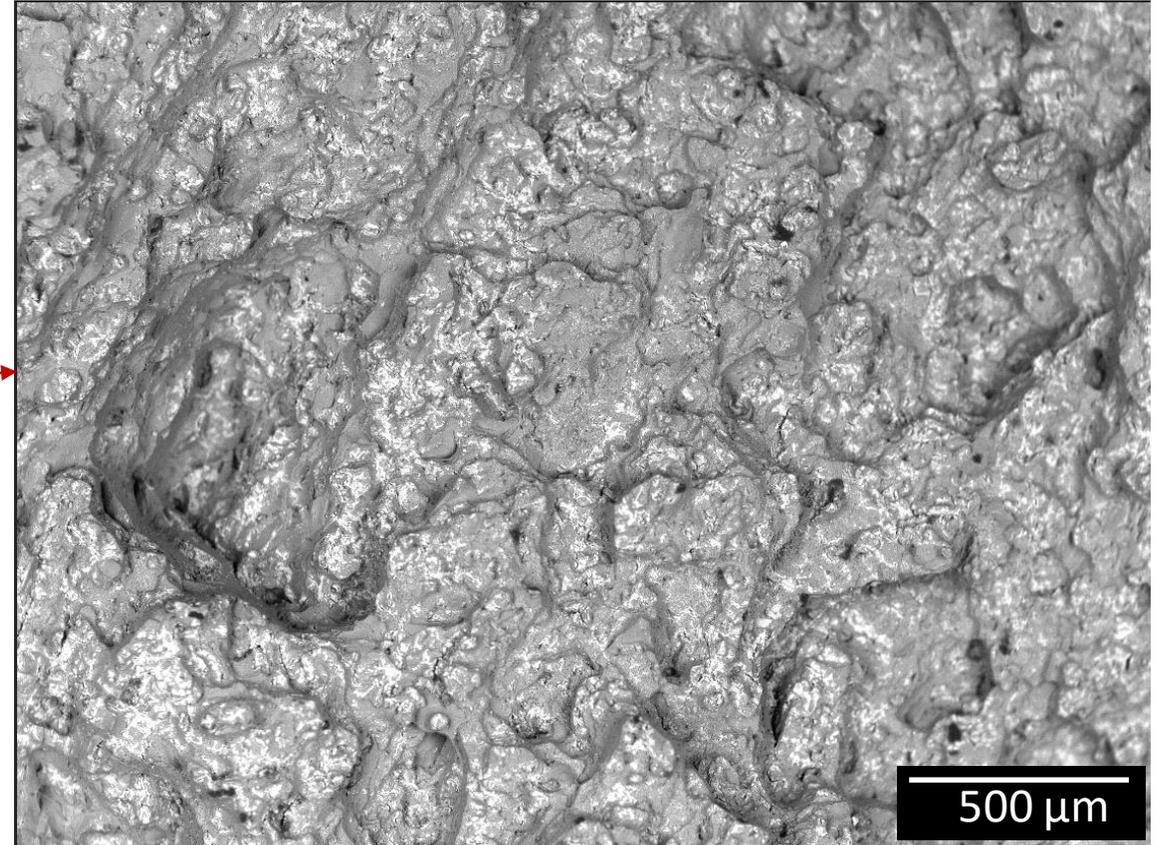
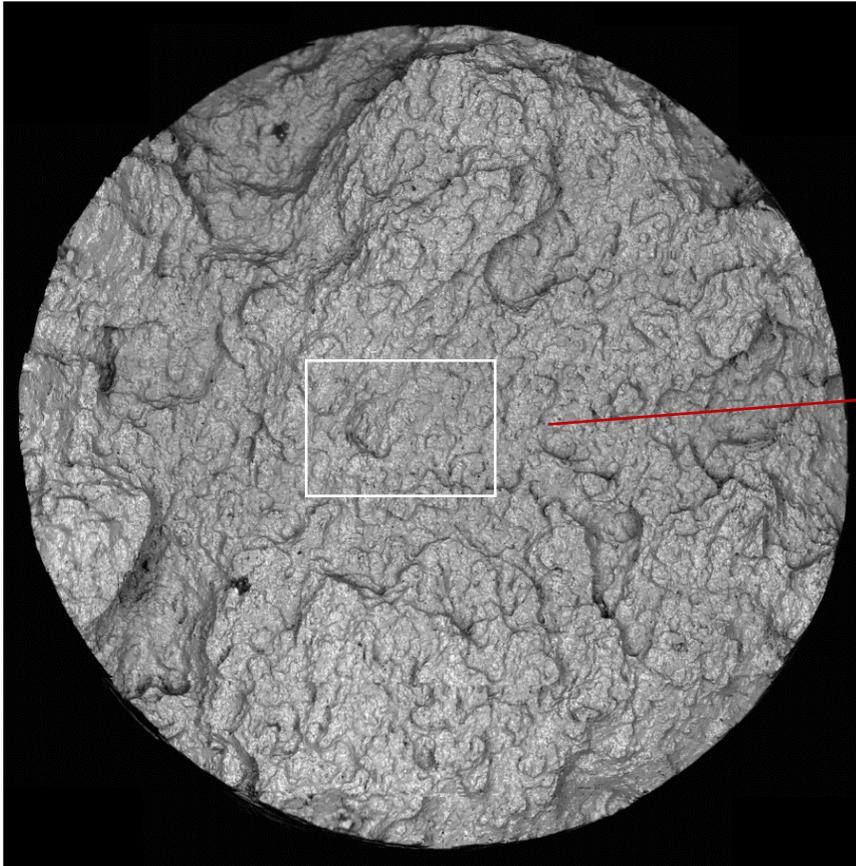
# **SEM/X-ray Map Results - Charts 20 - 31**

SEM lead: Ellen Rabenberg

# SEM Fractography of Sample 720-2

720-2 LN2 – 7.23%

White particles are Cu-rich precipitates

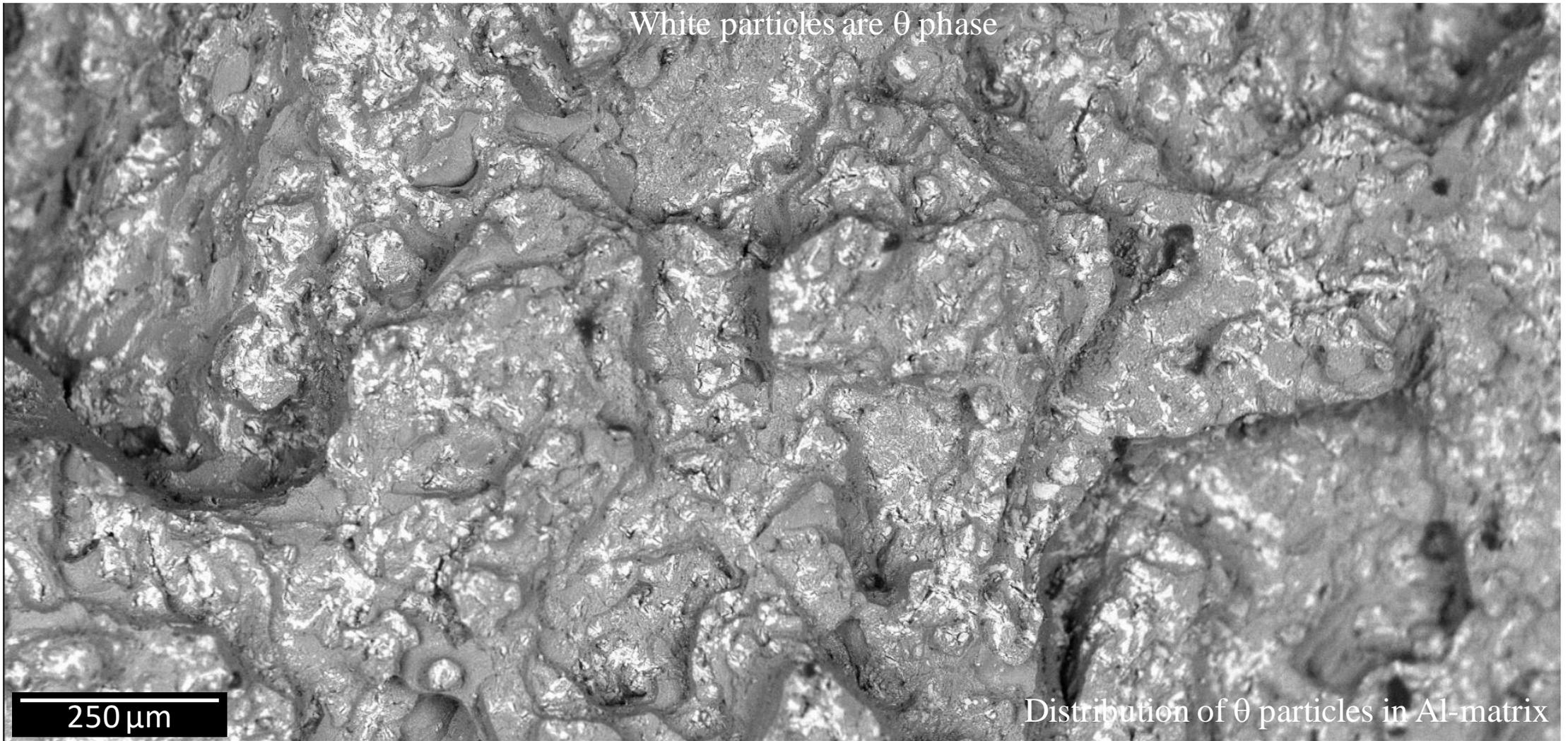


SEM image – entire fracture surface

Distribution of Cu-rich particles in Al-matrix

Cu-rich particles are finer and have more uniform distribution

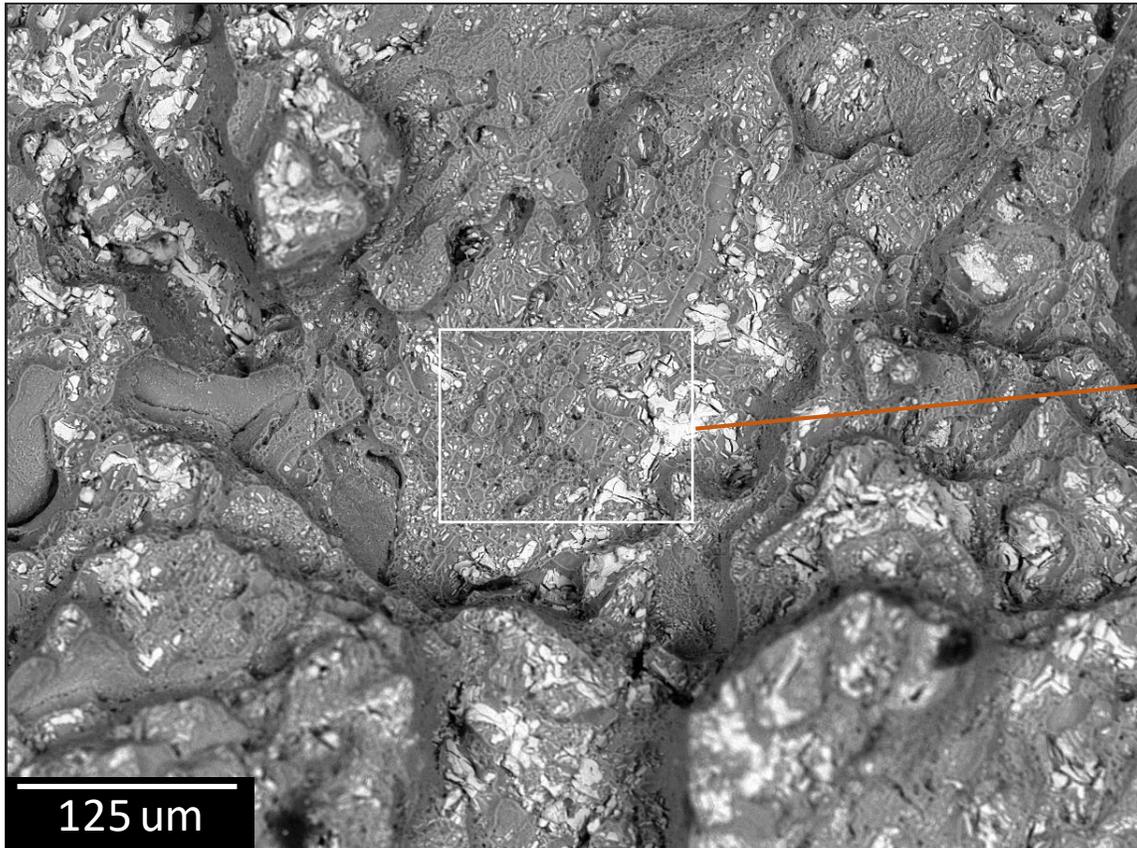
## SEM Fractography of Sample 720-2, LN2 – 7.23%



Cu-rich particles have more uniform distribution

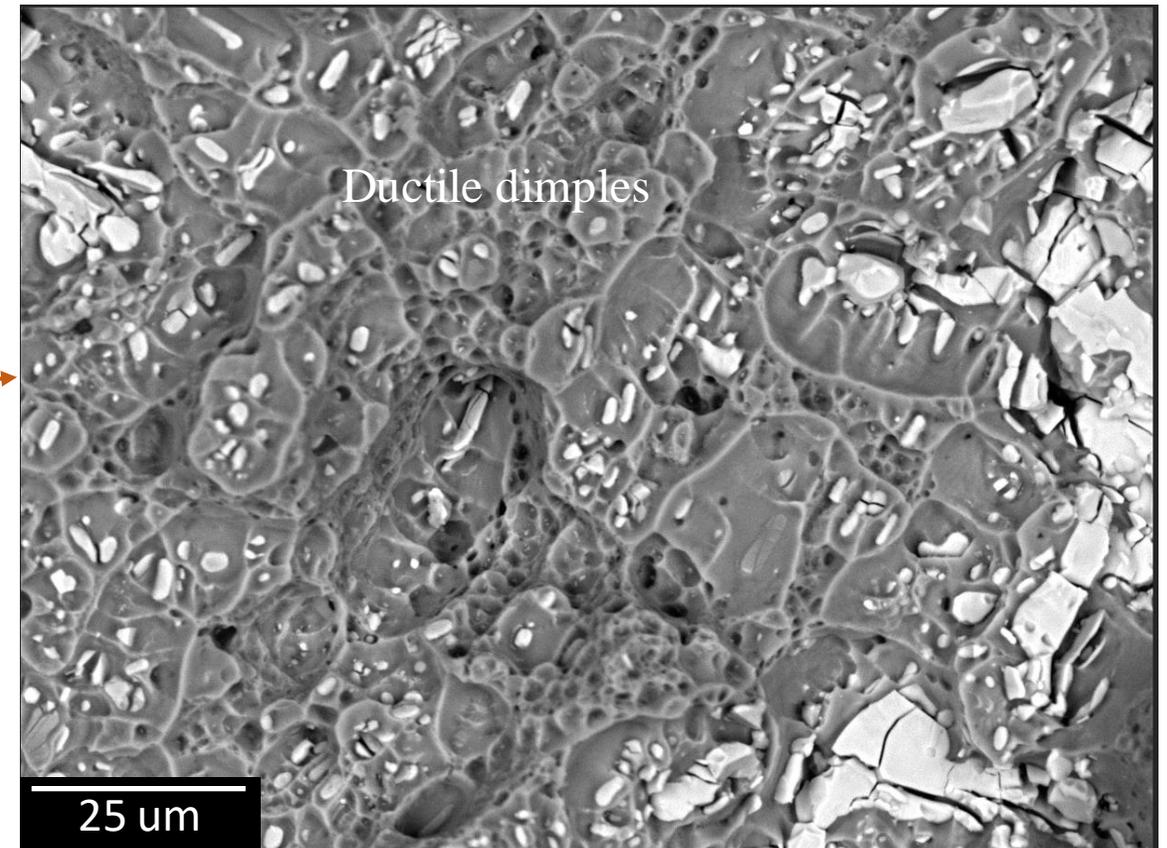
# SEM Fractography of Sample 720-2

720-2 LN2 – 7.23%



Distribution of Cu-rich particles in AL-matrix

White particles are Cu-rich precipitates

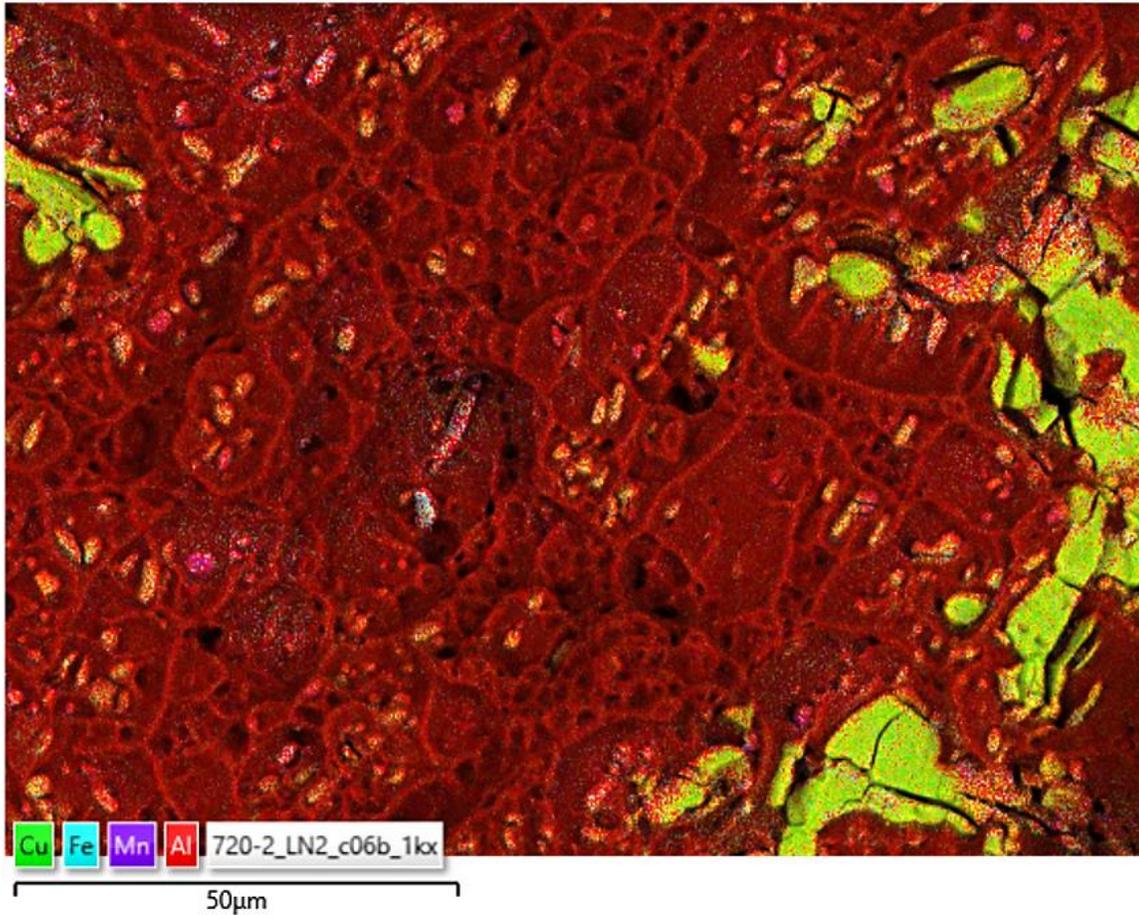


Most Cu-rich particles are in dimples

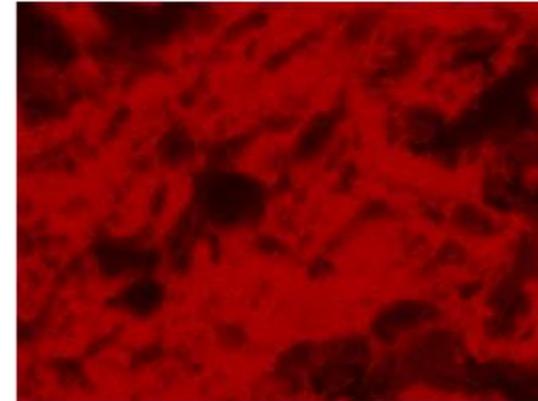
1. Cu-rich particles are finer and have more uniform distribution, signs of ductile fracture
2. Fine Cu-rich particles are in the middle of dimples, indicative of higher ductility

# SEM/X-ray Map for Sample 720-2

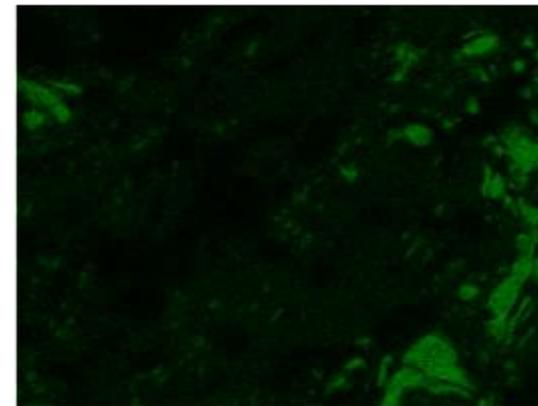
720-2 LN2 – 7.23%



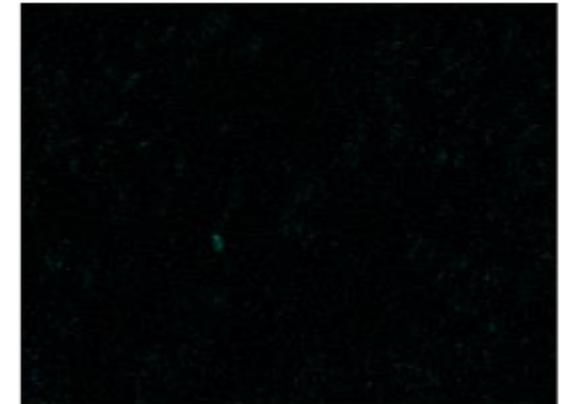
Al K series



Cu L series



Fe K series

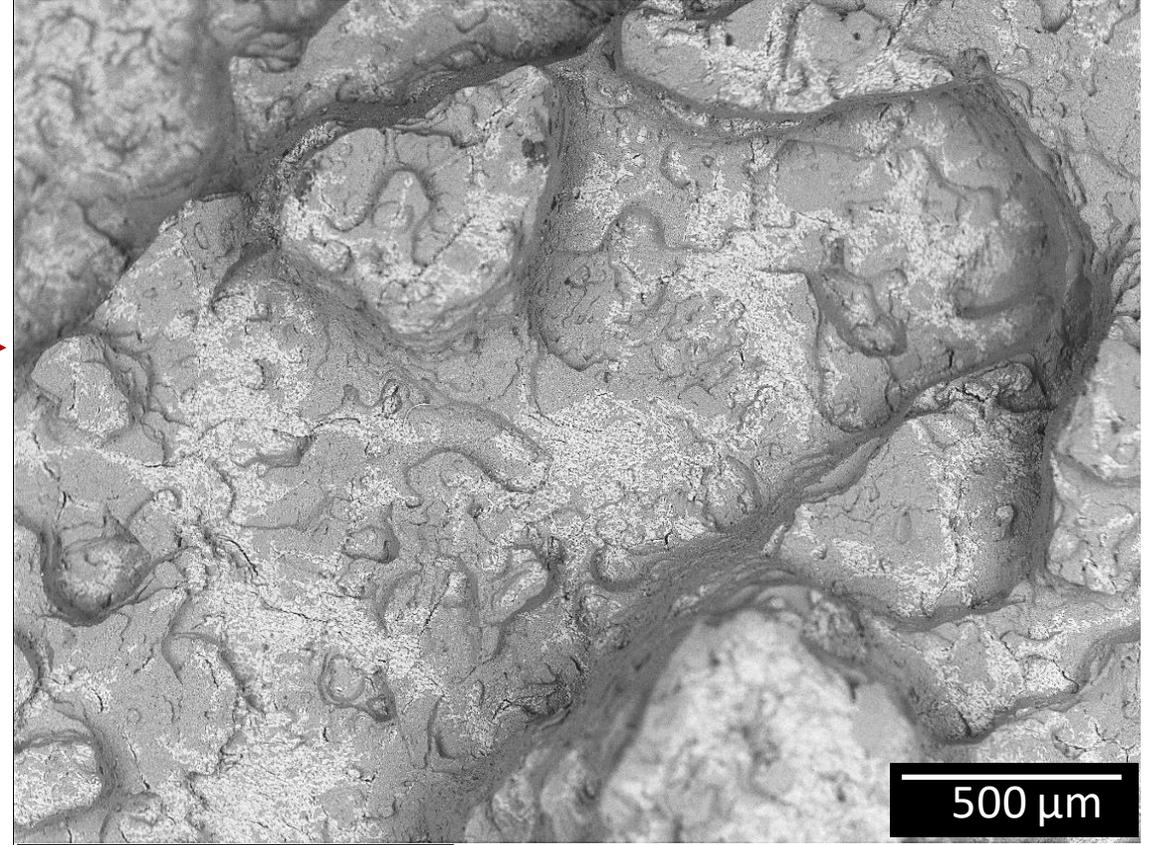
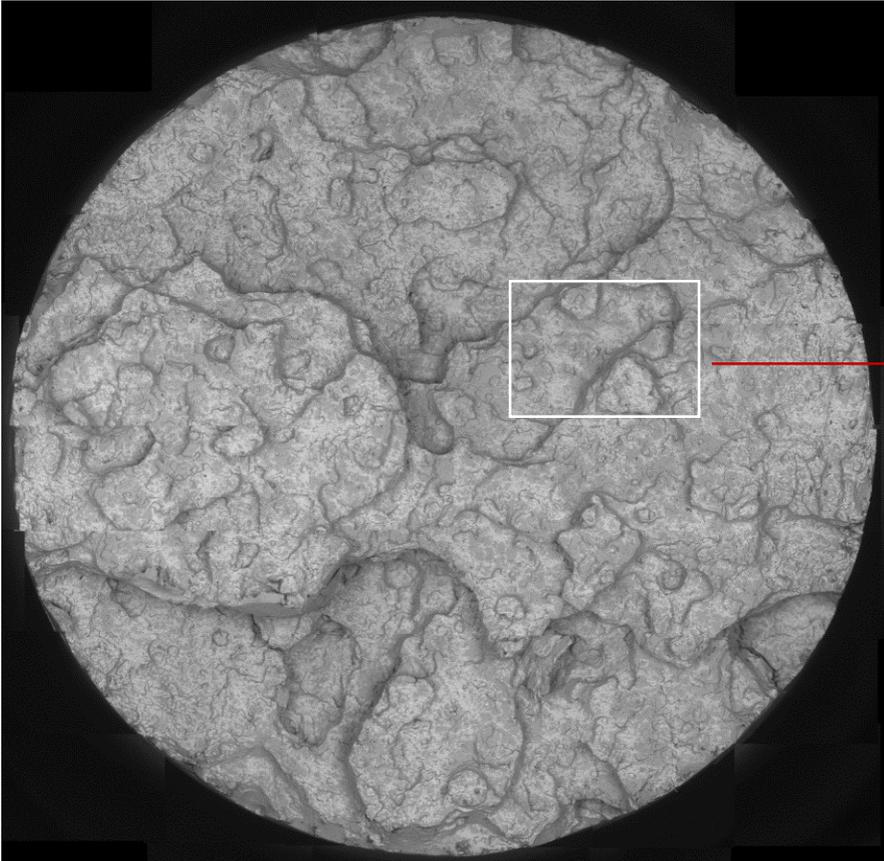


EDS confirms the white particles in SEM image (green color on x-ray map) are Cu-rich

# SEM Fractography of Sample 722C-5

722C-5 LN2 – 1.00%

White particles are Cu-rich precipitates

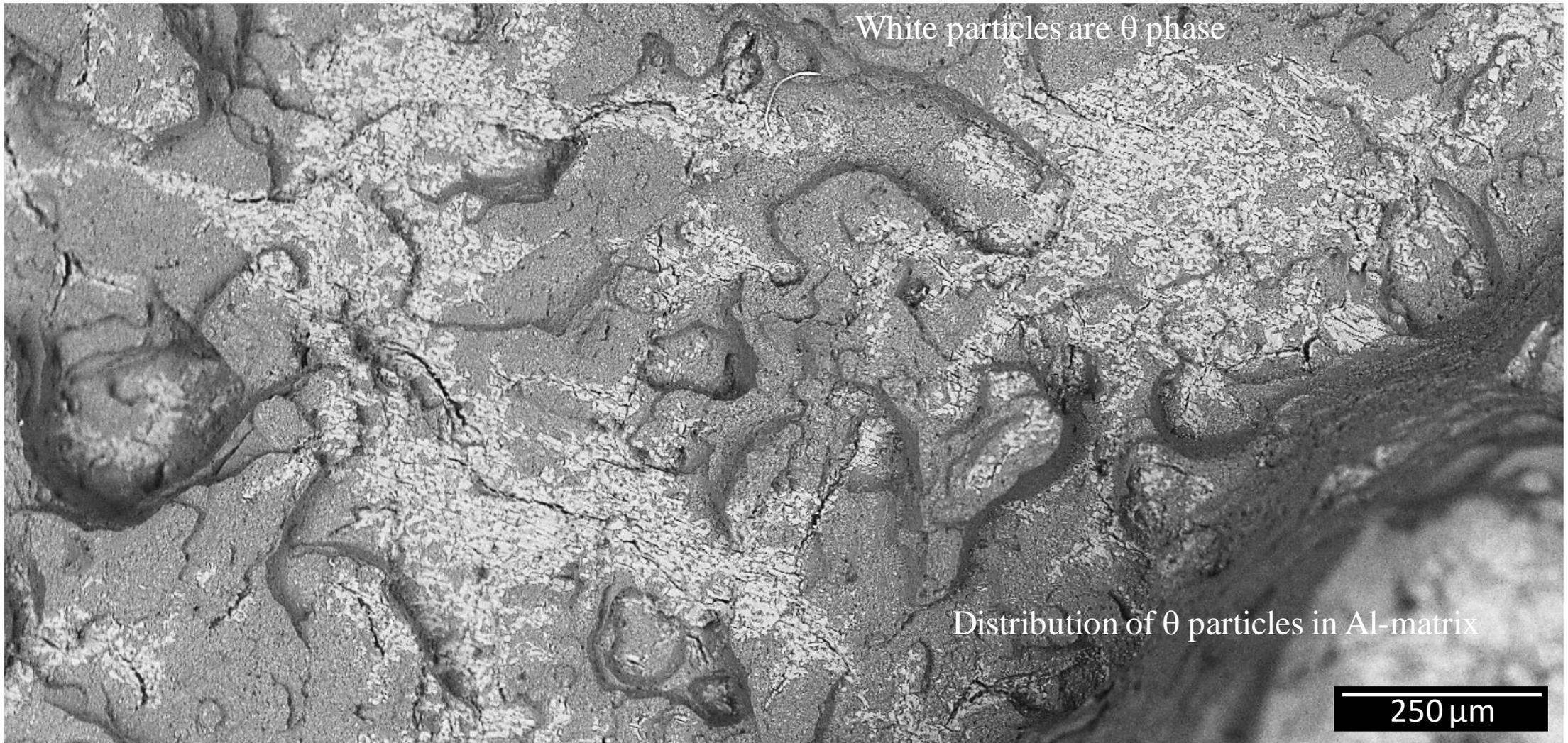


SEM image – entire fracture surface

Distribution of Cu-rich particles in AL-matrix

Cu-rich particles are coarser and have non-uniform distribution

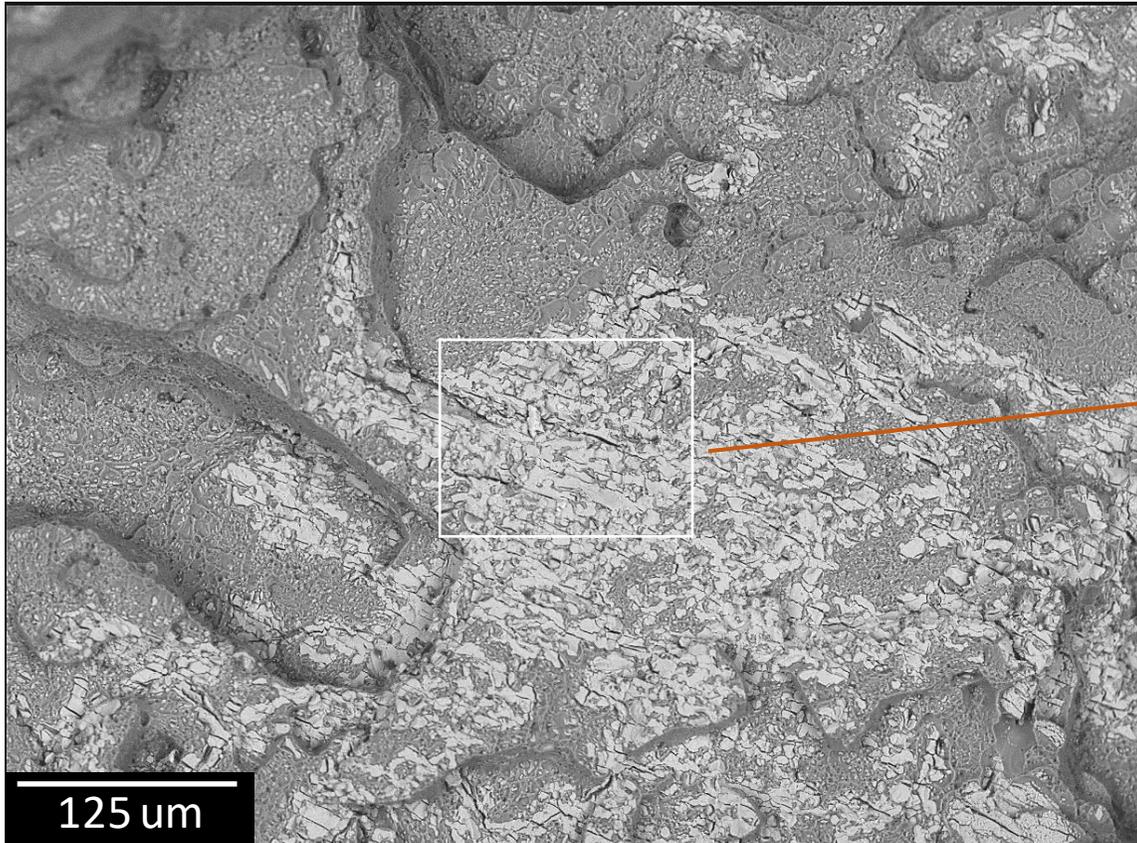
## SEM Fractography of Sample 722C-5 LN2 – 1.00%



Cu-rich particles have non-uniform distribution

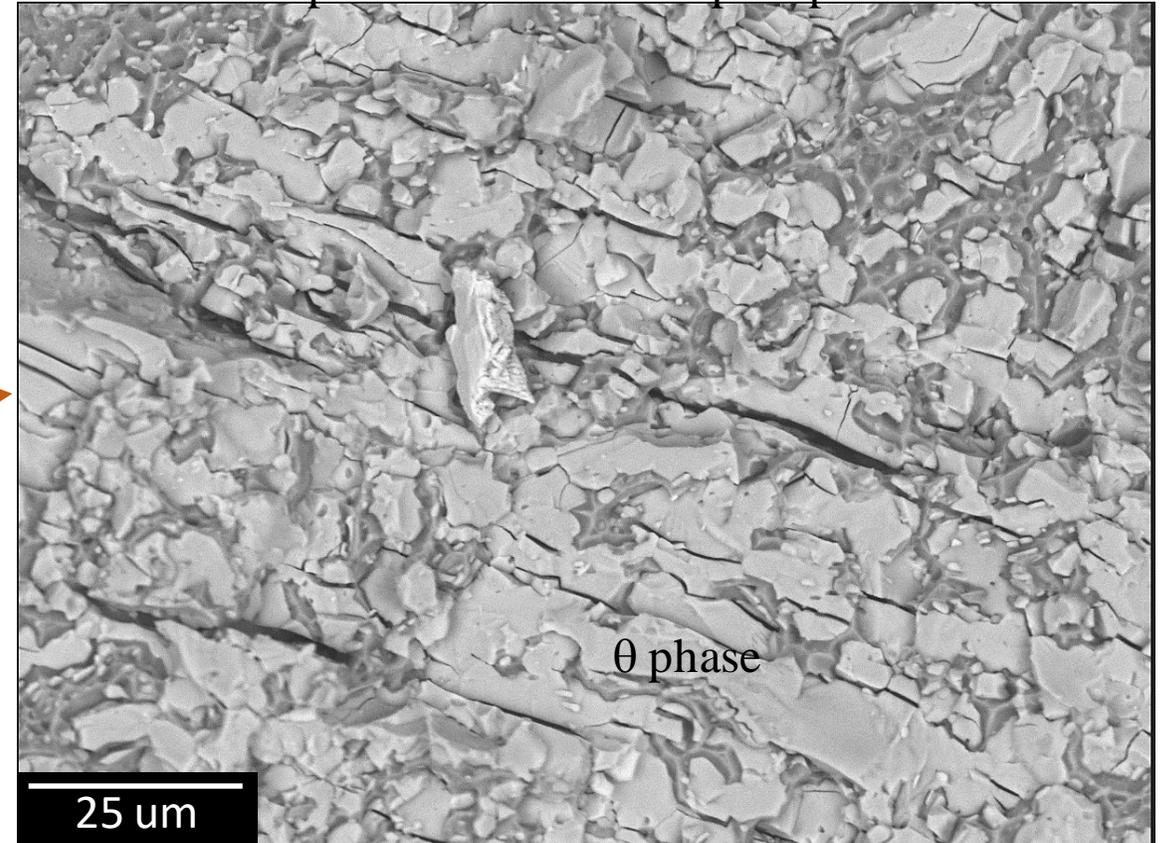
# SEM Fractography of Sample 722C-5

722C-5 LN2 – 1.00%



Clusters of Cu-rich particles with micro-cracks

White particles are Cu-rich precipitates

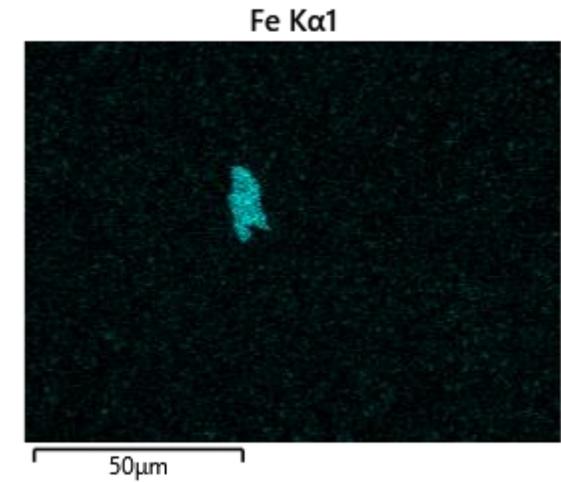
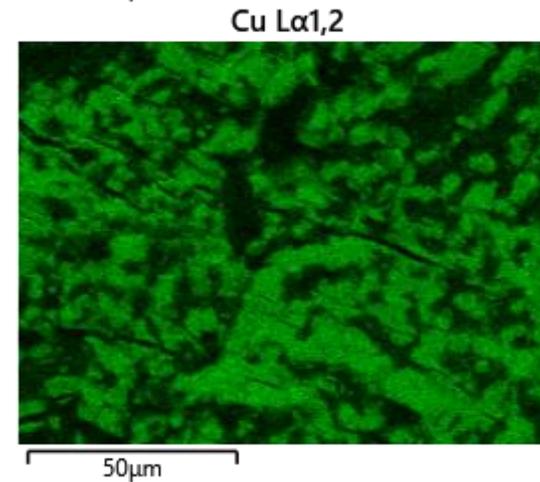
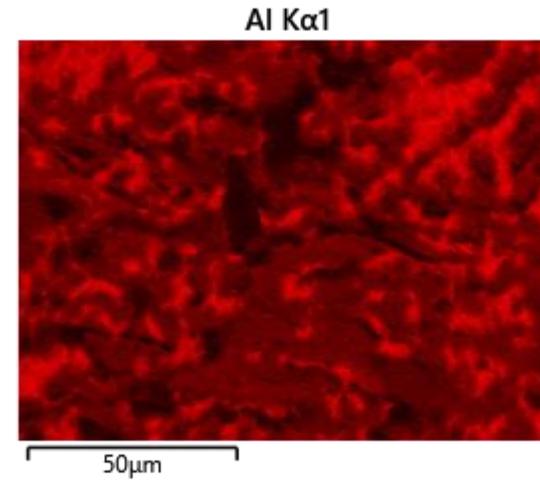
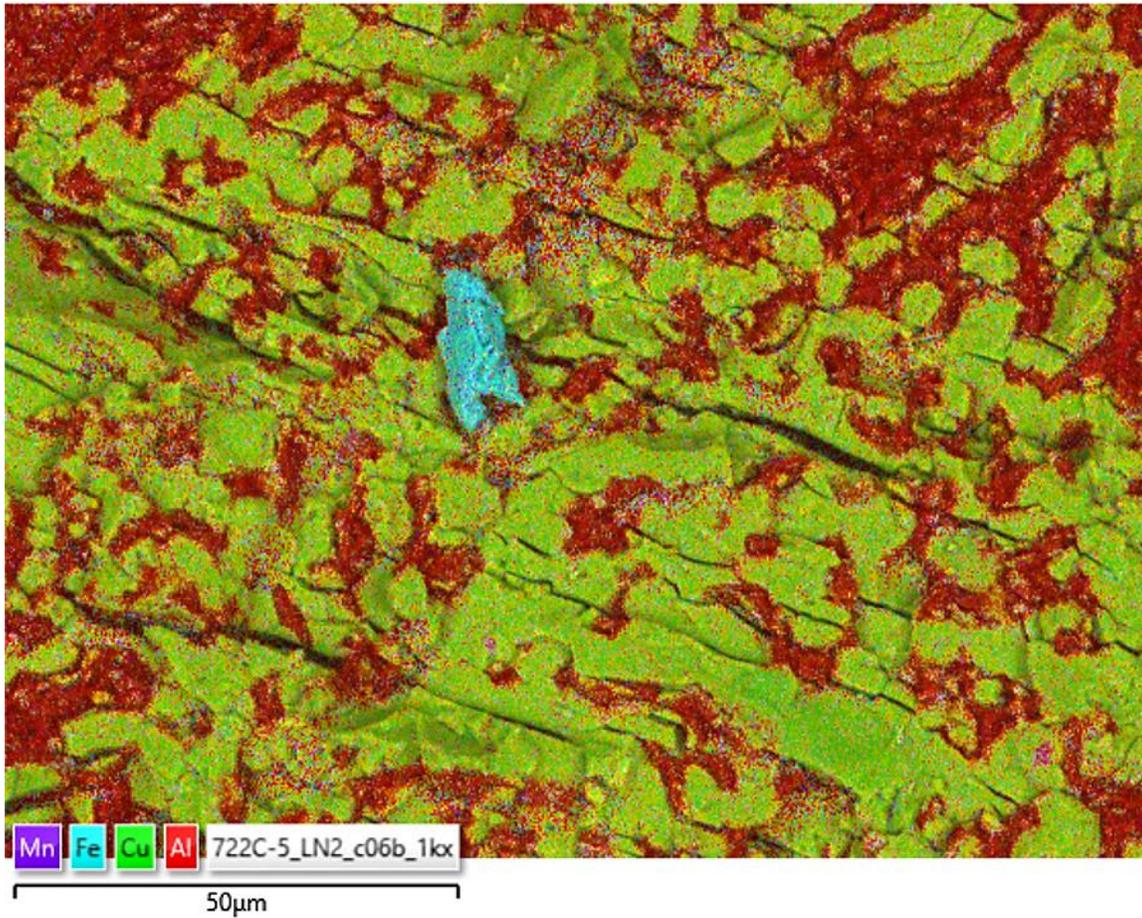


Cracking along or through Cu-rich particles

1. Cu-rich particles are coarser and have a tendency to aggregate together
2. Cu-rich particles are hard and brittle, acting as stress risers during tensile deformation
3. Many micro-cracks are present in the regions that have numerous Cu-rich particles

# SEM/X-ray Map for Sample 722C-5

722C-5 LN2 – 1.00%

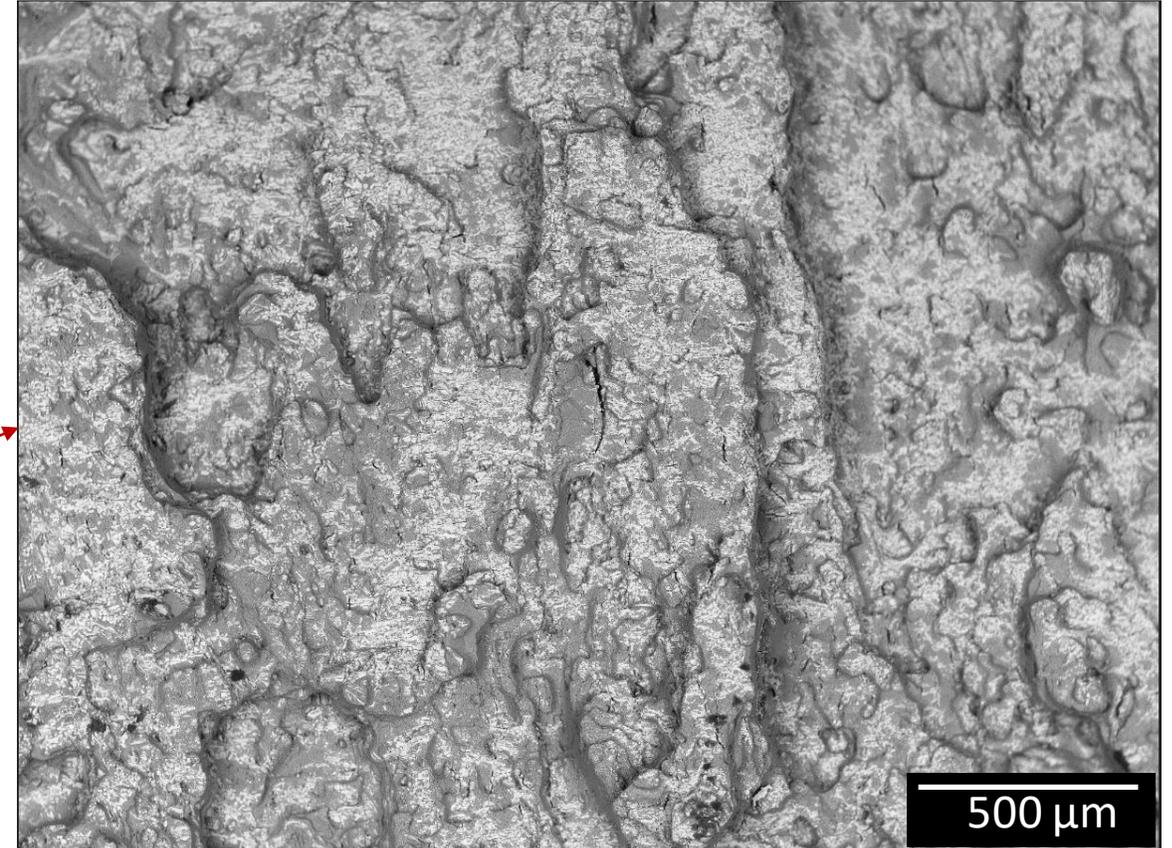
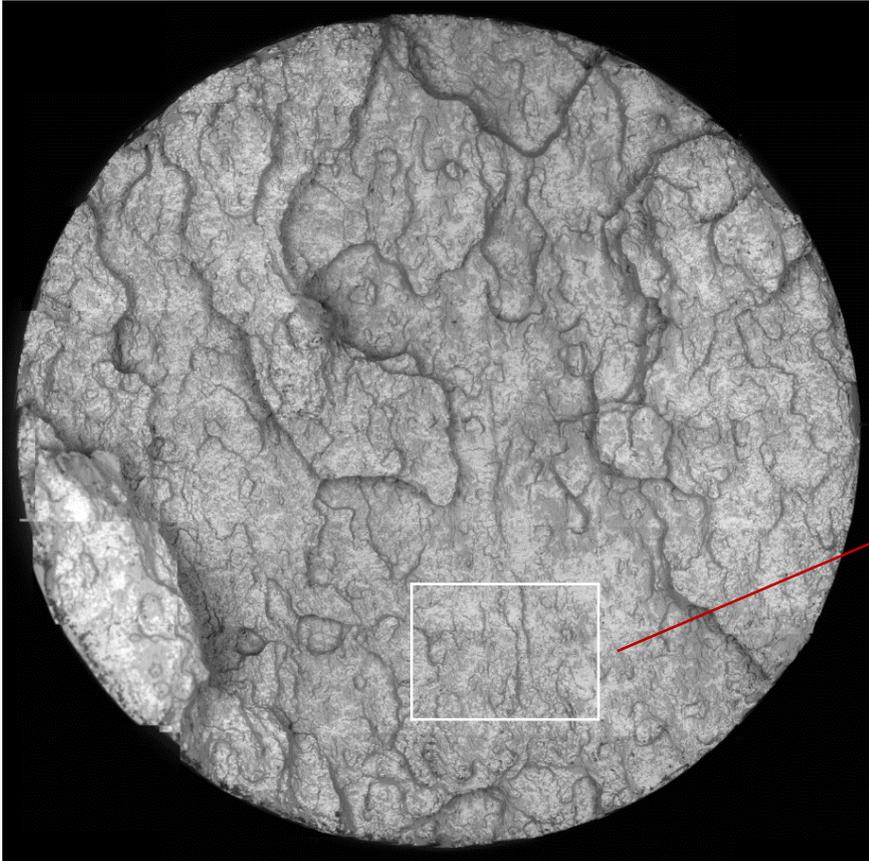


EDS confirms the white particles in SEM image (green color on x-ray map) are Cu-rich

# SEM Fractography of Sample 723C-6

723C-6 LN2 – 1.25%

White particles are Cu-rich precipitates

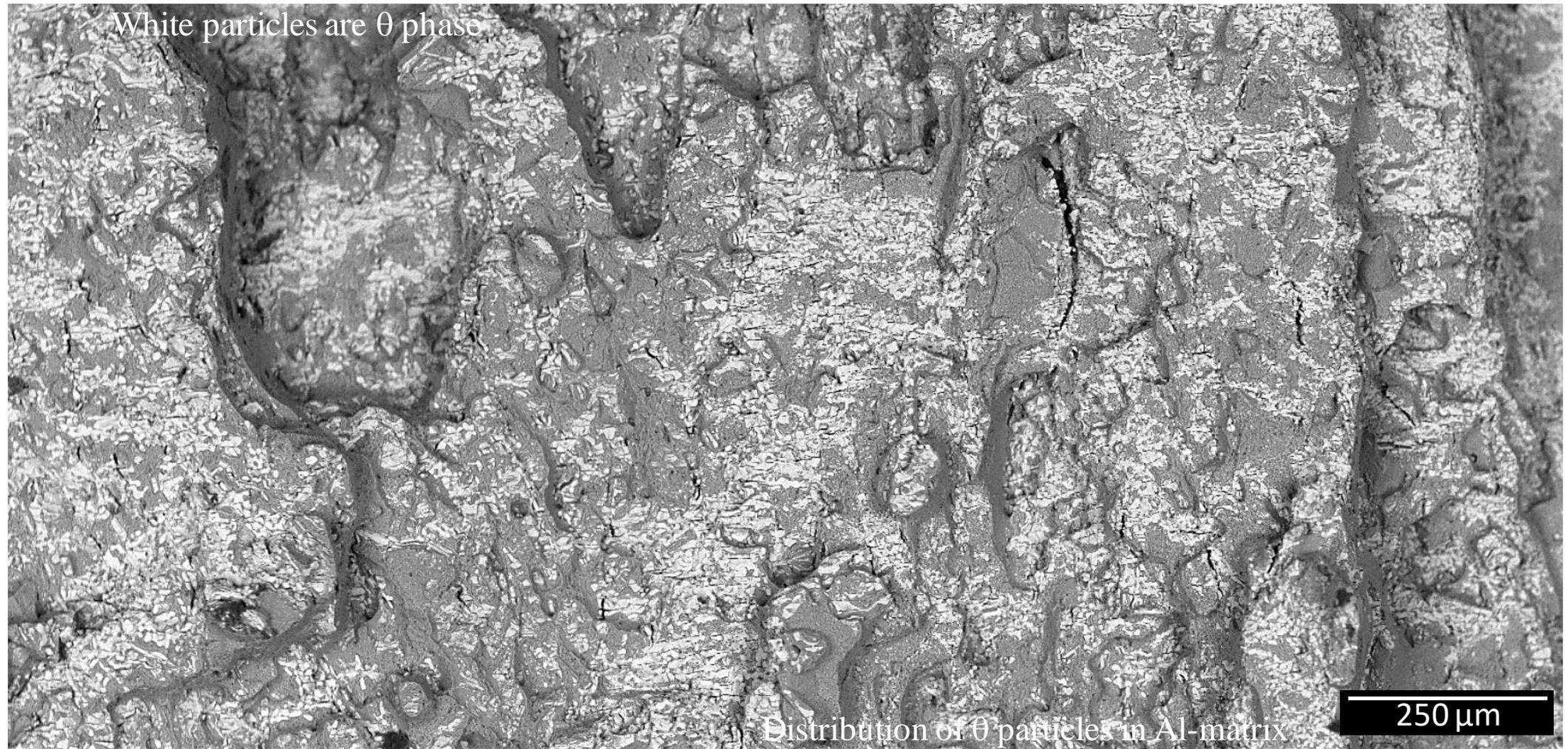


SEM image – entire fracture surface

Distribution of Cu-rich particles in AL-matrix

Cu-rich particles are coarser and have non-uniform distribution

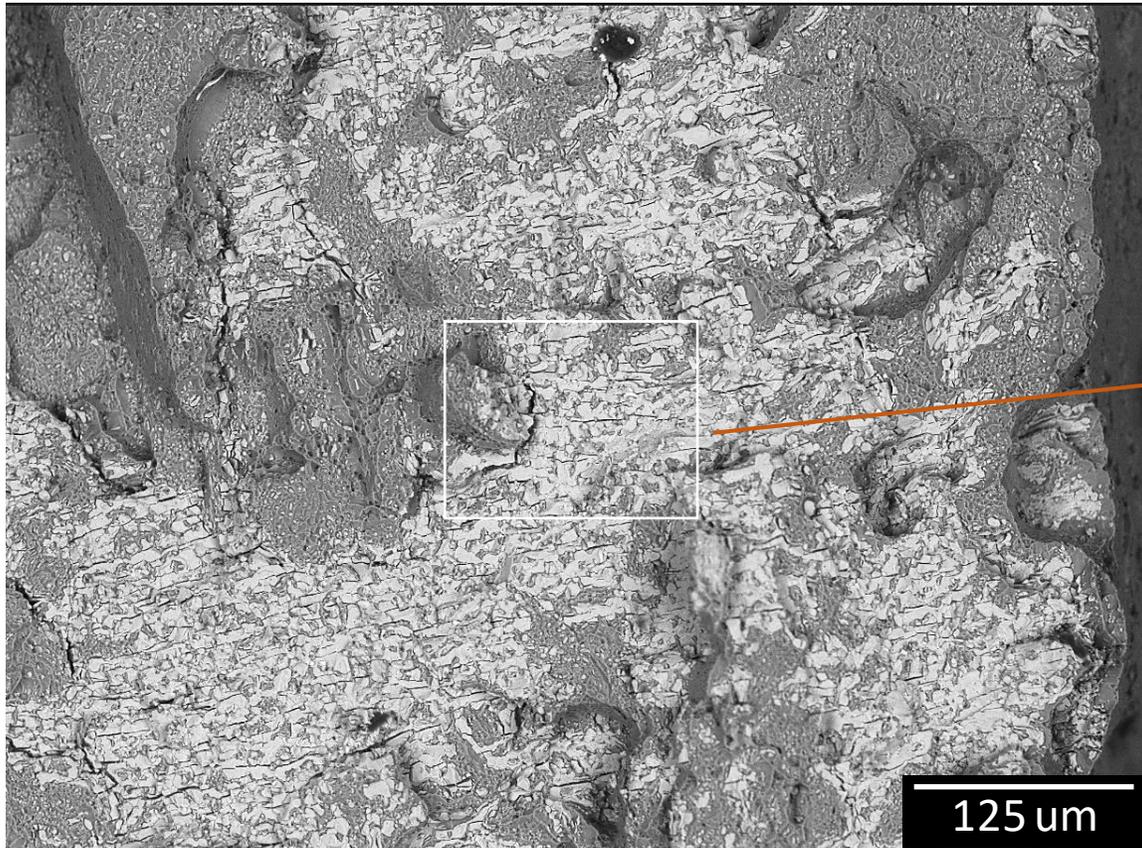
## SEM Fractography of Sample 723C-6 LN2 – 1.25%



Density of Cu-rich particles is unusually high in this region

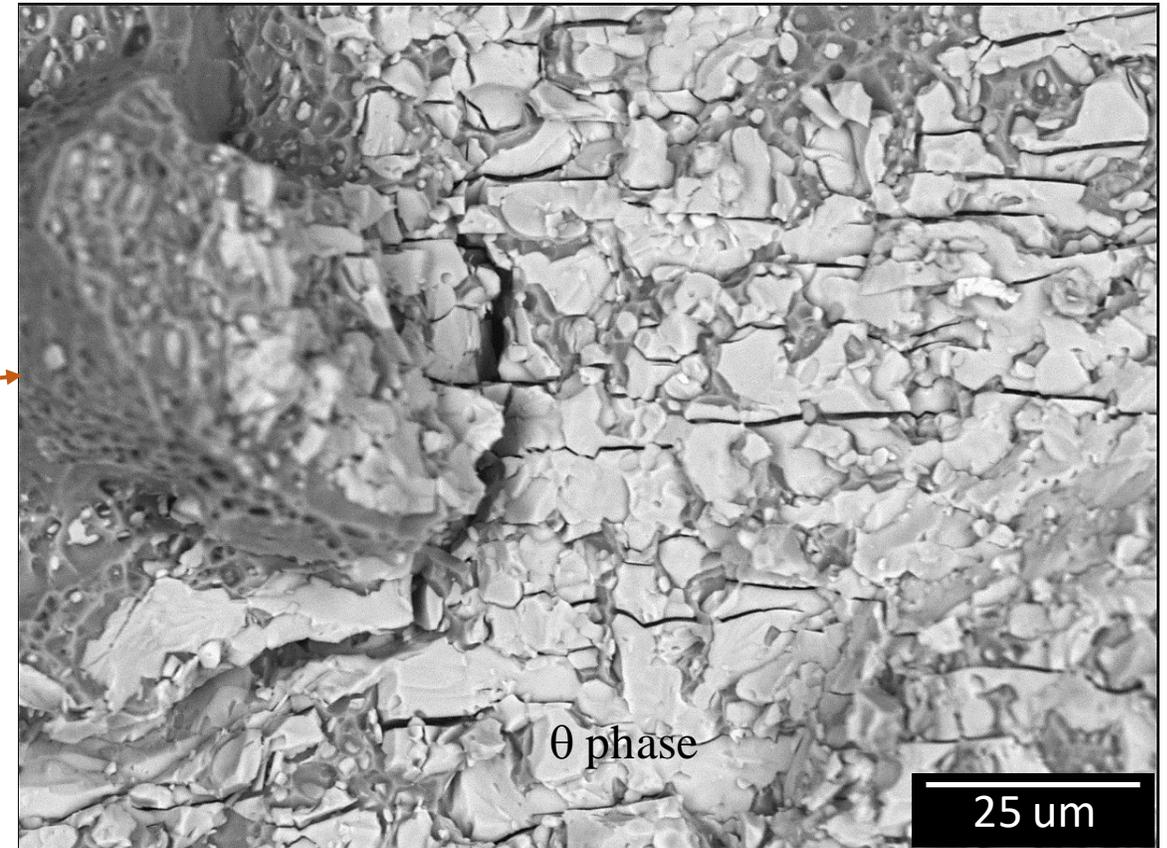
# SEM Fractography of Sample 723C-6

723C-6 LN2 – 1.25%



Clusters of Cu-rich particles with micro-cracks

White particles are Cu-rich precipitates



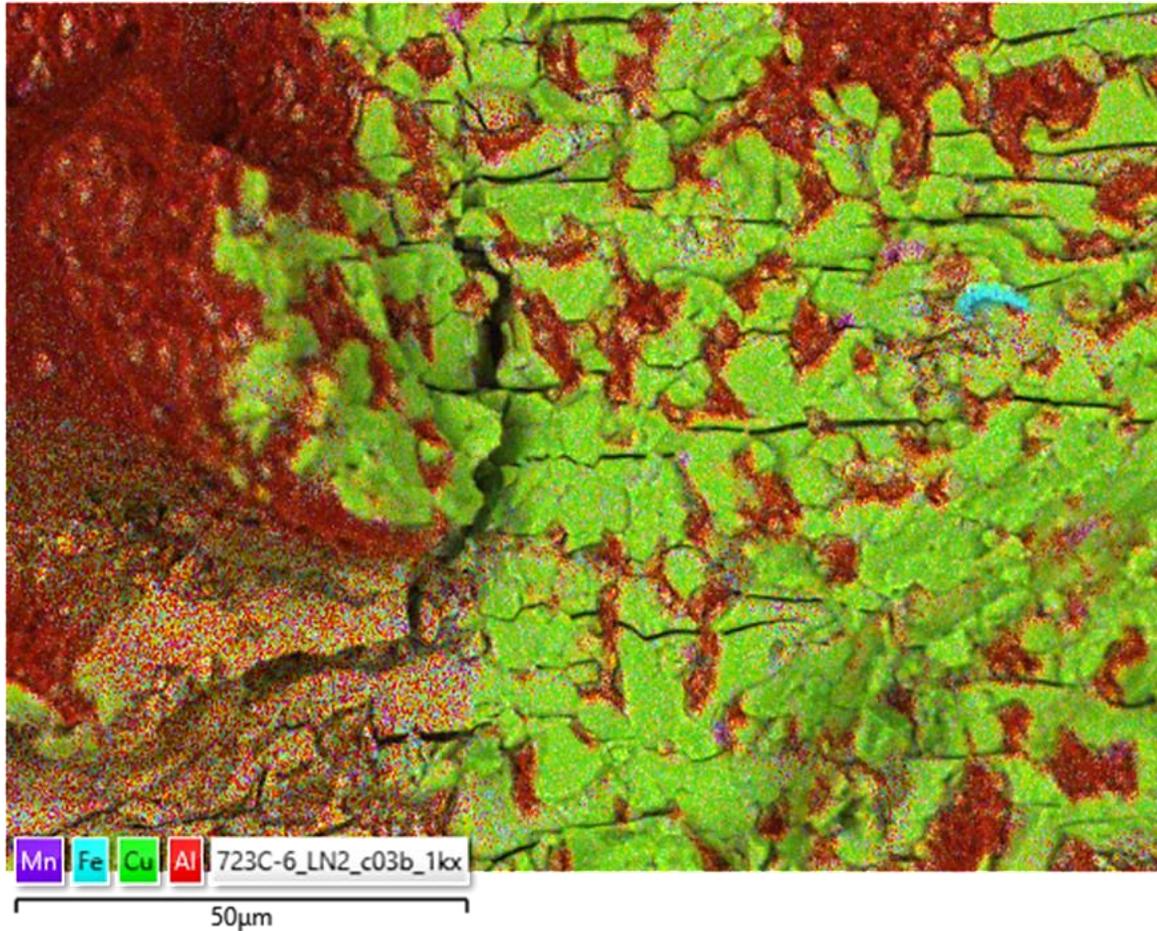
Cracking along or through Cu-rich particles

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3. Many micro-cracks are present in the regions that have numerous Cu-rich particles

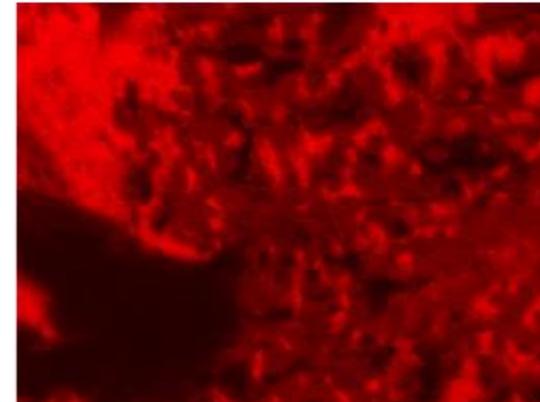
# SEM/X-ray Map for Sample 723C-6

723C-6 LN2 – 1.25%

EDS Layered Image 1

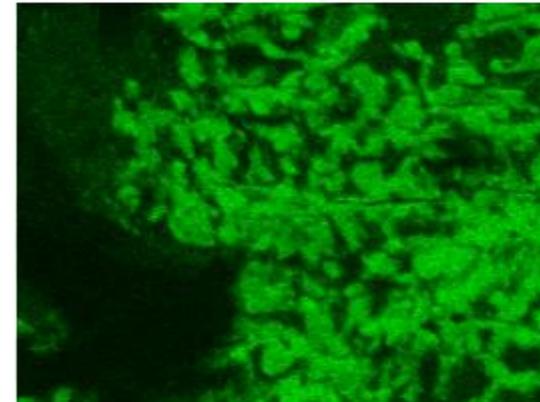


Al Kα1



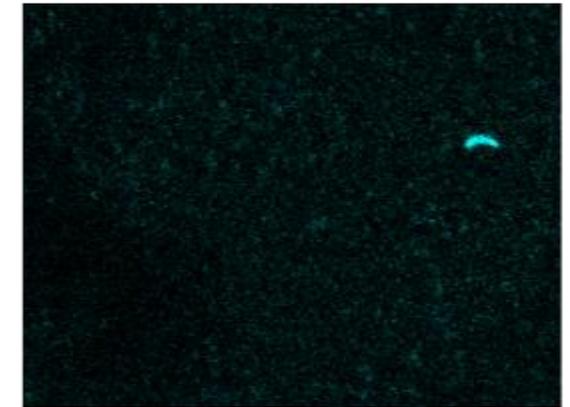
50µm

Cu Lα1,2



50µm

Fe Kα1



50µm

EDS confirms the white particles in SEM image (green color on x-ray map) are Cu-rich

# Summary of Key Findings



## **The high ductility sample (9492854-720-2, LN2) has the following microstructural features:**

- Higher degree of recrystallization, smaller grain size,
- Transgranular fracture, lower area fraction of coarse particles (qualitative analysis)
- Lower electricity conductivity that can be attributed smaller Cu-rich particles and smaller spacing between them



## **The low ductility samples (930071-720C-5, 930082-723C-6 LN2) has the following microstructural features:**

- Lower degree of recrystallization, distinct signs of prior dendritic structure, large grain size
- Intergranular fracture, higher area fraction of coarse particles (qualitative analysis)
- Higher electricity conductivity that can be attributed larger Cu-rich particles and wider spacing between them



## **The drastic reduction in tensile ductility can be attributed to the following factors:**

- The presence of numerous coarse Cu-rich particles ( $\text{Al}_2\text{Cu}$ ,  $\theta$  phase) at grain boundaries
- They are stress risers and will increase  $K_t$  during tensile testing and promote crack initiation
- The greater the number of coarse Cu-rich particles, the easier for micro-cracks to connect
- Many connected micro-cracks lead to larger cracks and a drastic reduction in tensile fracture elongation